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SUBU CHARACTERISATION: BATH FLUID DYNAMICS VS ETCHING RATE

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Abstract

The chemical polishing bath SUBU is widely used at CERN to prepare copper RF cavities surfaces before niobium thin film coating; examples are HIE-ISOLDE, LHC and future FCC accelerating cavities. The performance of the polishing process is affected by bath temperature and fluid dynamics. As part of on-going activities to characterise SUBU, the actual study was done to identify a correlation between the etching rate and physical parameters linked to the bath fluid dynamics. A first approach was made using experimental data from a simplified model setup, transposing them via numerical simulation to a real cavity geometry and verifying the agreement with an experiment in a real size (HIE-ISOLDE) mock-up. In a second approach to improve the accuracy of the calculation, the relation of the measured local etching rates, extracted from the mock-up, to flow dynamics quantities extracted from simulation was investigated. As a result, a relation between the local etching rate and the turbulence kinetic energy was obtained. This relation can be exploited to improve the polishing tools and so optimise the current process, as well as to predict the etching rate in other cavity geometries.

INTRODUCTION

The chemical polishing SUBU (mixture of sulfamic acid, hydrogen peroxide, n-butanol and ammonium citrate) is a surface treatment carried out at CERN to ensure the optimal performance of accelerating RF cavities based on niobium thin film on copper. The SUBU treatment is used to remove the damaged layer of the copper substrate originated during the cavity fabrication and to smooth the inner surface to hinder the electron emission induced by the high electric field. This copper surface treatment is widely used as it is relatively easy to setup, independently of the cavity geometry. In the specific case of the HIE-ISOLDE quarter wave cavities, the polishing bath SUBU flows into the cavity, which is kept in vertical position, through four tubes and it flows out through the top and the beam apertures, as shown in Fig. 1.

The two variables that have a major impact on the polishing process are temperature and bath fluid dynamics (FD); the first is easily kept constant across the entire surface, but constant FD conditions along the whole cavity's structure is much harder to achieve.

In a previous experimental study, the impact of the SUBU bath fluid dynamics on the etching rate was assessed; samples were exposed to controlled bath agitation and characterised in terms of etching rate and resulting surface roughness.

This investigation was conducted in a laboratory set-up consisting of a rotating cylinder electrode (RCE) that enabled to impose a known angular velocity on a sample.

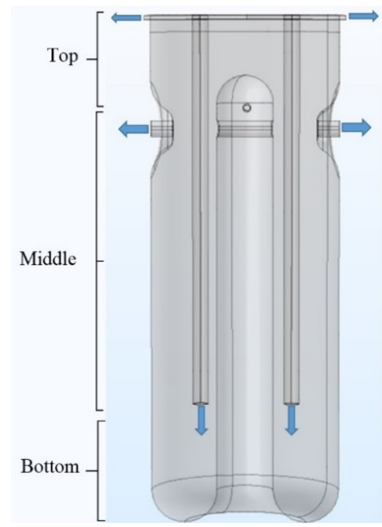

Figure 1: Scheme with the SUBU bath inlets and outlets during the polishing process.

The outcome of this work was that fluid dynamics plays indeed a fundamental role on the etching rate distribution, and on the achieved roughness; as expected, the removal of material is proportional to the bath FD values neighbouring the sample wall.

RATIONALE OF THE APPROACH

The experimental work mentioned previously justifies the study of the impact of the FD on the etching rate. This would allow to analyse the homogeneity of the polishing and to study its impact on the RF performance.

Therefore, the objective of the present study is to identify, through modelling, a possible correlation between the etching rate and variables linked to the bath fluid dynamics. Two different approaches were made; they are described hereafter.

First Approach

In the first approach, the etching rates measured in a laboratory setup at different agitation conditions were correlated to two FD variables (shear rate and turbulence kinetic energy (TKE)). These two variables were obtained with the help of a simulation software using as model the configuration of a laboratory setup and, finally, tested in a real cavity geometry.

The laboratory setup was composed of a sample holder RCE immersed in a defined volume of the SUBU bath, as shown in Fig. 2.

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The sample surface to bath volume ratio (0.23 m^{-1}) was chosen to be as close as possible to the actual polishing facilities (HIE-ISOLDE and LHC cavities).

The software COMSOL® was used to model the fluid dynamics and thus to extract the values of shear rate and TKE at the different rotation speeds.

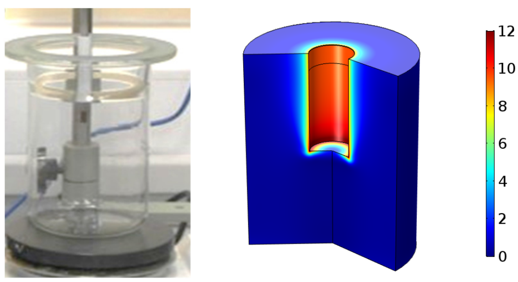

Figure 2: Laboratory set-up; Shear rate distribution [s^{-1}] resulting from the simulation.

The shear rate as well as the TKE were considered good variables to work with as they can be easily assessed by the software and are directly defined on a boundary; in this case, the wall to be etched. The shear rate represents the gradient of velocity at the vicinity of a reference layer; the TKE is the mean kinetic energy per unit of mass associated with eddies in the turbulence flow and it can be due to fluid shear, friction and buoyancy as well.

Figures 3 and 4 show the correlations obtained with the etching rate and shear rate or TKE. Linear functions were used as an interpolation of the obtained data. When there is no bath movement the etching rate is non zero, and with high flow rates the etching rates increase.

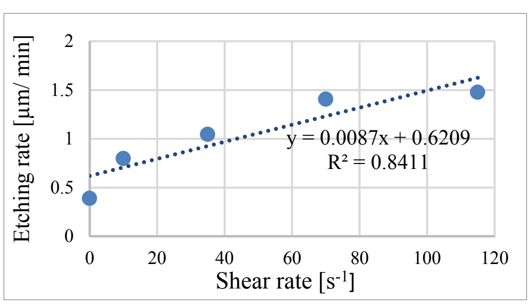

Figure 3: Measured etching rate as a function of the simulated shear rate.

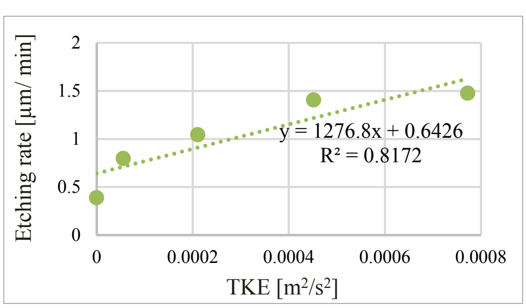

Figure 4: Measured etching rate as a function of the simulated TKE.

The linear interpolating functions were then transposed to the real geometry of the HIE-ISOLDE cavity using COMSOL. The CFD (Computational fluid dynamics) module was used to simulate the bath flow inside the cavity and extract the shear rate and TKE. Then, the correlations shown previously (Figs. 3 and 4) enabled to calculate the etching rate distribution across the cavity as represented in Fig. 5.

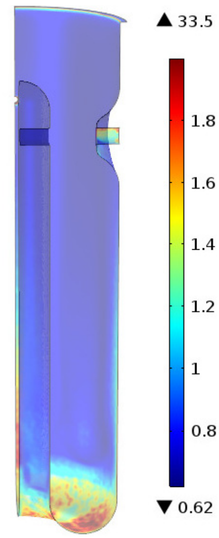

Figure 5: Etching rate distribution [µm/min] in HIE-ISOLDE obtained using the correlation in Fig. 3.

To validate the results, a test was performed making a SUBU polishing in a real size mock-up cavity with the geometry of the HIE-ISOLDE one.

The HIE-ISOLDE mock-up cavity allows to attach samples onto exact locations, enabling to check the agreement between the real local etching rates and the values given by the simulation.

Taking as reference the etching rates measured in the different areas of the cavity, in Fig. 6 the plot of the relative error from the results obtained by the simulation is shown.

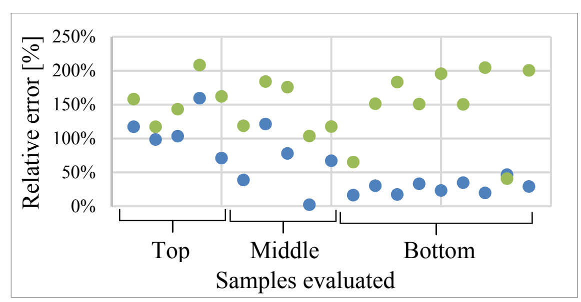

Figure 6: Relative error from: ● Correlation etching rate-shear rate ● Correlation etching rate TKE.

As observed in the previous plot, there is an important (up to a factor of 3) mismatch between etching rates determined in the mock-up and the values calculated with the software.

Second Approach

In this approach, a fitting was made between the local etching rates measured on the mock-up cavity (instead of the laboratory setup) and the flow dynamics quantities extracted from simulation on the same geometry, as shown in Fig. 7.

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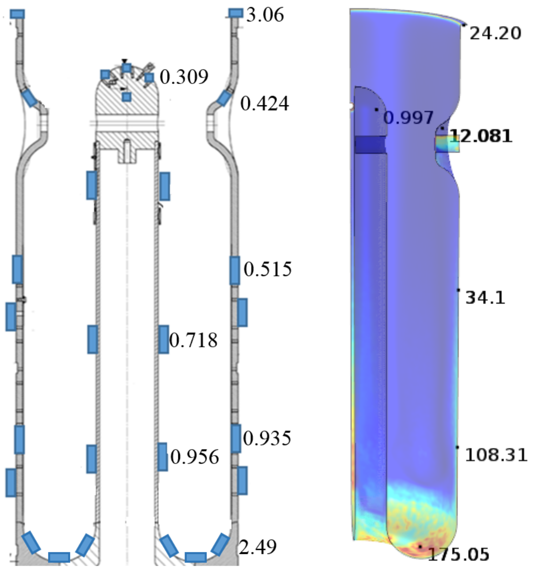

Figure 7: Local etching rates [μm/min] from the mock-up cavity and calculation of the shear rate [s^{-1}] in this locations.

The test in the mock-up cavity was performed during twenty minutes for different flow rates of the SUBU bath in order to get a wider range of data.

In Figs. 8 and 9 the two obtained correlations are shown.

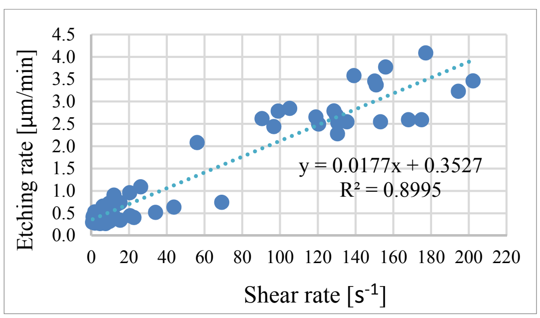

Figure 8: Correlation etching rate from mock-up test vs shear rate.

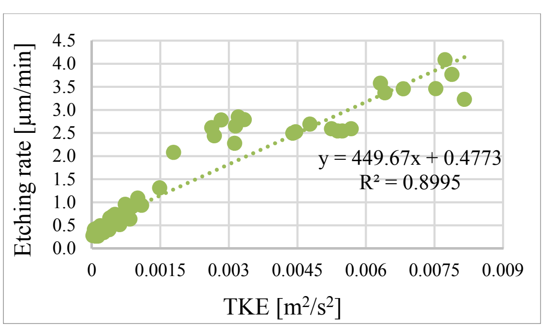

Figure 9: Correlation etching rate from mock-up test vs TKE.

As observed both entities correlate in a similar way, thus, in principle, they could be applied indistinctly.

ROBUSTNESS OF THE COMPUTATIONAL SIMULATIONS

In this part of the study, the simulations to calculate the flow dynamic variables were analysed in order to evaluate the impact of certain choices in the modelling software.

Transient- Steady State Analyses

Both studies were conducted to understand how different software parameters influence the calculated shear rate and the turbulence kinetic energy.

From the transient study, it was observed, as shown in Fig. 10, that the steady state can be considered attained after approximately 70 seconds, which corresponds to 6 % of the process time. Once in steady mode the turbulence kinetic energy and the shear rate evolve with some oscillations.

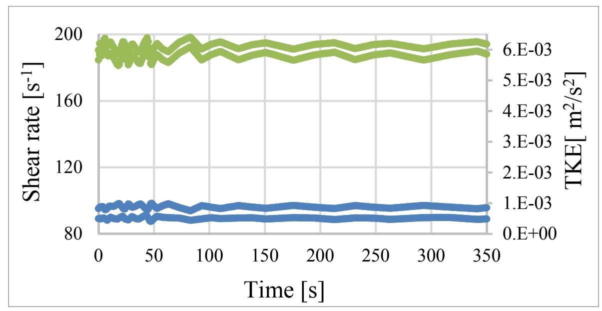

Figure 10: Plot of the evolution of the shear rate and TKE with time in two points: ■Shear rate ■TKE.

The solutions obtained in the steady state study were compared with the previous results, obtaining a good agreement as shown in Table 1.

Table 1: Transient and Steady State Results

	Shear rate [s^{-1}]		**TKE [m^2/s^2]**	
Sample	Transient	Steady state	Transient	Steady state
1	13.7	13.7	3.7E-04	3.7E-04
2	89.3	89.6	9.6E-04	9.6E-04
3	17.5	17.6	6.1E-03	6.1E-04
4	96.1	96.4	5.8E-03	5.6E-03

Therefore, the impact of the transient period on the computed shear rate and turbulence kinetic energy is not meaningful. It also shows that the obtained results are robust, as the two different computing approaches converge to the same solution.

Mesh Impact Evaluation

Another mean to evaluate the robustness of the model is to see if the obtained data converge with increasing mesh density (smaller size).

Two new simulations were performed using finer meshes, with a bigger number of boundary layers in the cavity wall (13 and 16 layers respectively) in order to evaluate the variables' convergence.

As shown in Fig. 11, it was observed a certain trend in the shear rate within the different meshes, however the

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convergence of this parameter is not achieved. It must be referred that due to computing limitations, the most appropriate model for shear rate calculations (SST model) was discarded and replaced by the k-epsilon turbulence model. The k-epsilon model is robust, however it makes some simplifications resulting in less accuracy near the boundary wall, where shear rate is calculated. A new software version might be able to tackle this constraint.

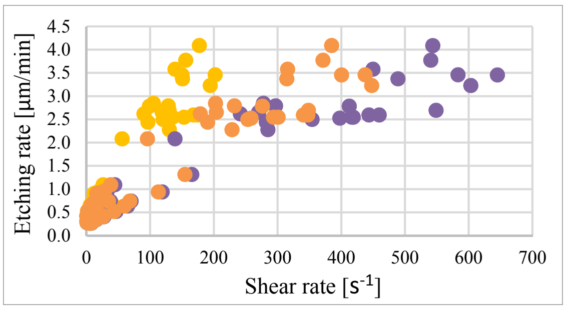


Figure 11: Comparison correlation etching rate-shear rate obtained with different meshes: ● Initial mesh ● 2nd mesh (13 layers) ● 3rd mesh (16 layers).

However, the TKE values remained almost invariable as shown in Fig. 12, which was an indicator of its convergence.

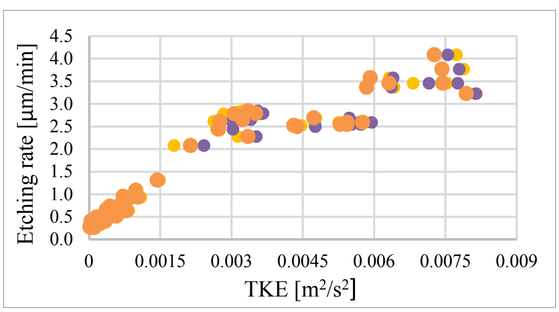


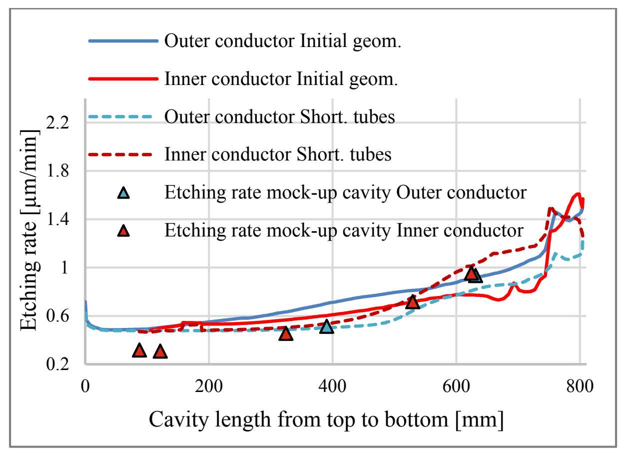
Figure 12: Comparison correlation etching rate-TKE obtained with different meshes: ● Initial mesh ● 2nd mesh (13 layers) ● 3rd mesh (16 layers).

CONCLUSIONS AND PROSPECTS

Experimental values from existing laboratory setup model were not able to predict the etching rate on a real cavity geometry. One possible explanation might be related to the fact that samples in the laboratory setup and in the mock-up cavity were processed differently. In the mock-up cavity, as in the real cavities, the bath is flowing inside the cavity and the cavity wall is not moving; while in the laboratory setup it is the wall that is moving and the bath standing still. In terms of FD, there is an important difference when defining the wall boundary conditions. In the first case, the velocity at the wall is zero in any circumstance; whereas in the second case, it is variable as it will correspond to the imposed velocity on the RCE. Thus, a future setup consisting on a tube sample standing vertically with the flow passing through will be put in place and studied; the extracted data will be compared with the existing ones.

A modification of the mesh in the simulation showed that the values of the shear rate were strongly dependent on the mesh density and did not show a convergence within the studied values. Instead, the TKE values were very robust against a change of mesh; therefore, the relation between this physical quantity and the etching rate has been selected to predict local etching rates in a complex geometry as the RF cavity. A reasonable correlation between the etching rate and the turbulence kinetic energy has been identified for the HIE-ISOLDE cavities.

In the polishing process, in order to guarantee the RF performance of the cavities, the thickness of material removed should be as homogenous as possible to keep the design geometry. So one of the applications of the identified correlation between etching rate and FD is to enable to define polishing tools that can lead to an even as possible thickness removal. As an example, in Fig. 13, it is shown the effect in the etching rate distribution along the outer and inner conductor of the HIE-ISOLDE cavity by reducing 10 cm the length of the bath inlet tubes, in the SUBU polishing tool.



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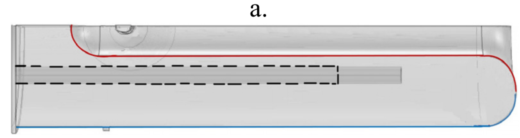

b.

Figure 13: a. Comparison between the etching rate of the inner and outer conductor using different tube lengths; b. Cavity cross section with inner and outer conductor.

Even in case of not achieving an even etching on the cavity surface, just being able to map and quantify the removed thickness could already be an important improvement as it would allow to evaluate the impact on the RF performance or even change the base design in order to cope with an uneven etching process. Thus, another use of the correlation is to provide the expected geometry changes after the etching process. In Figure 14, it's shown a 3D representation of the removed thickness for a 20 minutes SUBU processing in a HIE-ISOLDE cavity with existing polishing tool.

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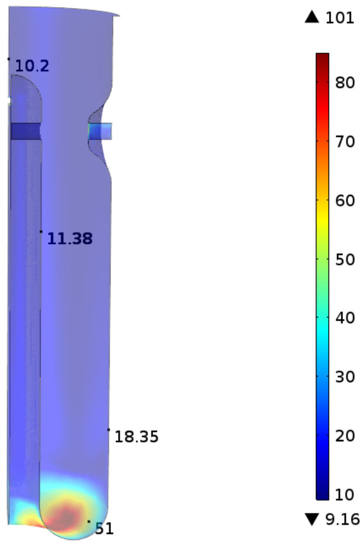

Figure 14. Distribution of the thickness removed [μm] in the chemical polishing.

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