

# SURFACE MORPHOLOGY AT THE QUENCH SITE

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## *Abstract*

It has been demonstrated recently that local magnetic field enhancement can originate from roughness (e.g. steps at grain boundaries) [1]. We are willing to investigate if the quench site detected in superconducting niobium cavities can be related to such morphological defects. We recently developed a replica technique which allows to reproduce the internal surface of cavities (non destructive testing). Modeling of local increase of the magnetic field has been done in accordance with the morphological characterization. In particular we could observe the displacement of the quench location, and the evolution of the former quench site before and after chemical etching.

## INTRODUCTION

Surface morphology is extremely difficult to characterize. Firstly, due to the fractal nature of the surface, the observation is strongly scale dependent, and only comparison made in the same scale condition are relevant. Secondly the measure with high accuracy itself is difficult to get, and is often disturbed by instrumental artefacts. And thirdly the translation of this measure into figures is generally relevant for a sole aspect of the surface (peak-to-valley height, density of defects, planarity, gloss, etc). For instance, roughness is a poor indicator for the local curvature radius of steps at grain boundaries: different distributions of shapes from the smoothest one to the sharpest, but with the same peak-to-valley height will exhibit the same  $\sigma_{rms}$ . Of course, the local radius cannot be measured in absolute, (what do we know about the disposition of the local steps in the atomic planes, should we even take it into account?), but it can be approached, keeping some restrictions in mind. We show hereafter how to measure the morphology in a way that refers to its influence on RF behaviour, but will also try to show the limitations in the measure procedure as well as in its numerical interpretation.

## HOW MEASURING THE SURFACE MORPHOLOGY?

Two main categories of techniques allow measuring the surface state: mechanical scanning with a tip which implies a direct contact with the sample, or optical techniques, which can be global like ellipsometry, or local, with a scanning optical sensor.

A global method is obviously not adapted to our issue, although it can give some statistical indication about the general "behaviour" of a surface treatment compared to another.

Scanning methods (mechanical as well as optical) allow measuring the local situation, there are thus more interesting. One has to keep in mind that whatever the sensor, the resolution is limited by the mechanical resolution of the displacement system.

### *Optical scanning microscopy*

This technique seems attractive since it prevents contact between the sample and the optical sensor. Unfortunately, they are sensitive to color change on sample, and slope with value above the half optical aperture of the objective cannot be measured. Electronic artefacts appear at dramatic variations and need to be mathematically filtered. Thus the issue of local curvature cannot be solved with optical sensors.

### *Mechanical scanning*

Two types of equipments stands in this category: classical roughness measurements like 2D-Talysteps, or 3D mechanical microscope, which can explore detail down to 1  $\mu\text{m}$  or slightly better, and atomic level apparatus like AFM or STM. In general, the gain in precision, accuracy is at the expense of the explored area. Difficulties of accurate locating and lack of clearance are also troubles with small scale of observation. That's why 3D- microscopy, even if it is limited to  $\sim 1 \mu\text{m}$ , constitutes a good compromise to investigate surface morphology. In our case, surface roughness acquisition is carried out with a profiler equipped with two motor for the  $x$ - $y$  displacements. The sensor used is a Perthen-Mahr FRW750 with a 250  $\mu\text{m}$  range and 0.1  $\mu\text{m}$  resolution. The stylus has a diamond tip of 90° conisphere shaped with a tip radius of 2.5  $\mu\text{m}$ , and the contact strength is 0.1 g. The height detection is inductive.

The acquisition is a convolution between the corrugation of the surface and the shape of the tip. In our case angles sharper than 45° cannot be accurately measured, but we shall show in the next § that we could overcome this difficulty. By the way, even on heavily etched samples, the roughness is about some microns mostly at grain edges, compared to the width which ranges from  $\sim 100 \mu\text{m}$  to some mm. Observed slopes keep relatively low, at least at this observation scale.

Another trouble arises when measuring internal state of cavities: their curved shape. In a first steps one has to make sure that the whole surface keeps within the range of clearance of the sensor. Secondly, one has to separate the curvature from the roughness itself. This problem can be solved partially by the use of mathematical corrections (least mean square, filters ...) But we will see in hereafter (§mathematical treatment) that one has to use these techniques very carefully to achieve comparative studies.

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## REPLICA TECHNIQUE

A polymer replica technique is a non destructive method (resolution < 1 micron) that allows to reproduce the inner surface of RF cavities [2]. There are many different types of replica which can be resin, rubber or plastic based. The criterion of choice is the accuracy with which they reproduce the surface: this accuracy must be better than the measuring precision.

### Critical analysis of the replica material choice

Accuracy of reproducing the surface is material dependent and may vary with the different types of replica polymers. Moreover it is a two-steps process:

- First making a negative replica of the inner surface. Co-polymerisation of vinylpolysiloxane. with a hydrogenated polysiloxane
- Then making a positive replica of the negative shape with a bi-compound mixture of polyurethane.

On samples, we have checked that afterwards ultrasonic cleaning in 65°C basic detergent leaves the niobium surface free of polymer. Thus this technique can be used on cavities between each RF test without leaving residue, provided a sufficient cleaning procedure. Other troubles occur: for example some invisible bubbles inside the vinylpolysiloxane will compress under pressure during the second step, giving artificial defects on the replica.

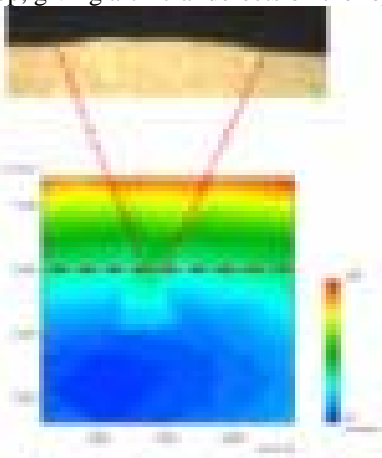


Figure 1: Micrograph of the cut of the positive replica, compared with the contour line of the 3D micrograph.

Meanwhile, the advantage of replica is that they can be reproduced many times, and can be cut in order to determine more accurately the profile of the surface. In figure 1, one can see that the contour of the prominent grain found in the vicinity of the quench is surprisingly smooth. Meanwhile calculation of the field enhancement with realistic shape values deduced from the both micrographs show a local increase of the magnetic field about 40% to 60%. (See experimental section).

## MATHEMATICAL TREATMENTS

As we need to evaluate the roughness resulting of specific surface treatment, mostly due to different etching rate for neighbored grains (with radius ranging from 100  $\mu\text{m}$  to 1 or 2 mm), 6 mm acquisition length was

chosen, so that we are sure to cross several grain boundaries.

### Levelling and filtering

It is best to level as well as possible in order to subtract the shape factor from the roughness itself. For cavity shaped replica (spheroid with two radii: 43 mm and 103 mm) and large acquisition length (6mm), manual as well as mathematical levelling is critical. We have done our best to orient the replica before the acquisition, so that the component stays within the clearance range of the sensor.

However, we need to level the surface after measuring by using software algorithms. We try two types of mathematical levelling in order to suppress cavity shape:

- Polynomial shape subtraction (calculated by least mean square) from degree 2 to 4,
- High pass filter by “sliding window” averaging of  $x$  and  $y$  tuneable half widths.

Changing the parameters values in levelling or filtering has a drastic effect on the overall result. In figure 2, this effect is shown for a non annealed electro-polished cavity on the welding seam (filtering window:  $x=1, y=50$ ).

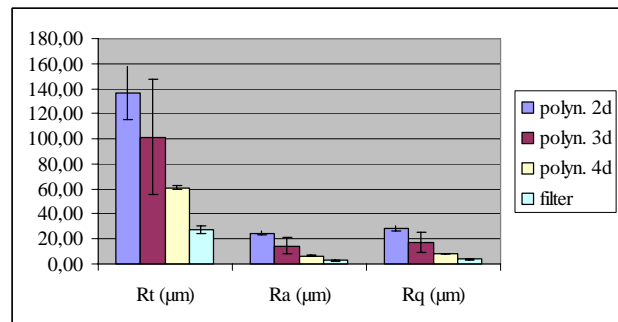


Figure 2: Influence of different mathematical levelling on amplitude parameters ( $R_t$ ,  $R_a$ ,  $R_q$ ). “polyn.  $i$  d” is polynomial by least mean square of degree  $i$ . Once more it shows that accurate comparison can be made only if every conditions rare the same. In this case, the use of the exact mathematical formula of the cavity like spheroid should be an interesting step.

## ROUGHNESS RESULTS

### Experimental conditions

Acquisition are taken on an area of 600 points with 10  $\mu\text{m}$  step by 600 lines separated by 10  $\mu\text{m}$ . A 3<sup>rd</sup> degree polynomial levelling is applied. We have removed results exhibiting bubbles due to negative replica. The welding seam is parallel to  $x$  axis.

We have compared the mean value of various roughness parameters ( $R_t$ ,  $R_a$ ,  $R_q$ ,  $S_k$ ,  $E_k$ ) on two different cavities: an annealed cavity chemically polished (FNP) and a not annealed cavity electro-polished (EP).

### Welding seam influence

The welding seam clearly influences amplitude parameters  $R_t$ ,  $R_a$  and  $R_q$ . For EP surfaces, values are 7 time higher on the welding seam than away from the thermal affected surfaces. This ratio is only 2.4 in the case of FNP.

### What about quench?

Classical roughness parameters are unable to distinguish quench areas from others. Topological analysis with conformal equivalent ellipsoid will be published elsewhere.

## MODIFICATION OF QUENCH SITE WITH POLISHING

### Quench location before/after chemical polishing

Quench location is determined via temperature mapping as described in [2]. In this first experiment we had noticed a prominent grain that was likable to provoke field enhancement (see figure 3). After further 20  $\mu\text{m}$  chemical etching, the quench site is displaced about 2 cm away from its first location. Replicas made on the new site as well as on the former one allow estimating the local field enhancement. Figure 3a shows the first quench site, 3b the same location after etching, and 3c the new quench area, along with field enhancement estimation.

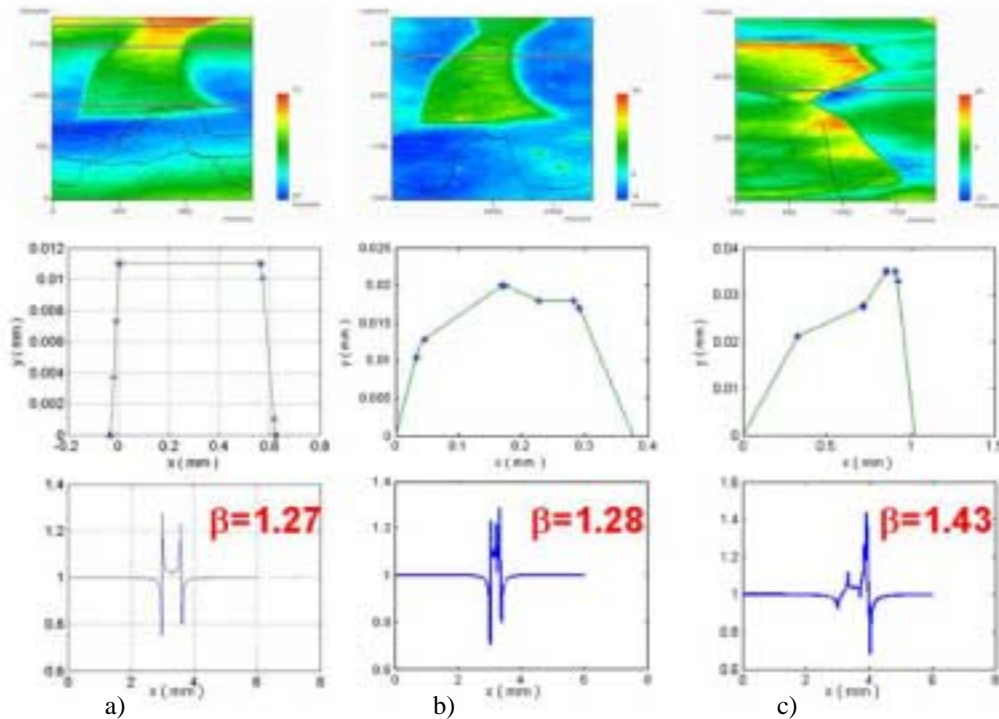


Figure 3: Contour line of replicas, modelling of the edge profile and  $\beta$  (field enhancement factor) for a) first quench site, b) same area after 20  $\mu\text{m}$  etching, c) new quench location.

## CONCLUSION

Welding seam and thermally affected zone affect roughness parameters differently from the rest of the cavity. This fact deserves a complementary study of the morphology in function of grain size.

The influence of the slope and height at edge of the grain seems to be predominant compared to the curvature radius. No effect of width on the field enhancement factor is observed.

We have clearly evidenced that, after a chemical etching, the field enhancement factor is higher in the new quench region than at the former one.

## FIELD ENHANCEMENT CALCULATION

The edge of the grain is modelled by series of segments and arcs in order to fit the shape of the grain (height, width, radii). Then the field enhancement is calculated for a field parallel to the x axis. The field  $H(x)$  is equal to 1 when away from the defect. Note that this 2D approach doesn't take into account the length of the grain. Thus the enhancement factor is only a relative estimation. We have also studied the influence of the local curvature radii and of the grain width (see in conclusion).

### Field at quench site

Figure 3 gathers the data for each quench site, including the field enhancement factor ( $\beta$ ). We show that this factor is not much changed upon 20  $\mu\text{m}$  etching, as well as the shape of the grain. But an edge exhibiting a larger  $\beta$  factor has been found in the area of the new quench. These experimental facts support a geometrical explanation of the quench, and we hope that further experimentation will allow to precise this trend.

Experimental evidences tend to support a morphological origin of the quench.

## REFERENCES

- [1] J. Knobloch, et al. "High field Q-slope in superconducting cavities due to magnetic field enhancement at grain boundaries". in 9th workshop on RF Superconductivity. 1999. Santa Fe, New Mexico, USA.
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