Review of high field Q-slope, surface measurements*

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Abstract

High field Q-slope remains one of the main physics problems in the field of niobium radio frequency superconductivity, which needs to be addressed. Mild temperature baking at 100-120°C in ultra high vacuum for about 48 hours was empirically found to improve or completely remove the high field Q-slope in niobium cavities. One of the approaches to tackle the problem is to utilize surface analytical techniques such as XPS, SIMS, EBSD etc. in order to look for mechanisms underlying baking-induced improvement and clues for the high field Q-slope origin. In this paper current results of surface studies are reviewed and their implications are discussed.

INTRODUCTION

High field Q-slope appearance in niobium superconducting cavities starting at peak magnetic fields of about 90-100 mT is a widely observed phenomenon in the absence of any multipacting or field emission.

In order to try to understand the effect, several key properties should be emphasized.

1. Mild baking at 100-120°C for about 48 hours was shown to consistently remove the Q-drop in elecropolished (EP) cavities, or generally improve it in buffered chemically polished (BCP) ones. Such an improvement is a crucial point, which most of the studies are focused on.
2. Baking benefit is preserved after cavities are exposed to atmospheric air for up to several years [1]. High pressure rinsing, hydrofluoric acid treatments, and anodizing to less than 30 V were shown not to make the Q-drop reappear too [1, 2].
3. Temperature mapping studies of the distribution of losses in the high magnetic field regions of cavities in the Q-drop regime indicate that heating distribution is not uniform but exhibits patchy character with some regions being heated up stronger than other whereas the onset field seems to be the same for all regions [4]. Such a non-uniformity suggests that whatever the cause of the Q-slope, its concentration is higher in the regions of stronger heating.

EXPERIMENTAL APPROACHES

Up to now two different approaches to high field Q-slope related surface studies were utilized.

The most widely used techniques are based on investigating niobium samples, which underwent treatments similar to those on niobium cavities. This approach is justified for addressing properties 1 and 2. Though the main disadvantage of it is the local character of surface studies, which might not be representative for the whole cavity wall surface. Thus a statistical approach based on investigating many similar samples is required.

In order to address property 3, another technique was recently applied. It is based on testing a non-baked cavity with the temperature mapping system attached, identifying and dissecting regions of stronger and weaker heating, and applying a range of surface analytical techniques to samples obtained.

MILD BAKING AND AIR EXPOSURE

In this section the overview of experimental results related to properties 1 and 2 is presented, thus addressing the question - *what changes during mild baking and is preserved after air exposure?*

Superconducting properties

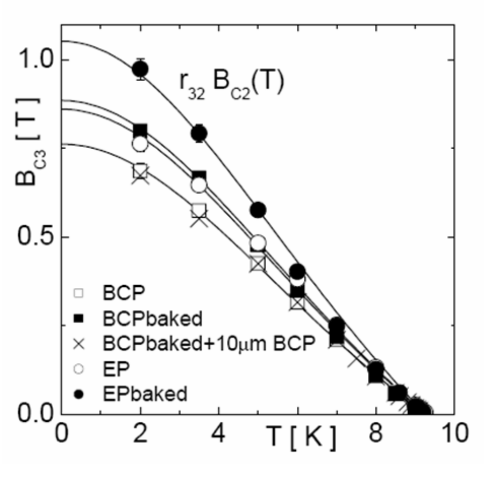

Figure 1: Change in the surface critical field B_{c3} due to mild baking [5].

A direct measurement of a critical field was recently performed on niobium samples.

One of the interesting results is shown in Fig. 1. In this study [5] surface critical field B_{c3} was measured for BCP and EP treated niobium samples before and after mild baking. It appears that B_{c3} is higher for EP than for BCP sample and baking effect is clearly observed in B_{c3} increase.

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Although it correlates to baked EP cavities performing better than baked BCP ones, it does not correlate well with the similarity between EP and BCP without bake.

A disadvantage of these techniques is that they do not provide any information on what the underlying Q-slope mechanism is, but just provide different aspects of baking improvement.

Among possible low scale properties that change during mild baking and are going to be addressed below are:

- Oxide structure, including interface
- Distribution of intestitial impurities
- Crystalline microstructure

Oxide structure

A most widely used technique for niobium oxide characterization is X-ray photoelectron spectroscopy (XPS), descriptions of which can be found elsewhere [3]. It allows obtaining detailed oxide structure with sensitivity of about one atomic percent and information depth of 7-10 nm depending on the X-ray source energy.

Several studies were performed on the effect of mild baking on niobium oxide structure [6, 7, 8]. From Fig. 2 the effect is the slight decrease in Nb_2O_5 thickness due to its partial conversion to NbO_x suboxides after baking at 100°C, the effect being much more pronounced at 160°C baking temperature. However 160°C baking is not used to treat high field Q-slope.

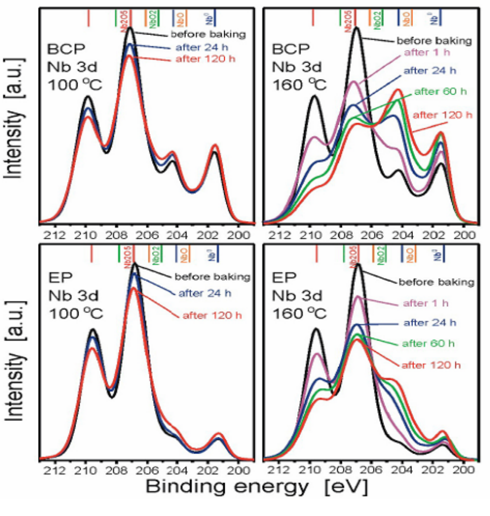

Figure 2: Effect of in situ 100 and 160°C baking on niobium oxide structure [6].

Air exposure effect on niobium sample baked in situ was studied in [9]. As shown in Fig. 3 all change in the oxide structure introduced by UHV mild temperature baking is eliminated by air exposure, whereas baking benefit in cavities is preserved even after few years in atmospheric air.

The conclusion, which should be drawn from the studies above, is that oxide modification is not responsible for the mild baking benefit suggesting that oxide and oxide/metal interface are not responsible for the high field Q-slope too.

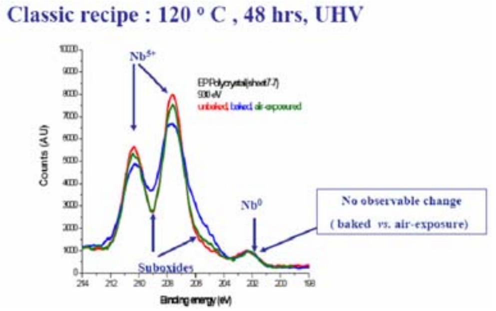

Figure 3: Air exposure effect on the baked niobium sample [9].

Distribution of interstitial impurities

Secondary ion mass spectrometry (SIMS) and its modification ToF-SIMS (time-of-flight detector) were widely used for studies on interstitial impurities in niobium. One of the valuable capabilities of these techniques is destructive depth profiling, which allows reconstruction of the distribution of interstitials. SIMS and especially ToF-SIMS are very sensitive with theoretically up to ppb detection capabilities but in the case of niobium samples certain limitations (e.g. preferential oxygen sputtering, roughness effect on depth resolution) often prevent from clear data acquisition.

Nevertheless several succesful studies have been recently performed.

In [10] four single grain BCP samples underwent BCP and high pressure rinsing followed by 100°C 48 hours UHV baking for two samples, and then were analyzed by ToF-SIMS. From Fig. 4- 5 no difference between baked and not baked samples is observed.

Thus no indication of interstitial oxygen or hydrogen involved in mild baking benefit is present in SIMS results.

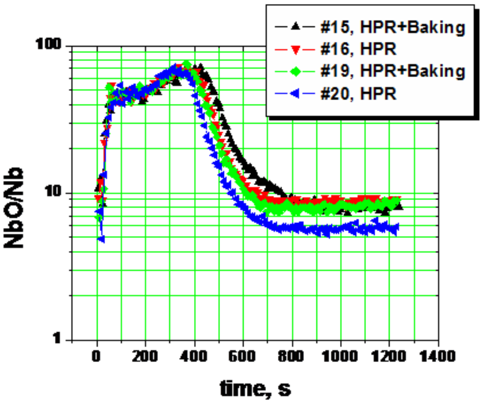

Figure 4: UHV 100°C baking effect on interstitial O distribution in single grain BCP samples [10].

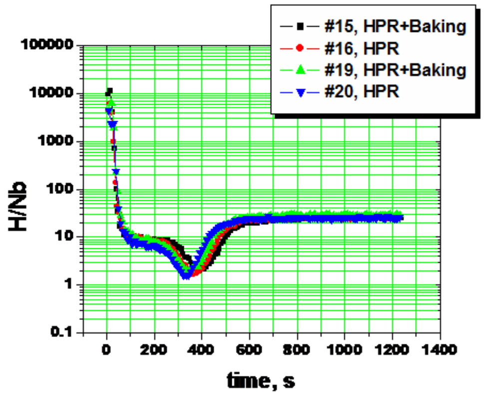

Figure 5: UHV 100°C baking effect on interstitial H distribution in single grain BCP samples [10].

Another SIMS study with different temperature and duration for UHV and air baking was reported in [11] with results shown in Fig. 6. Again as opposed to clear benefit in cavity results "fast" UHV baking did not change interstitial oxygen distribution, while air baking, which caused Q-slope degradation in cavity performance, resulted in observable oxygen loading.

To summarize, interstitial oxygen diffusion does not seem to be the cause of mild baking effect but might be the reason for Q-slope degradation after baking in air.

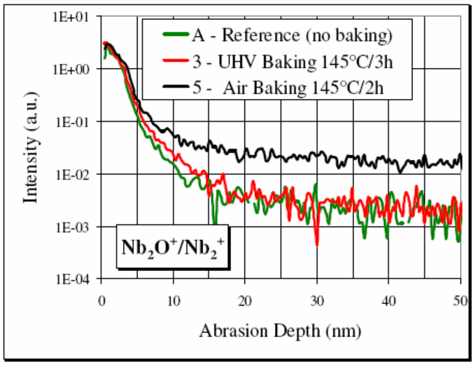

Figure 6: Change in interstitial O distribution during "fast" UHV and air baking [11].

Crystalline microstructure

Rapid development of electron back-scattered diffraction (EBSD) in recent years allowed its application to studies on niobium samples. EBSD allows obtaining crystallographic orientation map of the sample surface with information depth of about 20-100 nm from backscattered electrons diffraction patterns (Kikuchi bands). Since niobium oxide is amorphous it does not contribute to the diffraction, so that EBSD looks directly at niobium underneath the oxide within approximately magnetic field penetration depth.

For the purposes defect (vacancies, dislocations) distribution analysis, which is also directly related to lattice strain, local misorientation maps were obtained from crystallographic orientation maps. Local misorientation is an average misorientation between the pixel and its eight direct neighbors in a 2D map.

In Fig. 7 results obtained on single grain EP and BCP samples before and after UHV 110°C baking for 48 hours are presented. Clearly, differences between EP and BCP and baking-induced changes are observed.

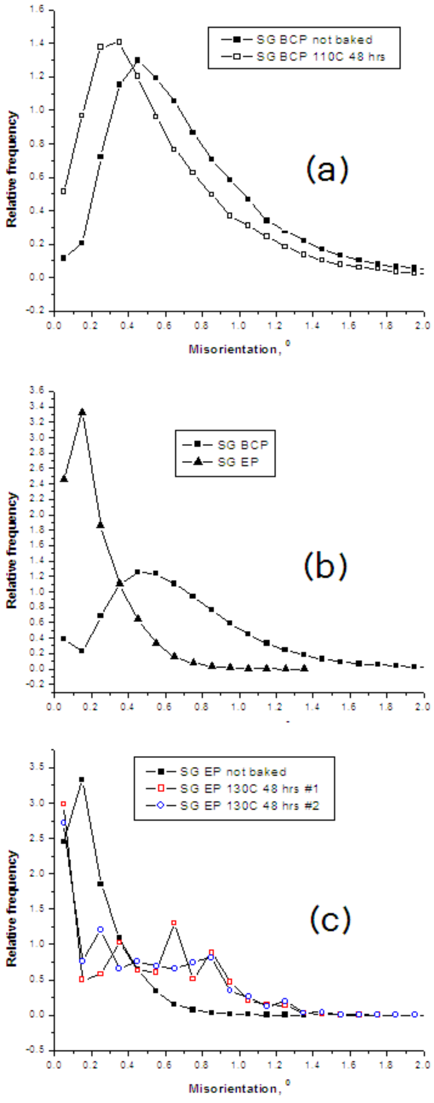

Figure 7: Local misorientation distributions for single grain niobium samples: (a) BCP baked/unbaked (b) BCP/EP (c) EP baked/unbaked. (From [10]).

In summary, change in a crystalline defect structure may be the underlying mechanism for a mild temperature baking effect.

Roughness

The role of roughness in the Q-drop was suspected in early stages due to possible magnetic field enhancement at surface morphological defects (i.e. grain boundary steps). Since roughness does not change due to mild baking it can be ruled out from the possible mechanisms for baking improvement. But observed superiority of EP over BCP after mild baking can be in part connected to different roughness produced by these treatments as shown in Fig. 8.

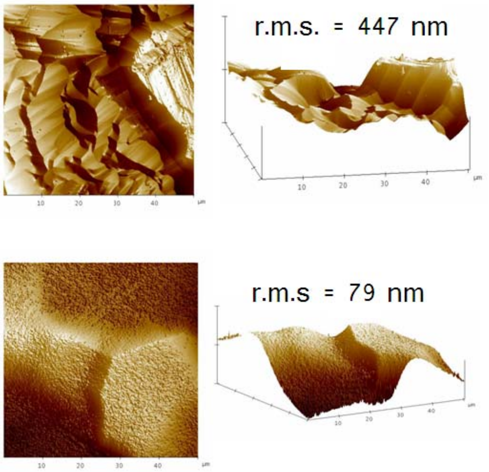

Figure 8: AFM measurements of BCP (top) and EP (bottom) roughness. Vertical scale - 3 μm (top), 0.4 μm (bottom). EP results in much smoother grain boundary steps.

Q-SLOPE HEATING NON-UNIFORMITY

Temperature mapping studies on cavities of all grain sizes [12, 13] indicate that observed losses in the high magnetic field region are distributed non-uniformly as shown in Fig. 9. One of the observed peculiarities of a large grain material is that ratio between temperature rise between "hot" and "cold" regions is several times that in the small grain material, though all regions exhibit a Q-slope behavior in the sense that losses start to increase dramatically everywhere in the high magnetic field region at about the same field level.

The question to be answered by surface studies is - *what causes observed non-uniformity of heating in the high field Q-slope regime?*

Grain boundaries

One of the reasons for patchy heating could be grain boundaries. DC magnetic flux penetration studies reported in [15] provided evidence that grain boundaries are weak regions for the magnetic flux penetration (see Fig.10) if a grain boundary plane is parallel to the applied magnetic field.

But in the case of RF fields in superconducting cavities, analysis of spatial distribution of losses indicated that "hot" regions were not primarily at grain boundaries. This conclusion was also confirmed by detailed studies in [13].

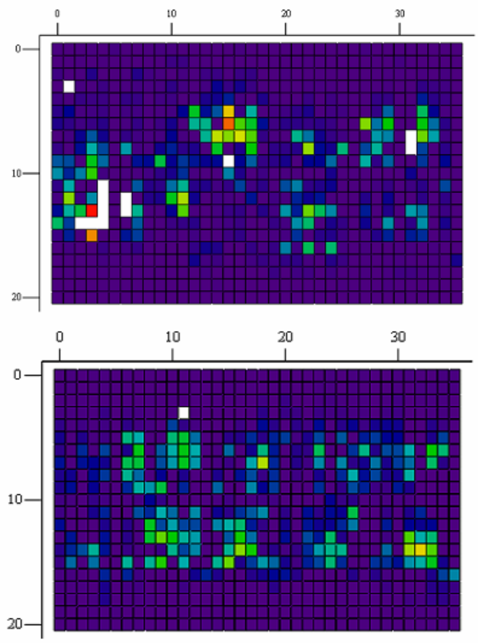

Figure 9: Temperature maps of cavity walls heating in the high field Q-slope regime. Large grain cavity - upper map, small grain cavity - lower map. [14].

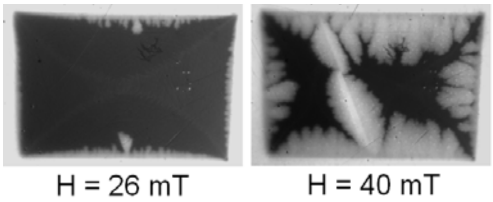

Figure 10: Magneto-optical imaging studies of DC magnetic flux penetration into niobium sample. Flux penetrates if a grain boundary plane is parallel to the magnetic field [15].

In order to investigate "hot" and "cold" spots directly, samples were dissected from regions of stronger and weaker heating and subjected to different surface analytical techniques in order to analyze possible causes of the non-uniformity. Results for small and large grain cavitites are reported in [14]. Following subsections present possible mechanisms investigated.

Roughness

Optical profilometry studies of roughness revealed no difference both in roughness on the scale of a grain size with r.m.s. roughness of about 1.5 μm, and on the macroscale of distances much larger than a grain size where roughness is dominated by grain boundary steps as shown in Fig. 11.

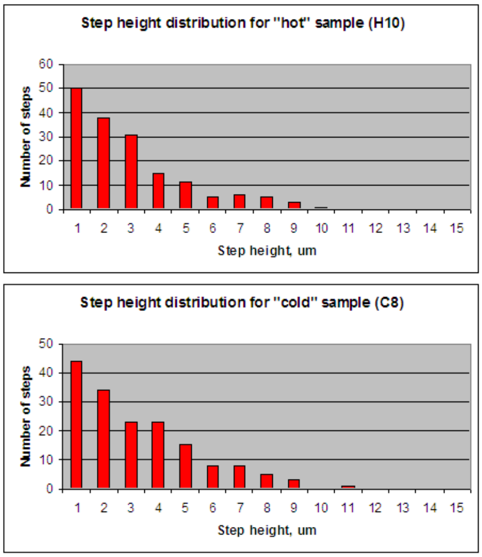

Figure 11: Step height distribution histograms for "hot" and "cold" regions.

Oxide structure and contaminants

Both laboratory XPS system and a synchrotron X-ray source at NSLS were used for the analysis of dissected samples. No difference was found in the oxide structure between "hot" and "cold" spots as shown in Fig. 12.

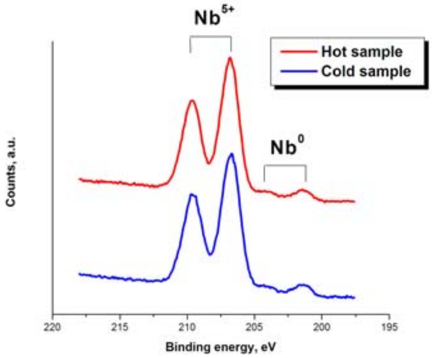

Figure 12: XPS Nb 3d spectra indicating no differences between the oxide in "hot" and "cold" regions [14].

The only difference found with XPS studies on samples cut from a small grain cavity was the presence of nitrogen at several hottest spots. In order to investigate the effect of a mild temperature baking, samples with nitrogen signal present had been baked at 110°C for 48 hours. As a result, as shown in Fig. 13, nitrogen signal was eliminated. Since the diffusion coefficient of N in bulk Nb is too small to explain N disappearence the possible mechanism is that N diffused away along grain boundaries.

On the contrary in the case of a large grain cavity no differences were observed between the samples, XPS spectra being remarkably similar for "hot" and "cold" regions. No excess N was found in the hottest spots.

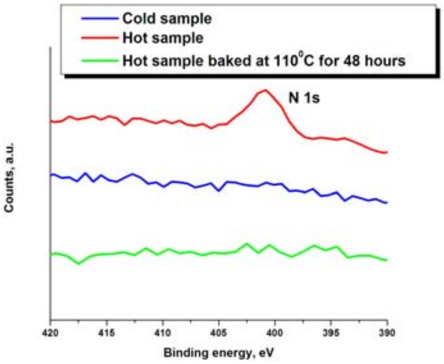

Figure 13: XPS N 1s spectra for "hot", "cold" and baked "hot" regions [14].

Crystalline orientation and microstructure

EBSD was used to analyze crystallographic orientation distributions in samples and local misorientation maps were obtained.

From the data, crystalline orientations of individual grains both in small and large grain cavities did not exhibit any preferential pattern, which might account for the heating non-uniformity, if different orientations posess different superconducting properties (e.g. critical field). Analysis based on orientation distribution functions showed that orientation was not the cause of the higher losses.

Though not all samples have been analyzed up to this time, local misorientation maps revealed the difference between "hot" and "cold" regions in a large grain cavity as shown in Fig. 14- 15.

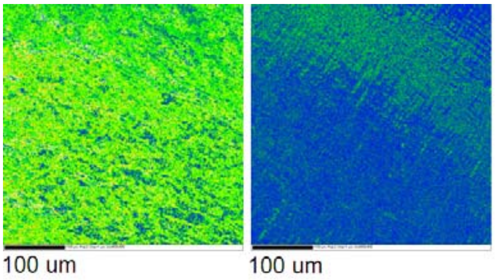

Figure 14: Local misorientation maps for "hot" (left) and "cold" (right) regions. Green color corresponds to 2° misorientation, blue - 0°.

As in the case of mild baking studies crystalline defects appear to play a role in the observed heating non-uniformity for the case of a large grain material.

DISCUSSION

Surface studies performed up to now allow us to narrow the circle of candidates for the high field Q-slope explanation.

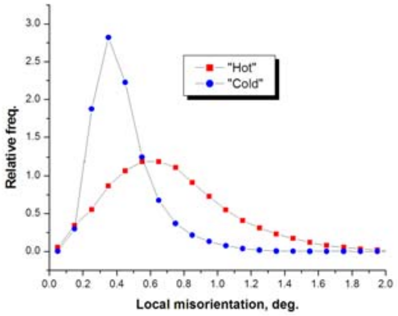

Figure 15: Local misorientation distributions as obtained from EBSD measurements.

Magnetic field enhancement at grain boundary steps is one of the proposed mechanisms for the Q-drop. Optical profilometry and AFM studies show that roughness is different for BCP and EP but RF measurements show no difference in Q vs. E behavior of BCP and EP cavities without mild baking. Furthermore analysis of "hot" and "cold" regions revealed that roughness was similar everywhere both within the grain and on the larger scale of many grain boundary steps. In summary, roughness can not be the dominant cause of the high field Q-slope but it may play a role in the full explanation of EP superiority over BCP after mild baking. Increased amounts of BCP (increased roughness) yields lower onset field for Q-slope of baked cavities [16]

Oxide structure and oxide/metal interface have long been suspected to play a role in the Q-drop. Possible mechanisms include a depressed superconducting layer of niobium suboxides (i.e. NbO, NbO_2 etc.) that become normal conducting at lower fields than bulk niobium. XPS studies give us a clear evidence that niobium oxide modifications caused by a mild baking are cancelled by the following air exposure. On the other hand, from cavity measurements it is known that air exposure does not remove a mild baking benefit and the Q-slope does not come back. Such a contradiction eliminates niobium oxide layer from the list of candidates for the mechanisms responsible for mild baking improvement. Analysis of "hot" and "cold" samples showed that oxide structure is not responsible for the Q-slope heating non-uniformity as well. Thus, oxide layer is not playing any role in the high field Q-slope.

Interstitial impurities and especially oxygen have been suggested as a possible cause of the Q-slope and baking improvement. Oxygen-enriched layer underneath the oxide with depressed superconducting properties, which is diluted by mild baking, was a suggested mechanism. Diffusion calculations show that oxygen diffusion length at the time scale of 24-48 hours is comparable to the depth of baking modified layer [2]. Nevertheless no conclusive evidence was found with SIMS and XPS that oxygen-enriched layer exists and that interstitial oxygen distribution changes due to mild baking. Thus, it is reasonable to exclude oxygen from possible causes of the high field Q-slope. Hydrogen distribution was found to be not affected by a mild temperature baking either, at least at the level of instrument sensitivity achieved. Since hydrogen in niobium is mobile even at room temperature it is unlikely that any change (if present at all) introduced by mild baking is sustained after prolonged atmospheric air exposure, which makes hydrogen an unlikely suspect too.

B_{c3} change observed in EP and BCP samples is the only change found so far in superconducting properties of niobium due to mild baking. Though it is unclear if B_{c3} is a critical field that limits achievable magnetic fields in RF cavities, it might reflect the superiority of EP cavities over BCP after mild baking. On the other hand, cavity measurements show that BCP and EP cavities perform similarly without baking whereas B_{c3} is found to be higher in EP ones.

Crystalline orientation of individual grains was introduced as a possible cause of the heating non-uniformity in the Q-drop regime. EBSD mapping of orientation distribution in "hot" and "cold" samples showed that it was not the case. Another argument to eliminate grain orientation from consideraton is the fact that some grains should be more lossy than others and the heating should be distributed uniformly over each individual grain, which is not observed on temperature maps.

EBSD measurements of a local misorientation in BCP and EP samples reveal a significantly different microstructure before baking. Mild baking results in the shift of distribution towards lower misorientation angles in BCP samples, which can be interpreted as a decrease in the intrinsic strain level, strain being caused by lattice defects such as vacancies or dislocations. In the case of EP mild baking causes a significant decrease in a lower angle misorientation density with a slight increase in the higher angle misorientation density. The full interpretation of that is not clear at this point. Local misorientation distributions for "hot" and "cold" regions are also different suggesting that crystal defect structure might be responsible for observed heating patterns.

Summarizing, among low scale mechanisms that were investigated by surface analytical techniques crystalline microstructure is the only one that exhibits changes due to mild baking, which are preserved after air exposure, and is connected to the heating non-uniformity.

CONCLUSIONS

Recent surface studies results exclude niobium oxide layer, interstitial oxygen, grain boundaries, grain crystalline orientation and roughness from the list of possible candidates for the explanation of a Q-drop origin and the observed heating non-uniformity. Crystalline microstructure and its role in the high field Q-slope is a promising direction for further investigations.

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