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Signs of plastic response in surfaces exposed to high electric fields.

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Cern: Lee Miler, Victoria Bjelland, Catarina Serafim, Sergio Calatroni, Walter Wuensch Uppsala: Iaroslava Profatilova, Marek Jacewicz



Conditioned samples – no observed local emitters

Copper electrodes are manufactured to create extremely uniform structures. Originally used to ensure high conductivity.





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Microscopy of BD events

Breakdowns lead to large-scale cathode material deposition: craters + sub-surface plastic activity.

Craters in two type of electrodes: different late-stage evolution but in both cases – melted cathode material ejected by plasma pressure.

The remnants of these violent events do not explicitly tell us what preceded them...

However, we see that events are NOT correlated with GB or other large-scale features.

(Catarina – not even blisters)





Intrinsic + Extrinsic events are clearly identifiable



V curr WD use case det mode HFW 500 5.00 kV 0.20 m² ⁶ 2² ⁹ ² ⁸ 863 mm Standard ETD^{Yino} ^{SE Ashkenaz} ² ¹ ⁹ ⁶ ^{ah} ^{Inst.} Of Physics – Hebrew University of Jeusalem

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Even better at close-ups. But the main point is that they do not control long-term "saturation" behavior

Copper

Tungsten (contamination)



When zooming in - conditioned surfaces contain sharp terraces, and even precipitates



So we asume this not a continuous surface evolution process. And conditioning is not purely "cleaning"





- Intrinsic events. Rare and do not demonstrate collapse on global scale.
- So searching for a fluctuating property related to a critical transition driven by the field.
- We search for constraints on plastic processes leading to such a transition. Assuming that the collective evolution of the mobile dislocation population may trigger this critical transition through surface modification



Dislocations are known to create persistent slip bands and protrusions

- Dislocations do demonstrate stress driven collective evolution leading to surface features, at sub-yield stresses. Not deterministic.
- PSBs exist in various scales, usually observed via SEM at micron level
- But: lower stress, no evident surface evolution (PSBs are stable).





Laurent et.al. Phys Rev STAB 14 (2011) 41001



Haël Mughrabi Phil. Trans. R. Soc. A 2015

Dynamic transition – Fluctuations in the density of moving dislocations

• A large number of sources and sinks for dislocations is available (verified by direct observations)

Governing equations:

- Increase in mobile population interactions with field and moving dislocations.
- Arrest due to collisions
- Cooperative critical transition in mobile dislocation population generates nucleation event.

We proposed that this transition - start of a runaway in the mobile population can lead to a nucleation event through its effects on the surface

PHYSICAL REVIEW LETTERS 120, 124801 (2018)

Stochastic Model of Breakdown Nucleation under Intense Electric Fields





Model results vs observations -Rate variation with field

Consistent fit to breakdown rate dependence on field Importance of temperature variation.

• "Classical" scenario: Temperature effect on BDR versus field curves. Dynamic - Ramping up field at various rates. Average "field for BD"

Time dependency - Non-Linear regime.

prior to BD (as field is increased) fluctuations in the population - and the dark current should be observed! (See L. Miler talk - Monday)

PHYSICAL REVIEW ACCELERATORS AND BEAMS 22, 083501 (2019)

Editors' Suggestion

Theory of electric field breakdown nucleation due to mobile dislocations



BDR (bpp/m)

BDR (bpp/m)

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Fluctuations - offer a possible explanation to spikes in dark currents (prior to BD)

• Event rate variation with the field is reproduced by MDDF based equation.

$$\lambda_0 = \frac{25\kappa C_t c}{G^2 b \Delta \rho} \sigma_0^2 \exp\left(-\frac{E_a - \Omega \sigma_0}{k_B T}\right)$$

• The distribution fits the expected hypoexponential distribution.







PHYSICAL REVIEW ACCELERATORS AND BEAMS 23, 123501 (2020)

Dark current spikes as an indicator of mobile dislocation dynamics under intense dc electric fields

Eliyahu Zvi Engelberg[®],¹ Jan Paszkiewicz[®],² Ruth Peacock,² Sagy Lachmann,¹ Yinon Ashkenazy,^{1,*} and Walter Wuensch[®]²

What might be expected to be seen in microscopy?

- Transition is due to an avalanche of moving dislocations
- Before critical transition dislocations move but at low numbers.

If true, then:

- Conditioning due to dislocation motion affecting the subsurface region in terms relevant to dislocation motion and interaction.
- The mechanism depends on surface interaction dictating a preferred direction. So, the effect is limited to the top submicron region.
- We should be able to observe a large density of mobile dislocation sources.

Cross sectional

Observe an Intricate dislocation pattern below the surface that correlates well with the surface structure.

Sadly a result of sample preparation.





march 6 2024

FIB damage of Cu and possible consequences for miniaturized mechanical tests

D. Kiener^{a,b,*}, C. Motz^b, M. Rester^b, M. Jenko^c, G. Dehm^{b,d}

D. Kiener et al. / Materials Science and Engineering A 459 (2007) 262–272

Fig. 2. (a) Dark-field TEM image of a Cu grain situated at the border of an ion damaged and undamaged region. The right part of the grain was exposed to Ga⁺ ions with a kinetic energy of 30 keV and an ion current of 50 pA. The g_{-220} diffraction condition is shown in the inset. (b) Bright-field TEM micrograph of the damage pattern of a grain bombarded by 5 keV Ga⁺ ions and an ion current of 100 pA. The g_{-11-1} diffraction condition is shown in the inset.

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Fig. 3. Bright-field TEM images of Cu grains exposed to perpendicular 30 keV Ga⁺ ions and an ion current of 50 pA under different diffraction conditions. The diffraction vector is shown for the grain on the right hand side. The strain contrast vanishes for certain conditions, as can be seen for several grains (compare a and b).

 Perpendicular, high voltage, high current – lead to FIC generated dislocation structures in all grains (observable in different diffraction directions)

So what can we do

4. Discussion

In order to reduce the damage introduced by the Ga⁺ ion bombardment, several possibilities exist. For example, use of a protective layer, blow incidence angle of the impinging Ga⁺ ions, blow ion energies, and boptimized milling geometries in order to avoid redeposition. A further possibility to reduce the

Completely covered? Almost... but not enough. Still in large grains of Cu – FIB damage prevent observing small scale structures After taking all precautions, soft Cu continues to show damage from FIB sample production - dislocations + loops.



Even in hard Cu - high concentration of artifacts







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But-large features are not affected





Within the current limitaitons

- We can use cross sectional FIB samples to study large scale dislocation related structures: grain boundaries, twins etc but not single loops or dislocations.
- So we compare conditioned to unconditioned regions . Using Cu tested at 30K and 300 K

Details : see – Marek Jacewicz & Iaroslava Profatilova etal. http://arxiv.org/abs/2403.03198

Cross-sectional STEM: Show regular, continuous structure up to the surface





http://arxiv.org/abs/2403.03198



The top 100 nm of conditioned regions are significantly modified also @30K



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Not all denuded zones are conditioned, and not all "un-denuded" (dislocation rich) are not-conditioned



But most are

Reference 007 – Cu 300K

🛏 100 nm

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Field exposed 007 –Cu 300K

🕂 100 nm



Cryo-treated regions – have various types of grain refinement but denuded zone formation is consistent





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Summary and Outlook

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- Pursuing a direct link between plastic mechanism and BD nucleation:
 - A model offering rare critical transition consistent with no observable localized emitter
 - Consistent description of observed variation of BD rates.
 - Consistent results for critical rare fluctuations in dark currents.
- Microscopy
 - It is a tricky business as sample manufacturing creates significant "red herrings."
 - However, larger-scale structures are not affected.
 - Looking for indication of significant dislocation activity at sub-critical levels.
- First indications of dislocation mediated variation between conditioned and unconditioned regions:
 - Formation of denuded zones in hard Cu surfaces.
 - Effect limited to top 100 nm.
 - Need to do comparative forensics more samples, sloped geometry, hope for results soon.
 - In parallel improved forensic methods.