Application Note for microsphere imaging.

Graphene Aging

LIGNANOWISE

NANORO

Graphene Imaging

What?

Since the Nobel prize was awarded for its discovery in 2010, the use of graphene within the scientific community has been extensive and wide-ranging. At just a single atom thick, graphene is the strongest, and thinnest, material known to man. The two-dimensional carbon array is made up of a network of strong covalent bonds, whilst free electrons delocalised from each carbon atom make the sheet highly conductive. The composition of these properties makes graphene useful to many industries, including electronics manufacturing and energy. Such heated interest has led to the synthesis and manipulation of graphene sheets to be an active area of research, with close attention paid to detail on a nanometre scale.

Why?

As graphene grown via chemical vapour deposition (CVD) ages, oxidation of the underlying substrate (often copper) that the graphene is deposited onto can reveal faults in the graphene membrane. These faults occur at the “grain boundary”; where two nucleation sites grow into each other. The way in which these boundaries form is an area of intense research, as a smooth mesh between sites produces a more uniform atomic layer. Using our Super-resolution Microsphere Amplifying Lenses (SMAL) along with other optical techniques, purposely defective samples provided by the Graphene Engineering Innovation Centre (GEIC) were imaged to study this oxidation. The extent of the oxidation can reveal the porosity of the graphene membrane at the the grain boundary, providing insight into the manufacturing and aging process.

“This work has shown the promise of using the Nanoro system to analyse graphene materials. We will continue to work together moving forward”
Dr. Andrew J. Strudwick
Graphene Engineering Innovation Centre
The University of Manchester

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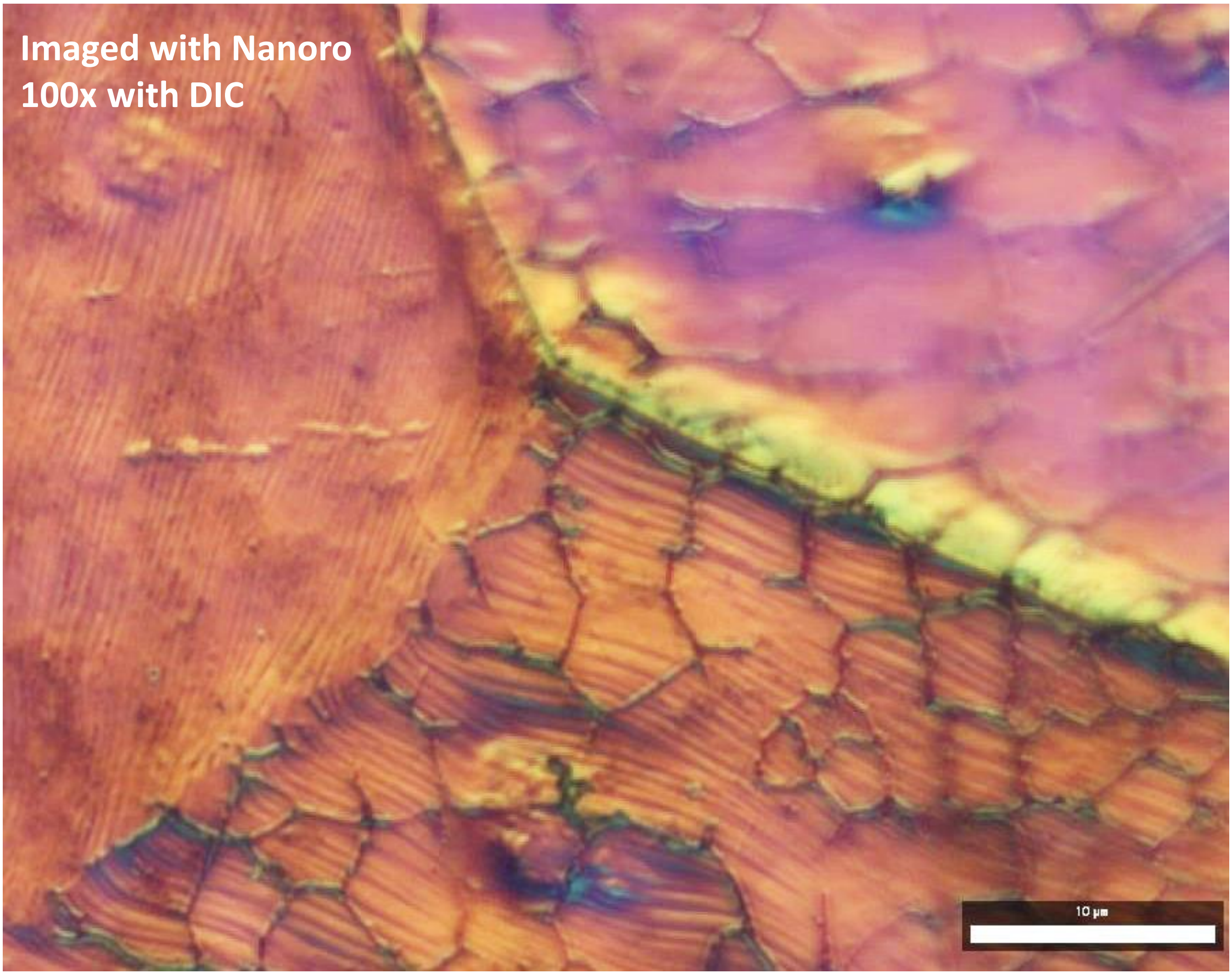
The University of Manchester

Differential Interference Contrast (DIC)

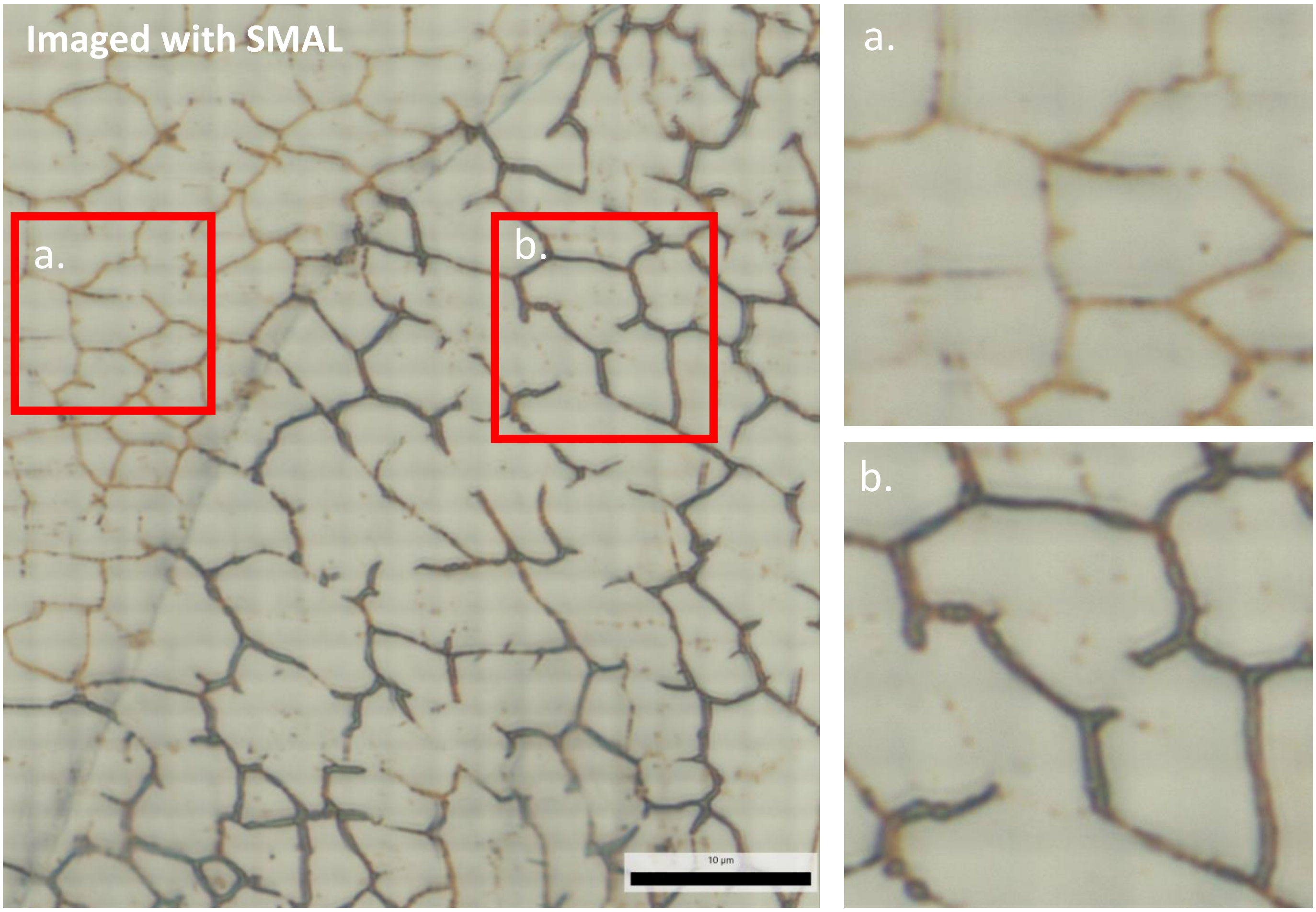
Differential Interference Contrast (DIC) was added to Nanoro to help investigate features of the graphene samples. By separating different components of the light source's polarisation, different colours are enhanced depending on differences in the reflectivity of each part of a sample. The outcome is an increased contrast between different materials in the sample, making it easy to see the grain boundaries in graphene deposited on copper.

The image below captured the interface between three crystalline phases of copper. The way that graphene has aged on each of these phases is different, evidence of this difference was captured and shown below. The dark lines visible in two of the three domains likely represent oxidation of copper at grain boundaries. Change in domain size and the boundary porosity across the different crystal domains can be easily observed by the change in the size of the regions outlined by black lines, and the opacity and width of the lines themselves.

This work has prompted an investigation into the effect aging has on the nucleation and propagation of the observed defects believed to be from oxidation. LIG Nanowise continue to work with the GEIC to develop understanding of the properties of aging graphene sheets, imaging features made clearer than ever before by the implementation of DIC on Nanoro.



Imaging Oxidation Aging SMAL



The use of SMAL enabled a closer study on the difference in the apparent oxidation of copper at grain boundaries across different crystal structures of copper.

The increased resolution offered by SMAL allows detail top be seen within the grain boundaries, and a further inspection of the extent of the discoloration. SMAL reveals that the black outlines observed in "b." are in fact, surrounded by regions of the orange discolouration observed in box a.

The two boxes represent graphene deposited onto two different crystal domains of copper. The evidence uncovered using SMAL technology suggests perhaps that the oxidation occur over different timescales, dependent on the underlying crystal structure of the substrate.

The source of this changing oxidation rate could be due to several factors. Further investigation of the oxidation process of copper substrates through graphene monolayers is still ongoing at the GEIC. Images captured using SMAL on Nanoro continue to be a key contributor to this research.

LIG Nanowise would like to thank the GEIC for not only supplying the samples to make this project possible, but also their continued support and guidance throughout this collaboration.

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