ESRF	**Experiment title:** Nanotomography study of individual gyroids and domain textures in multi-hierarchical structures of butterflies for bioinspired photonics	**Experiment number**: SC-5228
Beamline: ID16A	**Date of experiment**: from: 27/04/2022 to: 02/05/2022	**Date of report**: 25/09/2023
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Background:

Biological systems evolved unique ways to assemble multi-hierarchical structures with functions ranging from sensing to light manipulation [1]. These structures have been tuned by evolution for efficiency and error tolerance, and exhibit self-organizing, reaction-diffusion, and pattern-forming behavior yet not fully understood. The properties of these structures are of great interest, and attempts have been made to replicate them for such applications as energy harvesting [2], optical switches [3], magneto-optical isolators [3], photocatalysts [4], energy storage [5] and other technologies with large societal impact.

To harvest the full potential of the bio-inspired structures it is important to understand the underlying processes of their formation in biological systems. The butterflies stand out among other species for the diversity of structure types that they exhibit, starting with ridges, honeycombs, and lamellae, and culminating with structures as exotic as gyroid bio-photonic crystals [1]. The structures also form in different ways: as isolated gyroidal superstructures [6] with domain orientations guided by the smooth endoplasmic reticulum membrane and as domains that nucleate from random locations [7] and merge generating domain boundaries and dislocation-like topological defects.

Recently we performed imaging of synthetic diamond morphologies [8] and observed emergent topological defects that in our current understanding form as the balance of competition between multiple domains and the surface energy. Moreover, it has been shown [9] that the topological defects recently observed in mollusc nacre facilitate the long-range structure synchronisation by absorbing other defects. We hypothesize that emerging topological textures play an important role in highly organized photonic structures of butterflies.

Thus, we propose to use X-ray nano tomography to understand the role of topological defects in multi-hierarchical structures of butterflies. We will use acquired high resolution volumetric data to: (1) to study the mechanism of their formation; and (2) to study photonic properties of individual topological defects.

Experiments and Setup at ID16A:

The ID16A beamline provides a unique opportunity for multiscale bioimaging due to its small beam size (down to 25nm), high energy of the X-rays (17.1 keV used in this experiment), and cryogenic sample environment that allows to mediate radiation damage induced by X-rays. The beamline also allows to perform phase contrast imaging with two powerful techniques: holographic X-ray imaging (HXI) and near-field X-ray ptychography (NFP). In the current experiment, we achieved an important milestone for the high-resolution X-ray imaging of biophotonic structures by investigating the advantages of the two techniques and their limitations. We also explored approaches to enhance the contrast by sputtering the photonic structures of butterflies with a thin layer of gold (~20nm). We have successfully imaged photonic structures in *Thecla opisena* (butterfly) and *Eupholus Schoenherri Petiti* (beetle). Both samples exhibit nanoscale photonic structures yet show different responses to the high dose of the X-rays. The beetle sample is naturally less radiation sensitive as its photonic structures are part of the protective layer with higher mechanical strength compared to the lightweight butterfly scales. Performing imaging experiments on the beetle sample allowed us to optimize the acquisition parameters, compare imaging techniques, and obtain high-resolution data on nanoscale photonic networks confined by both top and bottom surfaces. The outcome of this methodological part of the experiment was to optimize sample preparation, push the pixel size down to 3nm, establish the reconstruction pipeline, and develop domain orientation analysis tools.

Results:

The images presented in Figure 1 demonstrate our preliminary analysis of the obtained data. We have shown that NFP at the ID16A beamline can be pushed down to 3nm pixel size, a resolution that is not immediately available for HXI technique. We have also established an approach of extended field-of-view NFP scanning where areas as large as 300x300 μm^2 can be explored with pixel size of 10 nm within 1 hour acquisition time and simultaneously reconstructed and stitched into multiscale image. This approach was instrumental in surveying multiple butterfly scales (see Figure1D) for the identification of signatures of topological defects (see Figure 1E). Finally, we have acquired several nanoscale tomographic datasets and are currently in the process of analysing the observed topological textures with respect to their role on the domain formation and photonic properties that they carry.

References:

1. M. I. Osotsi et al. National Science Review 8, nwaa107 (2021);
2. R. H. Siddique et al. Sci. Adv. 3, e1700232 (2017);
3. W. Peng et al. Adv. Funct. Mater. 22, 2072-2080 (2012);
4. J. Fang et al. ACS Appl. Mater. Interfaces 10, 19649–19655 (2018);
5. X. Guo et al. Ceramics International 44, 9696-9702 (2018);
6. B. Wilts et al. Sci. Adv. 3, e1603119 (2017);
7. A. Singer et al. Sci. Adv. 2, e1600149 (2016);
8. D. Karpov et al. arXiv preprint https://doi.org/10.48550/arXiv.2304.14819 [in review];
9. M. Beliaev et al. Nature Physics 17, 410–415 (2021).

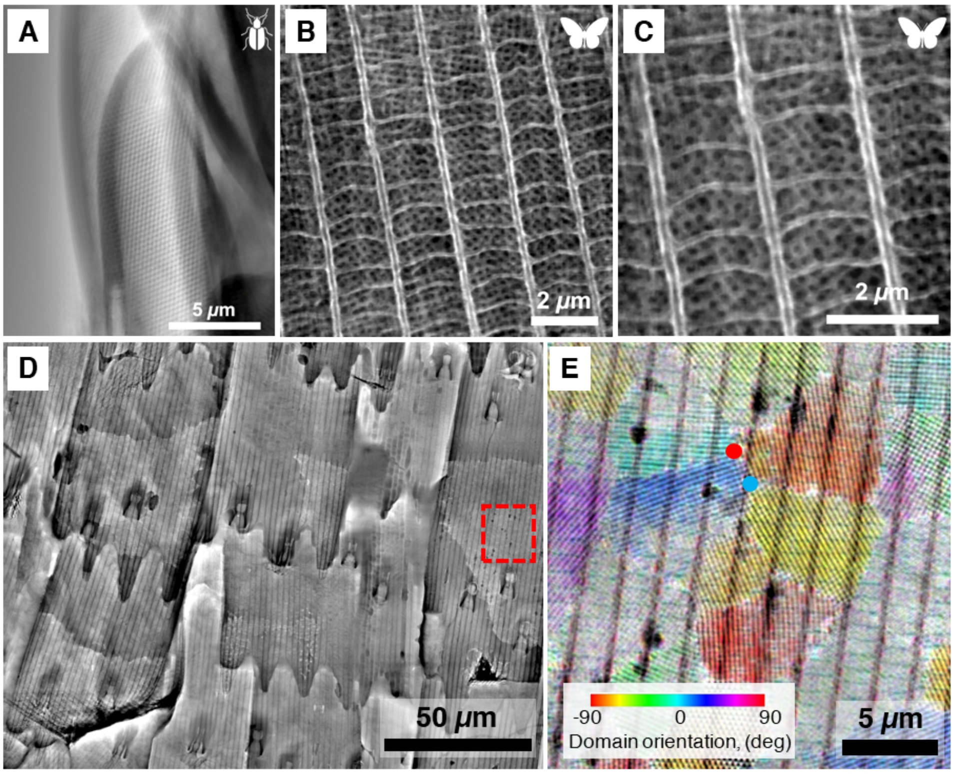

Figure 1: **A**, NFP projection of photonic structures in beetle scale at 10 nm pixel size. **B**, NFP projection of photonic structures in butterfly scale at 5 nm. C, NFP projection of photonic structures in butterfly scale at 3nm pixel size. **D**, extended field of view NFP projection of multiple butterfly scales acquired with 10nm pixel size. **E**, region of the butterfly scale marked with red square in **D** with domain orientation analyzed and topological defects identified.