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A novel automated method for structural characterization in material science with an Ultramicrotome

- With an example of pyrolyzed origami structure of cellulose paper

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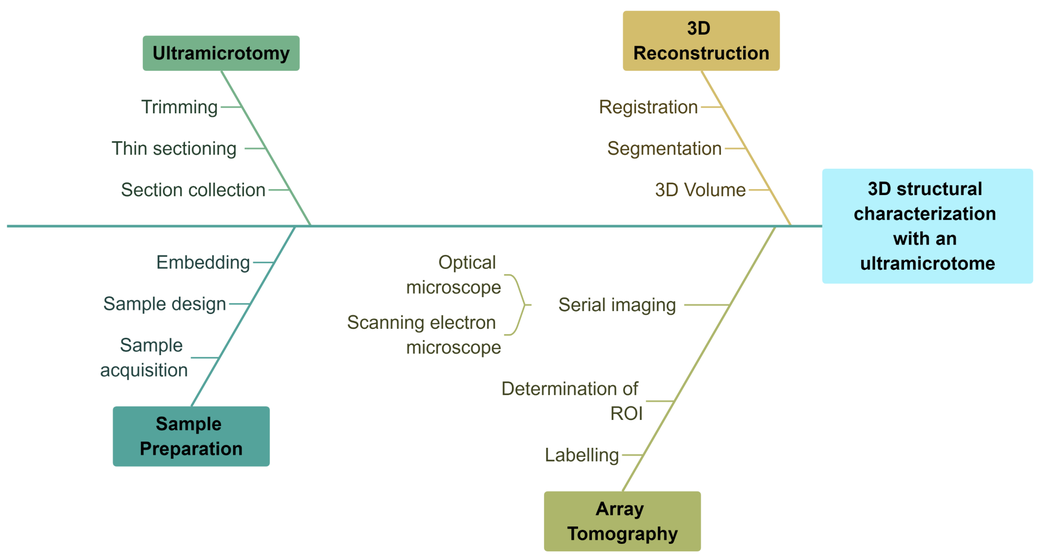
Main process steps



Introduction

Ultramicrotome sectioning is widely applied in the field of (bio-)medical science for decades. It allows cutting of sample sections with thicknesses from 200 nm - 30 nm and enables imaging down to the nanoscale.

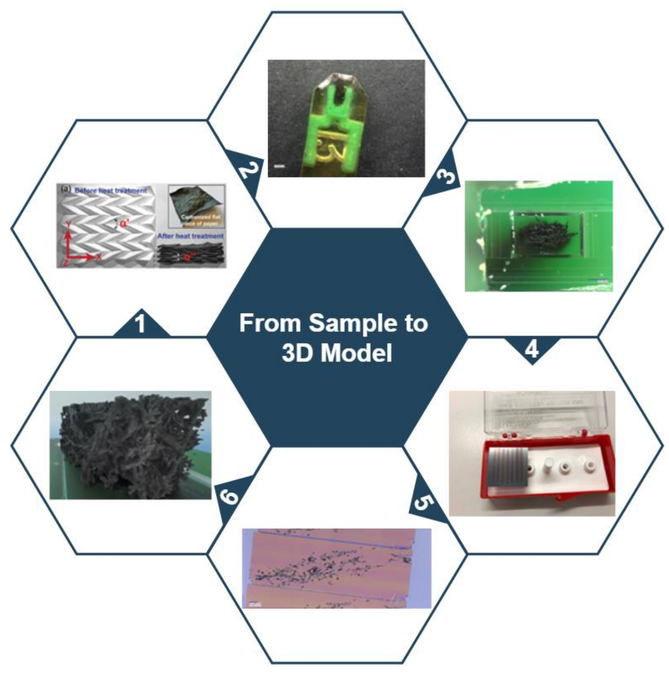
Hence, this tool is introduced to the material science community and wins a place for structural characterization of new materials.

Nevertheless, there remain challenges to apply this tool to novel materials and to optimize the preparation and sectioning process to achieve a good section quality.

Case study

Carbon origami has been recently reported to enable the fabrication of lightweight, 3D and architectured shapes of carbon materials using renewable cellulose paper as the carbon precursor [1]. Lack of insight into the 3D-micro-/nanostructure of carbon origami still impedes further fine-tuning of its properties.

In this work, carbon origami(1) is embedded into a commercial epoxide resin (Spurr, firm)(2). The resulting sample block is manually pre-trimmed. With the ultramicrotome, the block face is precisely trimmed into a rectangular shape with a size of 1045 x 400 µm, to make the embedded origami structure accessible for sectioning(3). By means of an in-house built motorized manipulator, a total of 1045 sections with a thickness of 200 nm are continuously cut and collected on three wafers(4). The section series is subsequently imaged with an optical microscope (Zeiss Axio Imager with Zen Software)(5). The obtained image stack is further processed with Fiji and Chimera to finally reconstruct a 3D model(6).



Numbers refer to text.

Highlights of this work:

- Process automation for ultramicrotome cutting and section collection to achieve significantly larger sample volumes for 3D reconstruction
- Validation of the potential of ultramicrotomy as a method for characterization in material science
- Step from 2D to 3D structural characterization for this class of carbon materials - to investigate structure-property relationships in information guided material design

[1] Islam M, Martinez-Duarte R. Tuning the mechanical stiffness of lightweight carbon origami[J]. Materials Today: Proceedings, 2020.

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