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## **3D investigation of a PS/ABS polymer using different microscopy techniques**

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### RéCaMiA Network

ReCaMiA is a regional Microscopists Network (Région Rhone Auvergne and Alpes) of the CNRS. **Technics available are :** SEM & TEM, Near Field Microscopy, Confocal Microscopy...  
**Interdisciplinary and sharing of experience :** Materials Science, Chemistry, Biology...

**Missions :**

- 🕒 Inventory microscopy equipment's and competences in the region
- 🕒 Take advantage of know-how diversity to develop mutual aid between the network's members
- 🕒 Facilitate contacts in microscopy community of the region
- 🕒 Organize structuring actions : Workshops to share scientific and technological knowledge, joint work on new technologies...
- 🕒 Participate to the practical training of new microscopists
- 🕒 Technology and competences watch group

### Context of the study

3-dimensional (3D) observation of nanostructured soft materials such as polymers is still challenging especially for organic-organic multiphase samples where no specific contrast is created in electron microscopy.

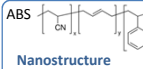
In this context, based on a control sample (a multiscale polymer blend), we present here a comparative study on the different 3D characterization technics available through the French Récamia network (FIB/SEM, TEM, SEM).

The obtained volumes are quantitatively compared and the main advantages, artifacts and limitations of each technic. are discussed.

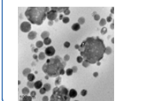
### Multiscale control sample

Recycled Polystyrene (PS)-Acrylonitrile Butadiene Styrene copolymer (ABS) blend.

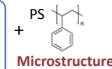
ABS



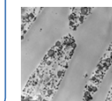
Nanostructure



PS



Microstructure



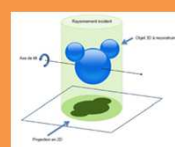
Staining of the Butadiene phase with OsO<sub>4</sub>

→ Stiffening of the material: microtomy and FIB milling at room temperature

## Experiments

### Electron tomography

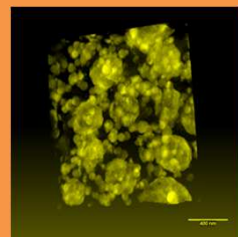
**Principle of electron tomography**



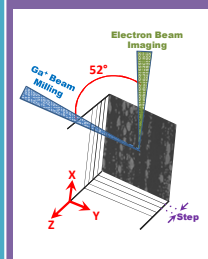
**Tomograms acquisition :**

	Sample thickness	Angular range
TEM JEOL 1400 120 kV	100 nm	-60° to +60° Step 1°
TEM JEOL 2100F 200 kV	400 nm	-60° to +60° Step 1°

**3D reconstruction and image processing with FIJI**

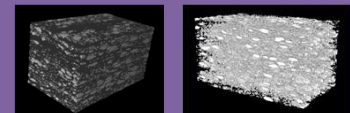


### FIB-SEM Slice and view




**FIB preparation :**  
Sample is gold coated once  
3D box is then prepared  
Sample is Au-coated again

	Analyte Volume	Voxel Size	Imaging Parameters	Imaging Time
FEI Helios 600i	8 x 5 x 0,5 µm <sup>3</sup>	5 x 5 x 6 nm <sup>3</sup>	TLD (SE), 2kV, 340 pA, 0,3µs	3h
ZEISS NVision 40	9 x 5 x 6 µm <sup>3</sup>	5 x 5 x 25 nm <sup>3</sup>	EsB (Grid 800V), 1,5kV	6h



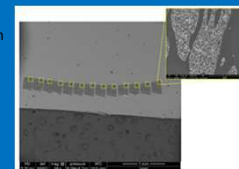
### Serial Section SEM



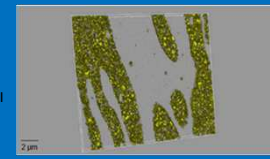
Serial sectioning of sample by ultramicrotomy - Thickness = 100 nm

Images acquisitions with FE-SEM - HT = 5 kV BSE Detector

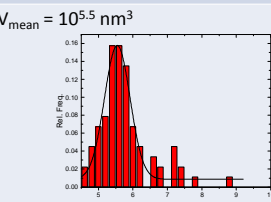
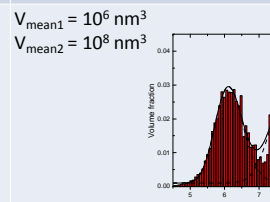
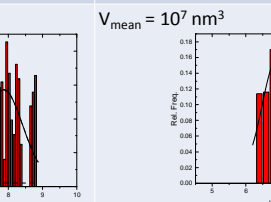
Serial sections collected on glass - Slide, and carbon coated.



**3D reconstruction and image processing with FIJI**



## Comparison and Discussion

	Electron tomography	FIB-SEM	Serial section SEM
Number of detected particles	89	12000	465
Volume fraction	30%	21%	20%
Degree of anisotropy <sup>a</sup>	NA	0.78	0.68
Particle volume distribution	$V_{\text{mean}} = 10^{5.5} \text{ nm}^3$ 	$V_{\text{mean1}} = 10^6 \text{ nm}^3$ $V_{\text{mean2}} = 10^8 \text{ nm}^3$ 	$V_{\text{mean}} = 10^7 \text{ nm}^3$ 

<sup>a</sup>Calculated from the "mean intercept length" in the volume

## Conclusion

Technics	Advantages	Drawbacks
<b>Electron tomography</b>	Acquisition is automatized Very good resolution in x, y and z, which allows to obtain good information's about morphology (size, shape, porosity...)	Only a small portion of volume is reconstructed which is not representative of the whole sample Sample could be modified during acquisition Reconstruction artefacts due to missing wedge make image analysis difficult (problems for segmentation) Particles larger than the section thickness cannot be reconstructed entirely.
<b>FIB-SEM</b>	Good contrast, and resolution Acquisition is automatized Large volume (x, y, z)	Sample could be modified during acquisition
<b>Serial section SEM</b>	Good visualization of particles distribution at large scale Contrast, resolution (single image)	Cut (ribbon) presents difficulties , Heterogeneous sections, Acquisition is not automatized Poor resolution in z Px size in x ant y is too large compared to particles size. Small objects cannot be detected correctly Px size in z (= section thickness) is much too large: not possible to obtain a good representation of the real particles shapes → Sources of errors in image analysis