EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal: *https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do*

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round **- 5th March**
- 10th September Proposal Round **- 13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.

- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

<table>
<tr><td>ESRF</td><td>Experiment title:
SAXS/WAXS for monitoring structure evolution in ultranarrow alkylamine-coated ZnS nanoparticle suspensions</td><td>Experiment number:
MA-5315</td></tr>
<tr><td>Beamline:
ID 02</td><td>Date of experiment:
from: 24/04/2022 to: 30/04/2022</td><td rowspan="2">Date of report:

Received at ESRF:</td></tr>
<tr><td>Shifts:
9</td><td>Local contact(s):
William Chevremont</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
Prof. Yuval Golan*[1]
Dr. Sofiya Kolusheva*[1]
Naama Gatenio*[1]
1. Dept. of Materials Engineering, and Ilse Katz Institute for Nanoscale Science and Technology, Ben-Gurion University of the Negev, Beer-Sheva, Israel.</td></tr>
</table>

Scientific background:

Zinc sulfide (ZnS) is a direct compound semiconductor that has a wide bandgap of 3.91 eV, a high index of refraction and high transmittance in the visible range. ZnS nanoparticles are useful as photo-catalysts and devices such as fluorescent displays, electroluminescent devices, light-emitting diodes, infrared windows, lasers and sensors. We synthesize in the lab highly uniform, ultranarrow ZnS nanowires and nanorods using zinc-ethylxenthate precursor molecule and octadecylamine as a surfactant and solvent [1]. TEM characterization of dried suspensions showed that the particles form ordered arrays with a typical particle-particle distance of a few nanometers (Figure 1).

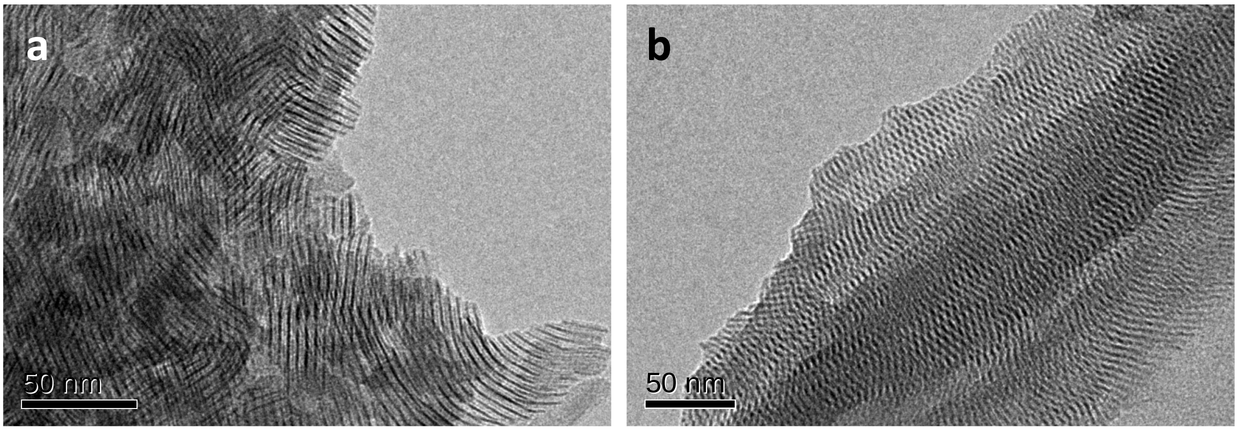

Figure 1. Bright field TEM images of (dried) ZnS nanoparticle assemblies (a) Nanowires (b) Nanorods.

Photoluminescence results of ZnS nanowires and nanorods in chloroform suspension show that the emission increases when decreasing the concentration, which is not an obvious result (Figure 2). Therfore, it is necessary to reveal the structure of these assemblies in the suspenion with various concentrations for understanding their optical properties.

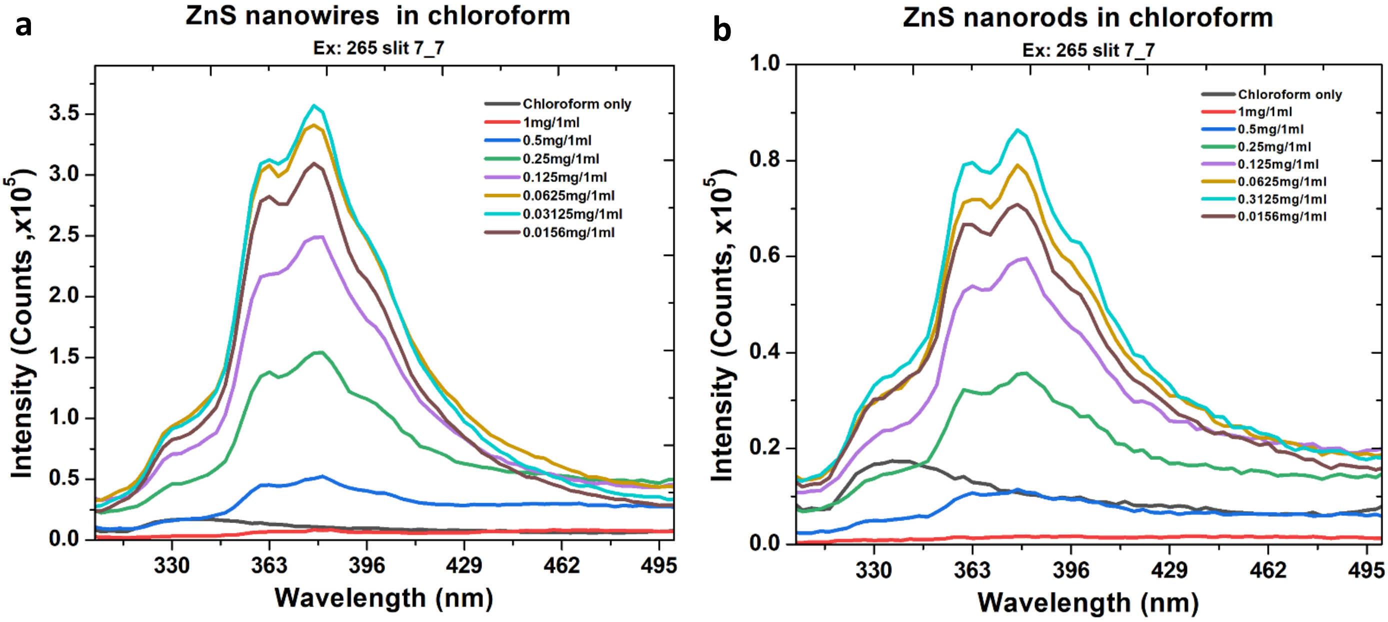

Figure 2. Photoluminescence results of ZnS (a) nanowires and (b) nanorods in chloroform suspension.

Results:

Our main goal is to investigate the structure evolution in suspensions of the highly uniform ZnS nanowires and nanorods in different various conditios: concentrations, temperatures and solvents for suspensions. As a part of a previous experiment, MX-2333, using SAXS at BM-29 we measured the ZnS nanowires and nanorods suspesions in different concentratios. For both nanowires and nanorods the results indicates a liquid crystal behaviour (Figures 3 and 4, respectively).

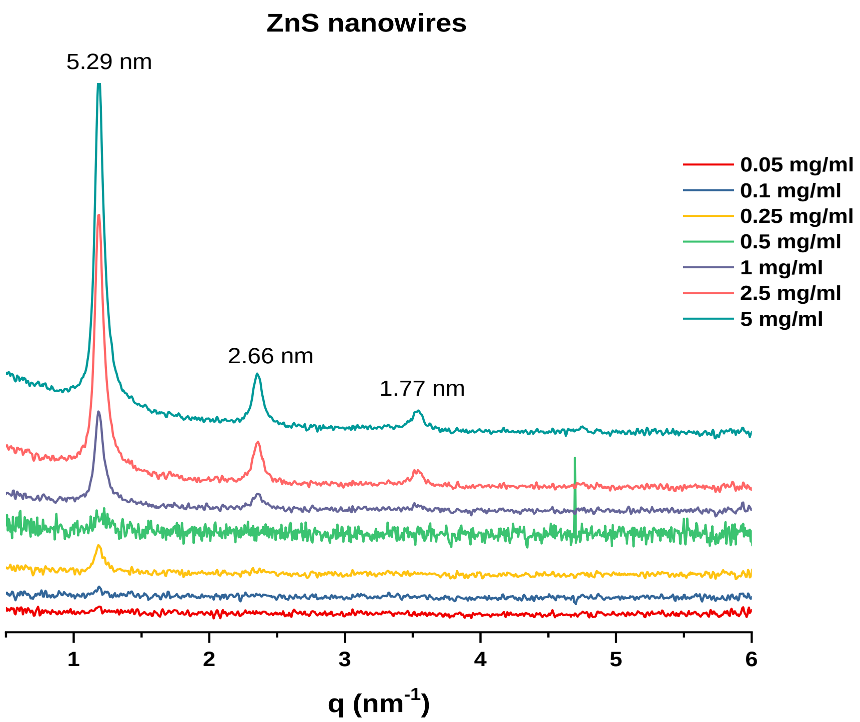

Figure 3. SAXS results of ZnS nanowires in chloroform suspension with different concentrations.

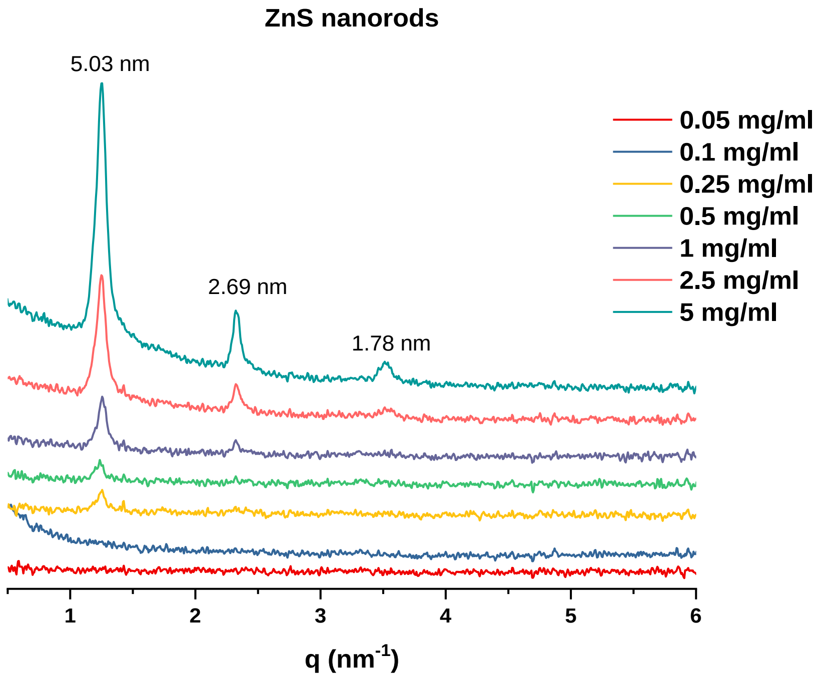

Figure 4. SAXS results of ZnS nanorods in chloroform suspension with different concentrations.

In this experiment at ID-02, we measured using SAXS the ZnS nanowires and nanorods suspesions at concentration of 5mg/ml under different magnitudes of applied magnetic field, starting from 10^{-4}T to 1.6T, aiming to see the effect of magnetic field on nanoparticle alignment. Figure 5a represents the 1d form of the norm image, Figure 5c, at a specific q value that was determined by the azimuthal image (not shown), obtained from the SAXS Utilities2 program. Using a special code, written by Dr William Chevremont, the image was translated into a txt file and analyzed as a 1d graph, to quantify the extent of alignment. From the norm images (Figure 5c,d) it is quite hard to point out a substantial change in alignment, and when comparing the 10^{-4}T curve to the higher magnetic fields curves (Figure 5a,b) there is only a weak effect for the chloroform suspensions studied.

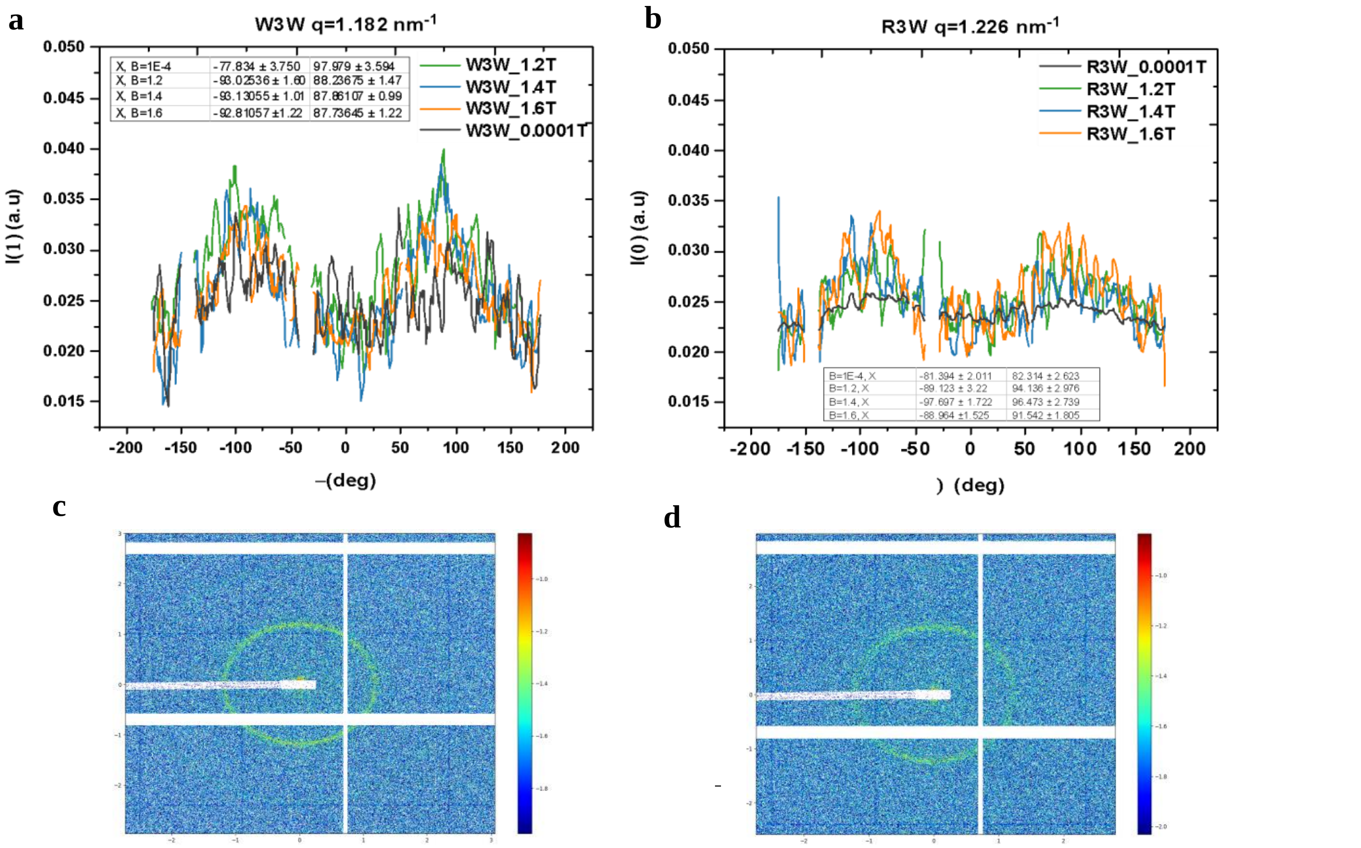

Figure 5- The influence of applied magnetic field on 5mg/ml chloroform suspensions of (a,c) nanowires and (b,d) nanorods. Image (a) describes image (c) in a 1d form at q= 1.182 nm-1. Image (b) describes image (d) in a 1d form at q= 1.226 nm-1.

The response of the chloroform suspensions was compared with the response of toluene suspensions of the same ZnS nanoparticles.

Figure 6 shows the same measurements of magnetic field effects on toluene suspensions with the same concentrations. Here, the norm images (Figure 5c,d) are much clearer and a certain alignment can be seen. Comparing the 10^{-4}T curves to the others (Figure 5a,b), the peaks obtained are much higher and clear, so it is easy to say that the magnetic field has a stronger impact in aligning the toluene suspension.

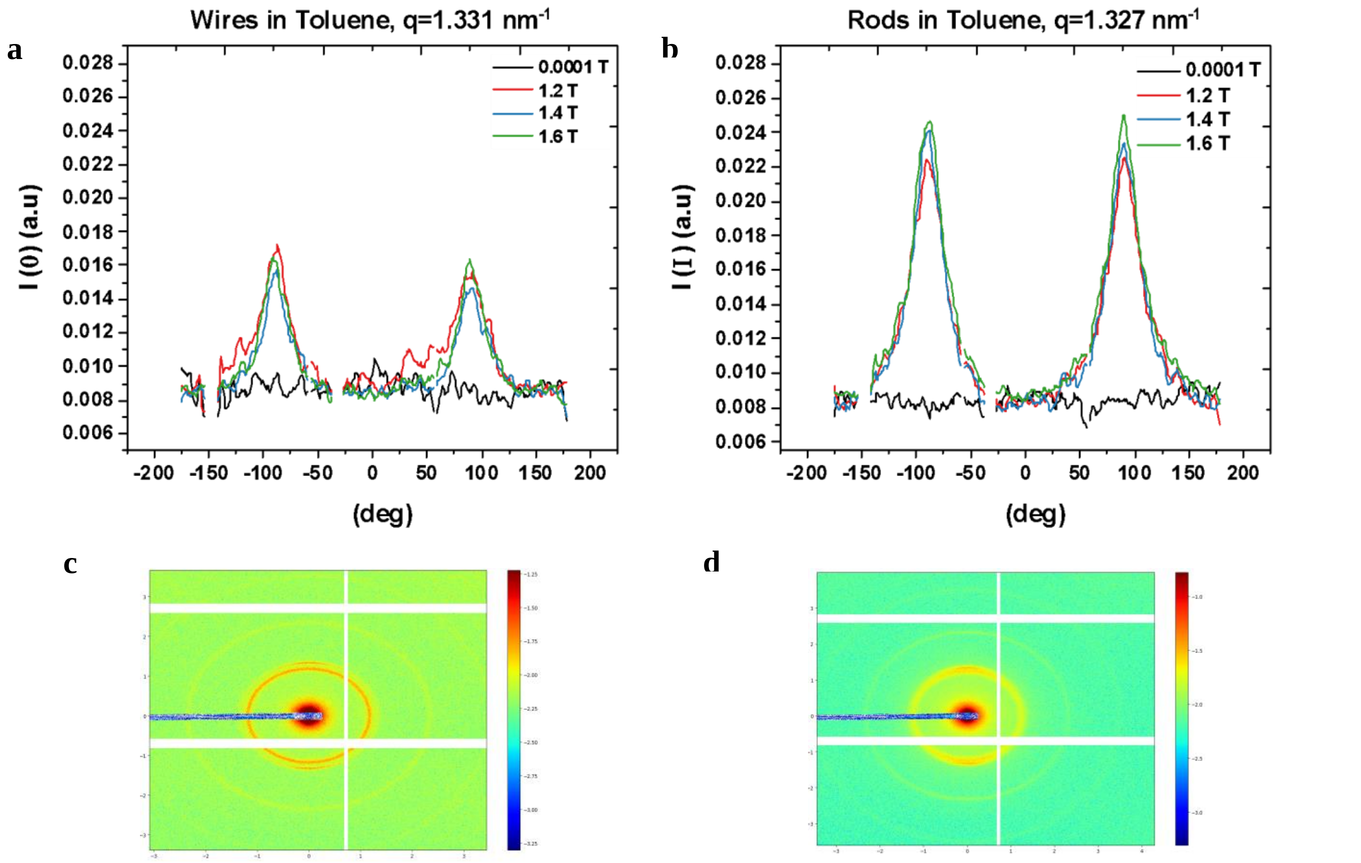

Figure 6- The influence of applied magnetic field on 5mg/ml Toluene suspensions of (a,c) nanowires and (b,d) nanorods. Image (a) describes image (c) in a 1d form at q= 1.331 nm-1. Image (b) describes image (d) in a 1d form at q= 1.327 nm-1.

Conclusions:

The results show that the alignment of the ZnS nanoparticles occurs under high values of magnetic field and strongly depends on the solvent of the suspension. The much stronger response of the toluene suspensions will clearly require further studies.

Future work:

In a future experiment at BM29, we are planing to carry out SAXS/WAXS measurements of the ZnS nanoparticles toluene suspensions with different conentrations. Together with the future result we hope to be able to better understand the differences between the chloroform and the toluene suspension systems.

References:

1. Belman, N., Israelachvili, J. N., Li, Y., Safinya, C. R., Ezersky, V., Rabkin, A., Sima, O., & Golan, Y. (2011). Hierarchical superstructure of alkylamine-coated ZnS nanoparticle assemblies. *Phys. Chem. Chem. Phys*., 13, 4974-4979.