APSYNC
Automated Precision Synthesis of Nanocrystals

SoPPoM –SBO4
Overview

• Apsync: what and why
• Objectives
• Situation within SoPPoM
• Status
• Concept and innovative aspects
• Conclusion
Apsync: what and why

- **APSYNC**: Automated Precision Synthesis of Nanocrystals
- What and why:

  - development and installation of:
    - an automated synthesis platform of parallel batch reactors
    - based on scalable reactor design concepts

  - offers the possibility to automatically execute:
    - different syntheses in parallel (high-throughput screening), or
    - the same synthesis multiple times (scaled up production)
Apsync: objectives

- **APSYNC : Automated Precision Synthesis of Nanocrystals**
- **Objectives:**

  - high-throughput screening of reaction chemistry/nanocrystal property relations for CI(G)S, QD and TCO nanocrystals
  - understanding of reaction chemistry/nanocrystal property relations via a combined approach of reaction simulations and experimental studies on reaction kinetics and reaction mechanisms
  - developing novel synthesis concepts where continuous precursor addition and in-situ reaction monitoring are combined to steer reactions towards a predefined nanocrystal size and size dispersion
  - large volume production of CIGS, QD and TCO nanocrystals
Apsync: situation within SoPPoM

**SBO 1: abCIGS**
- Synthesis precursor, NP's
- Surface chemistry
- Stable printable dispersions
- CIGS & hybrid inorganic-organic
- Annealing – CIGS
- Characterization

**SBO 2: weTCOat**
- Metal oxide screening
- Synthesis precursor, NP's
- Surface chemistry
- Stable printable dispersions
- Annealing
- Characterization

**SBO 3: phyCIGS**
- Cell integration
- Cell characterization
- Cell modeling
- Cell semiconductor physics

**SBO 4: APSYNC**
- Automated synthesis
- Scaled up synthesis of CIGS, QD and TCO
- QSPR for CIGS, QD and TCO
- Active reaction steering

**ICON 1: OPvTECH**
- Linear deposition process of multilayered stacks
- Design, fabrication and characterisation of multijunction OPV modules

**ICON 2: CIGstack**
- Inks and formulations
- Deposition technologies
- Rapid annealing

**ICON 3: SOLCAP**
- Improvement of CIGS cell efficiency and reliability at the module level (not cell level).
Apsync: interactions with SoPPoM projects

• larger volume precursor production
  - SoPPoM SBOs have delivered a first set of precursors that are relevant for the industrial research projects. These are being synthesized in larger quantities within Apsync.

• high-throughput screening
  - To shorten the time for materials development Apsync will use high-throughput screening of reaction conditions where the reference recipes developed within abCIGS and weTCOat are taken as a starting point.

• insight in reaction mechanisms.
  - Relations between reaction conditions, yield development, and final nanocrystal size that result from high-throughput screening experiments help understanding reaction mechanisms: information needed by abCIGS and weTCOat to further improve synthesis recipes or identify general concepts underlying the synthetic methods used.

• Whereas for the larger volume production, APSYNC will mainly rely on input from abCIGS and weTCOat, the high-throughput screening is an activity that will involve considerable feedback between the different projects.
Apsync : status

• Activities in four areas:
  
  – Platform for automated synthesis and purification of nanocrystals.

  – Delivery of Cl(G)S nanocrystals
    • To ICON 1 and 2 (through SBO1)
    • Initial delivery of 50 g (exceeded amounts, within agreed time frame)

  – Delivery of TCO nanocrystals
    • To ICON 1 and 2 (through SBO2)
    • Initial delivery planned for end of this month (in progress)

  – Delivery of colloidal QD
    • planned for 2014
Apsync : CIS nanoparticles

- Procedure from SBO-1 (UGent)

Copper(II) acetylacetonate → Indium(III) acetylacetonate → Sulfur → ODE → OLA

Heat 1h @ 240 °C → Centrifugation washing → CIS nanopowder

T2BKO – 170 nm x 170 nm
Apsync : CIS nanoparticles

- **Transfer to platform at Flamac**
  - Batches analysed by
    - X-ray diffraction,
    - UV-VIS and
    - TEM (selected)

![Graph showing 2θ angular distribution](image-url)
Apsync : AZO nanoparticles

- Procedure from SBO-2 (Imec/UHasselt)

<table>
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Apsync : AZO nanoparticles

- Transfer to platform at Flamac
  - Variation in manual procedures during transfer procedure
  - Optimization of procedures regarding dopant addition
Apsync: platform

- Objectives:
  - development and installation of:
    - an **automated synthesis** platform of **parallel batch reactors**
    - based on **scalable reactor design** concepts
  - offers the possibility to automatically execute:
    - different syntheses in parallel (**high-throughput screening**), or
    - the same synthesis multiple times (**scaled up production**)

- Uniqueness ??
  - High-throughput methodologies are established as research tool
  - Scale-up is regular process
  - BUT: not if combined and aiming for “larger” volumes
Apsync : platform

- Challenges and advantages:
  - High throughput screening of materials: reduce time-to-market
Apsync : platform

- **Challenges and advantages:**
  - High throughput screening of materials
  - Automation, parallel/rapid sequential, miniaturize
  - Nanocrystal workflows : typically mg scale ...
  - Screening: optical, chemical, ... : OK
Apsync: platform

• **Challenges and advantages:**
  – **Screening:**
    • How about dispersing agent selection and optimization?
    • How about printing ink formulation and optimization?
    • How about post-processing of films?
    • How about ...
    • Much more material needed then mg scale : 100 g scale
  – **Upscaling:**
    • How relevant is a 250 mg scale lab synthesis for a 100 kg pilot batch, if reaction kinetics, diffusion and mixing are extremely important for particle morphology and size (distribution) (apart from chemical composition)?
  – All challenges (and more) tackled in the Apsync project ...
Apsync : platform

- Platform – Hardware
Apsync: platform

- **Platform – Hardware**
  - Five parallel reactors
    - Temperatures up to 300 °C
    - Atmospheric pressure
    - Inert atmosphere
    - Reflux (option)
    - Reactor volume ± 75ml each
    - Rapid heating-up (>100 °C/min)
    - Rapid cooling (> 60 °C/min)
  - **Dimensions**
    - Downscaled from DIN reactor dimensions
    - Including (baffled) stirring
      - From magnetic flee to Rushton turbine
Apsync : platform

• Platform – scaling :
  – Choice 1 : larger reactors than typical in high throughput screening
  – Choice 2 : multiple batches (fastest in screening phase)
  – Choice 3 : building knowledge on
    • Influence of process parameters
    • Kinetics and mechanisms
    • Providing input for application of scaling laws
Apsync : conclusion

• A unique platform for automated nanocrystal synthesis
  – Allowing high-throughput screening and experimentation
    • Materials (composition)
    • Process conditions
  – Allowing fundamental understanding of reaction mechanisms and kinetics
  – Allowing deduction inputs for scaling parameters

• Also for process research : high-throughput concept allows studying broader parameter space

• Concept under demonstration and first upscaled output (parallel batches) delivered to SoPPOM ICONs

• Important :
  – Nanocrystal is initiator and first case, but not limited to nanocrystal synthesis
  – Sustainability is important aspect of the study as well (use of alternative sources (eco-nomic/logic aspects), re-use/recycling of solvents, influence on reaction conditions, Eco-scale comparison of reaction settings, ...)

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