

## Characterization of Magnetic Nanomaterials Synthesized within Thin Centrifugally Motivated Fluid Films

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Magnetic nanomaterial synthesis is commonly a batching process [1, 2] with tightly controlled and highly limited product characteristics [3]. Spinning Disk Reactor (SDR) technology provides a novel methodology for continuous production of nanomaterials with highly variable reaction and production parameters. As demonstrated by Cafeiro, *et al* [4] for nonmagnetic nanomaterials, variations in these parameters can result in particle size distributions with tunable characteristics.

SDR synthesis is a fluid film based process conducted on the surface of a rapidly (<3000RPM) rotating disk. A binary fluid precipitation reaction occurs within turbulently mixed, centrifugally motivated thin (5-100 $\mu$ m) films of reactants during short (<1s) residence periods on the disk (see Fig. 1). Factors influencing particle morphology and size distribution characteristics achieved by the reactor include film thickness and speed, a direct result of the angular velocity of the disk, feed rates, feed concentrations, atmospheric conditions (in the case of a non-inert atmosphere), disk temperature and the presence/concentration of surfactant chemicals.

Transmission Electron Microscopy plays a key role in this study, providing information on particle composition, morphology and size distribution characteristics. High Resolution TEM (HR-TEM), Scanning TEM (STEM) imaging and compositional analysis using Energy Dispersive Spectroscopy (EDS) and Electron Energy Loss Spectroscopy (EELS) are proving valuable tools for the characterization of materials produced using this novel processing technique. The key issues involved with the application of these TEM techniques include sample preparation and the degree to which results are representative of the particle/distribution characteristics of bulk samples, as well as the selection of suitable TEM operating conditions and image/result analysis.

This presentation will provide an introduction into SDR synthesis, and a study into particle characteristic and distribution dependencies on disk speed and reactant concentration. Results from magnetite produced using an adaptation of Massart's binary reagent nanoparticle precipitation process [1] will be presented. The value and validity of a range of TEM-based analyses conducted will be explored and compared with the results of other techniques of analysis.

### References

- [1] Massart, R., *Magnetic fluids and process for obtaining them*, USP: 4329241 (1982)
- [2] Bonnemant, H., *et al.*, *Inorganica Chimica Acta*, 350 (2003) 612-624
- [3] Hyeon, T., *ChemComm*, (2002) 927-934
- [4] Cafeiro, L.M., *et al.*, *Ind. Eng. Chem. Res.*, 41 (2002) 5240-5246

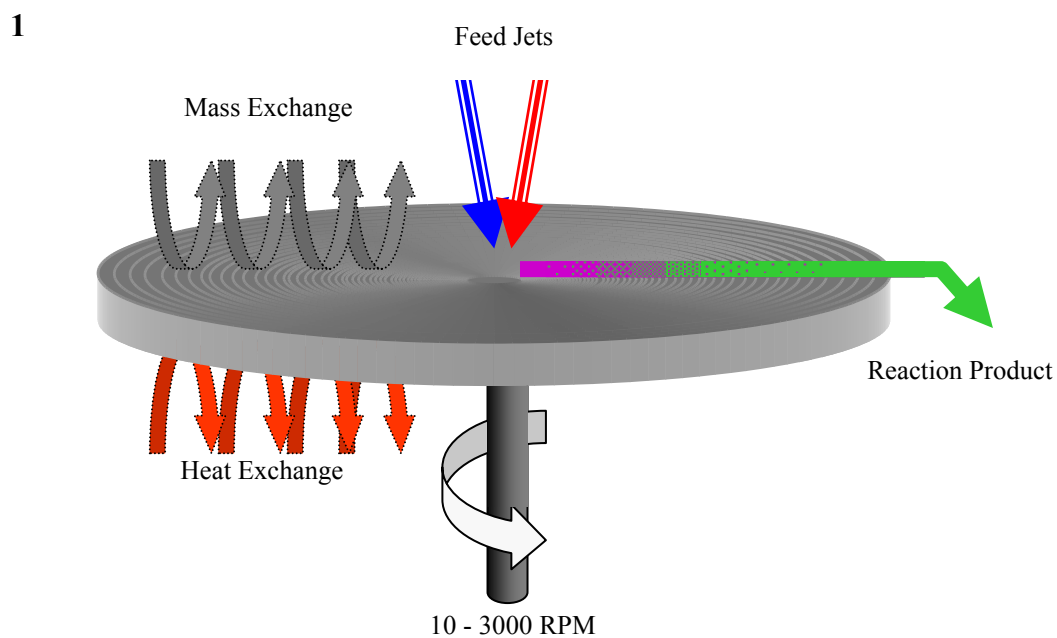


FIG. 1. Conceptual diagram of SDR demonstrating basic operation and key operating parameters

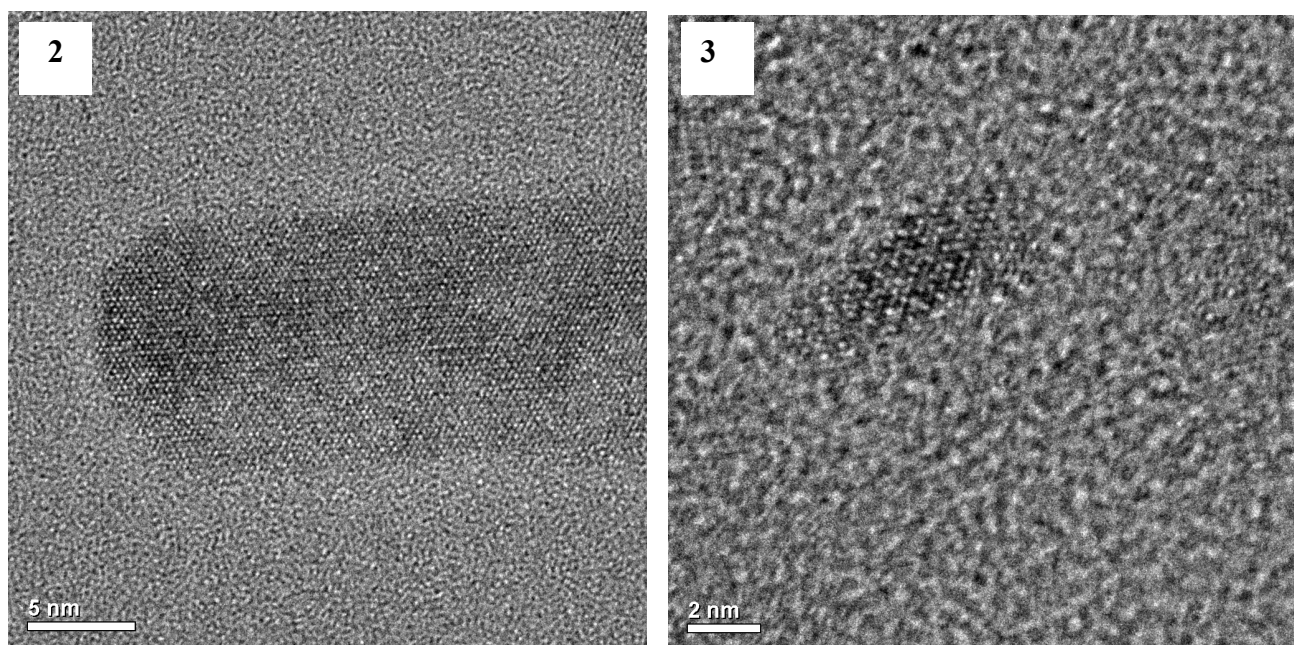


FIG. 2. Magnetite particle ( $\sim 45 \times 15 \text{ nm}$ ) from control sample produced without the use of the SDR.  
 FIG. 3. Magnetite particle ( $\sim 7 \times 2 \text{ nm}$ ) generated within SDR at 3000RPM.