Enclosed Flame Spray Pyrolysis: Control of Product Particle Characteristics by the Air Entrainment

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Synthesis of functional nanoparticles requires more and more refined production processes. Enclosing the scalable flame spray pyrolysis (FSP) process facilitates synthesis of a number of sophisticated nanomaterials (Strobel and Pratsinis 2007). That way, for example, it is possible to make metallic nanoparticles (Athanassiou *et al* 2006), coat nanoparticles with thin silica (Teleki *et al* 2008) or carbon layers (Waser *et al* 2011) and control the $FeO/Fe_3O_4/Fe_2O_3$ particle phase composition (Strobel and Pratsinis 2009).

In-situ coating or functionalizing such particles' surface with SiO_2 requires a conveyed aerosol stream for efficient precursor utilization and hermetic coating (Teleki *et al* 2008). Such conditions can be achieved in tube-enclosed FSP configurations where for in-situ carbon coating of $LiFePO_4$, both, oxygen control and conveyed aerosol are required (Waser *et al* 2011).

Primary particle size is another important aspect of functional nanoparticles and its control by FSP settings (including precursor solution composition and burner configuration) is well established. The underlying mechanism for this size variation lies in controlling the temperature history of the particles (Pratsinis 1998) and droplet/particle concentration of the respective flames (Strobel and Pratsinis 2007). So tube-enclosed flame configurations lead to larger product particles compared to open FSP at equal flame conditions (Strobel and Pratsinis 2009) as air entrainment and heat losses can be limited substantially. So there are further opportunities for size and composition that still remain unexplored.

Here we investigate the primary particle size and structure of CuO nanoparticles made in a tube enclosed FSP configuration as a function of entrained air a) flow rate, b) radial velocity and c) preheating temperature. The air is thereby supplied through a ring gap of variable height at the FSP surface level. The resulting particles were analyzed by X-ray diffraction (XRD), nitrogen adsorption (BET), transmission electron microscopy (TEM) as well as differential mobility analyzers (DMA) and aerosol particle mass (APM) analysis to determine the aerosol particle structure.

The entrained air flow rate plays a key role in nanoparticle synthesis in tube-enclosed FSP: The product primary particle size decreases from about 40 to 10 nm (d_{BET}) with increasing air flow rate from 0 to 300 L/min at constant flame settings and thus constant production rate (Fig. 1, circles). Also the CuO crystallite size (d_{XRD}) follows the same trend indicating formation of monocrystalline nanoparticles (Fig. 1, squares).

By supplying higher air flow rates into the tube than an open flame would naturally entrain (≥ 300 L/min), even smaller primary particles can be obtained than in open configuration (open symbols in Fig. 1). This is a result of more efficient cooling and dilution of the freshly formed aerosol in these over-ventilated flames that reduce particle growth by coagulation and sintering.

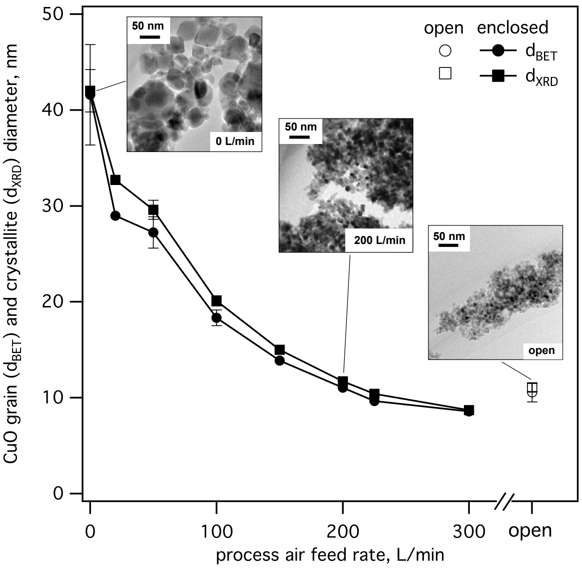

Figure 1. The CuO grain (d_{BET}, circles) and crystallite sizes (d_{XRD}, squares) decrease with increasing entrained air feed rate allowing precise primary particle size control at constant production rate and even smaller particle sizes than with open-FSP due to more efficient flame quenching.

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