Continuous Flow Synthesis of ZnO Nano-Array integrated Monoliths (Supported by NSF SNM Award # CBET-1344792)

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Introduction

One-dimension nanostructures of ZnO in the forms of nanorods and nanowires have evolved as a unique family of technologically important nanomaterials in the past decade[1]. However, cost-effective and scalable nanomanufacturing of ZnO nanostruture integrated monolithic devices is of a great challenge, which calls for new methods, instrumentations, as well as new manufacturing protocols and principles.

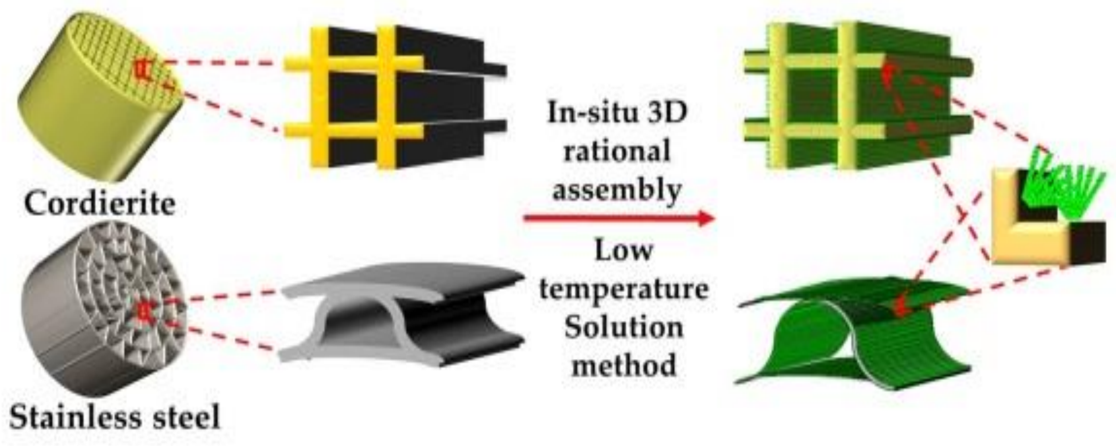

Figure 1. *In-situ* grown nanostructure arrays on 3D monoliths as new catalytic devices.[2-7]

Method

The fresh zinc acetate and HMT solution is driven by the peristaltic pumps. Thus, the solution flow is generated inside the honeycomb substrate. The solution is preheated before injection into the reactor, then cooled down and filtered before injection into solution reservoir.

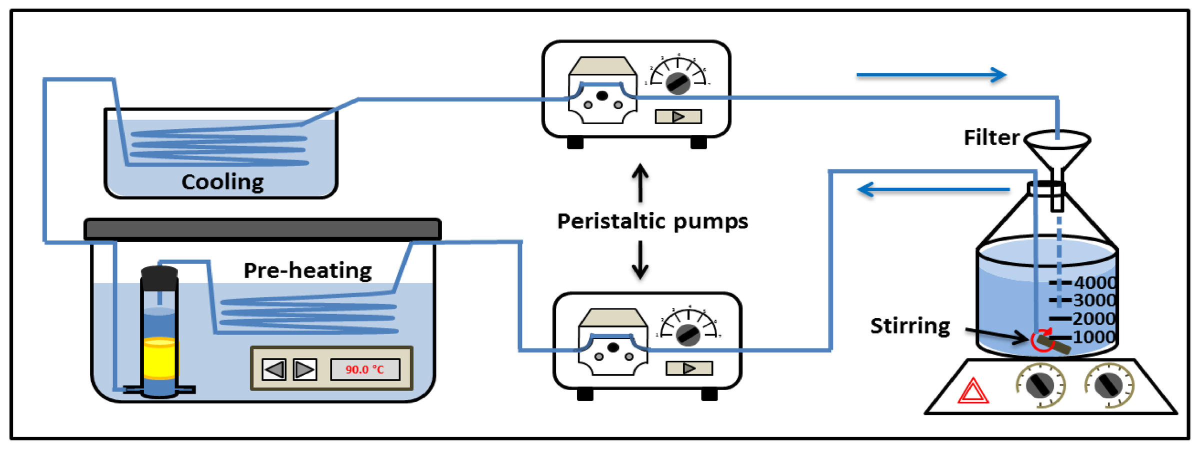

Figure 2. Experimental setup of continuous flow synthesis

Results and discussion

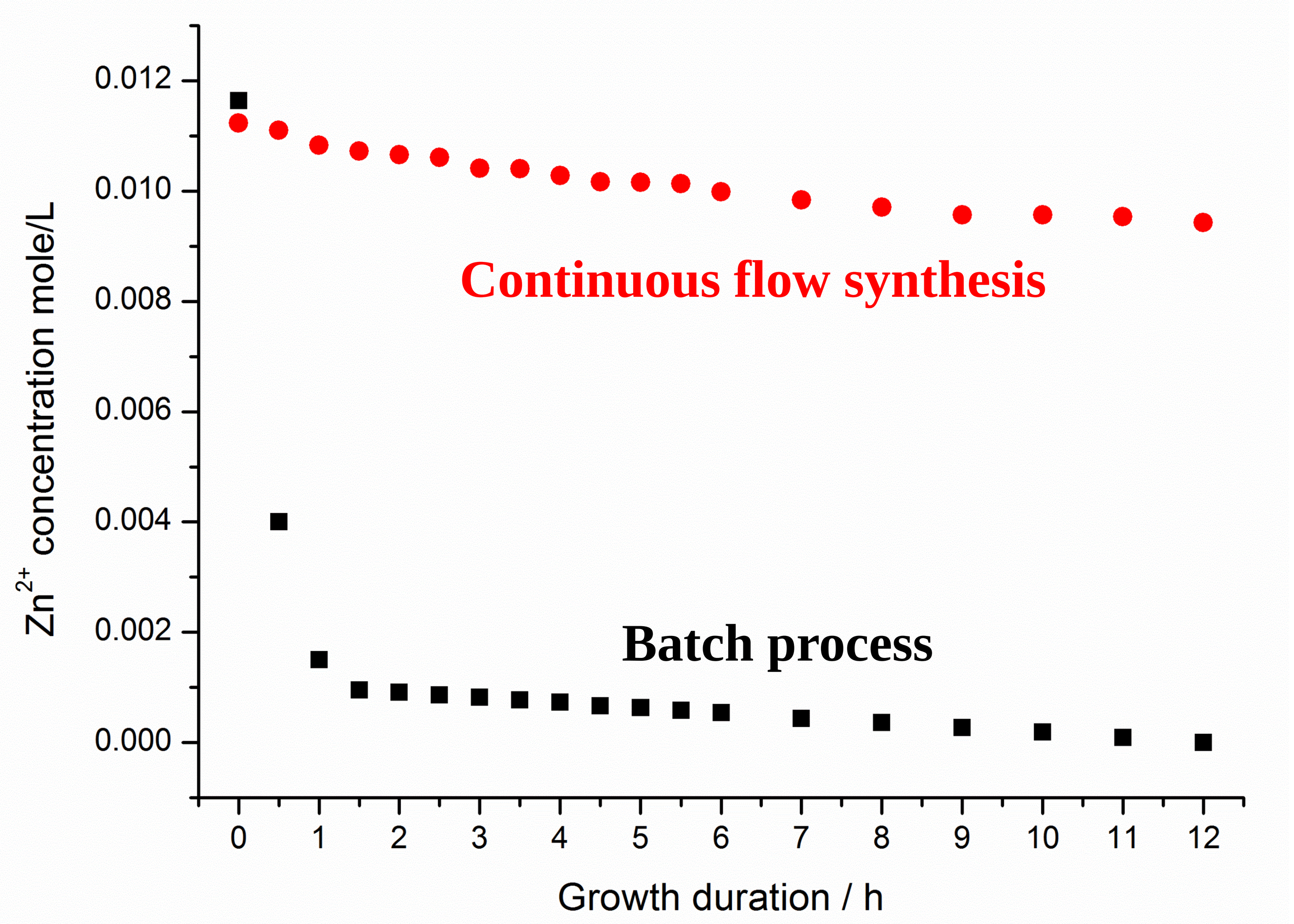

Figure 3. ICP characterization result of Zn^{2+} concentration as a function of growth duration

	Solution concentration (mM)	Solution usage (ml)	Growth temperature (° C)	Growth time (h)	Stir rate and pumping rate	Average length of nanorods (μm)	Average diameter of nanorods (μm)	Weight loading ratio (%)
Batch	12.5	600	90	3	600 rpm	2	180	4.3
CFS	12.5	600	90	3	3.3 ml/min	3.5	250	5.5

Table 1. Statistical results of batch process vs. continuous flow synthesis

Compared to conventional batch process, continuous flow synthesis (CFS) possesses enhanced materials utilization efficiency (**Figure 3**) and growth rate (Table 1) due to the suppression of precipitate growth and heating-cooling circulation.

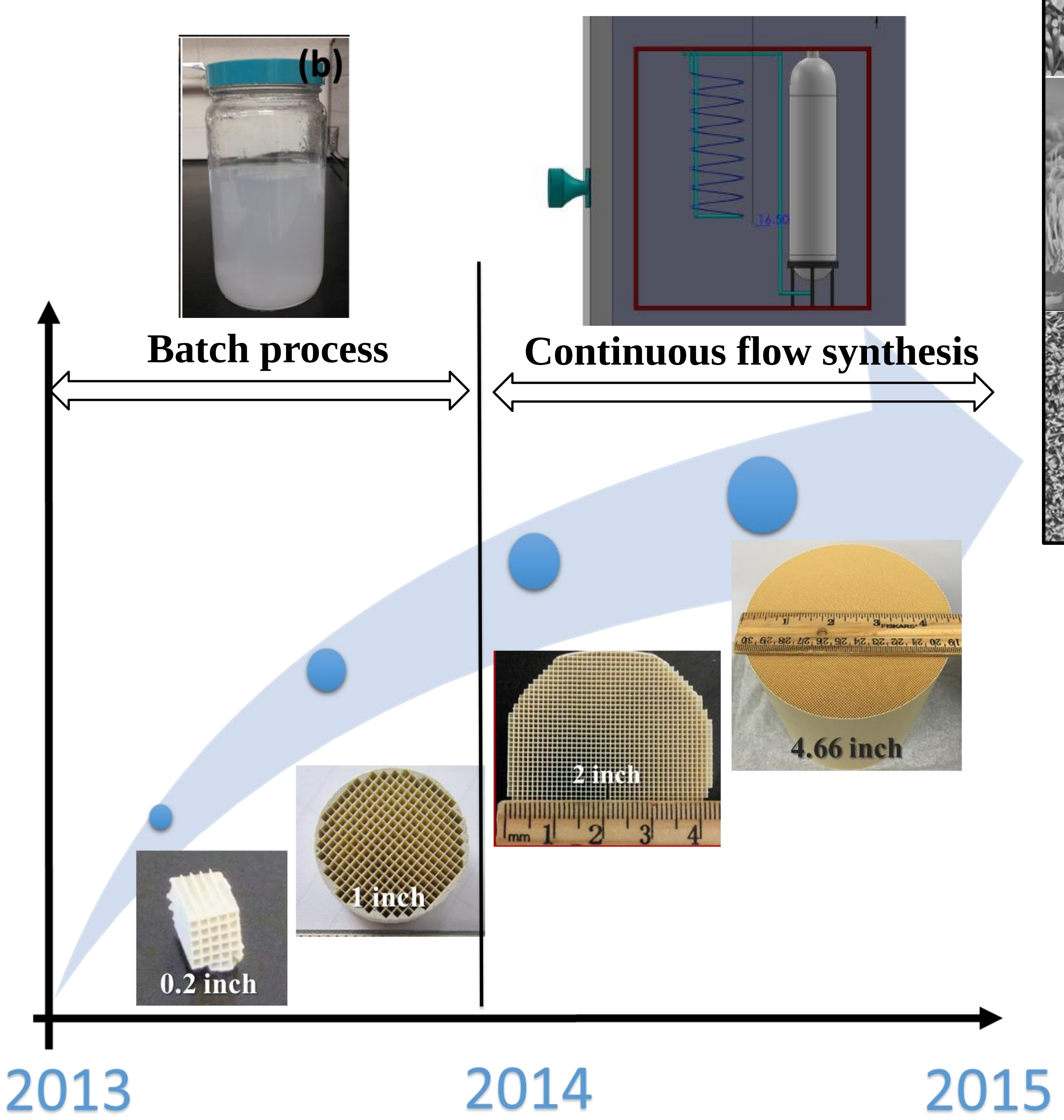

Figure 4. Manufacturing timeline of nano-array based monoliths

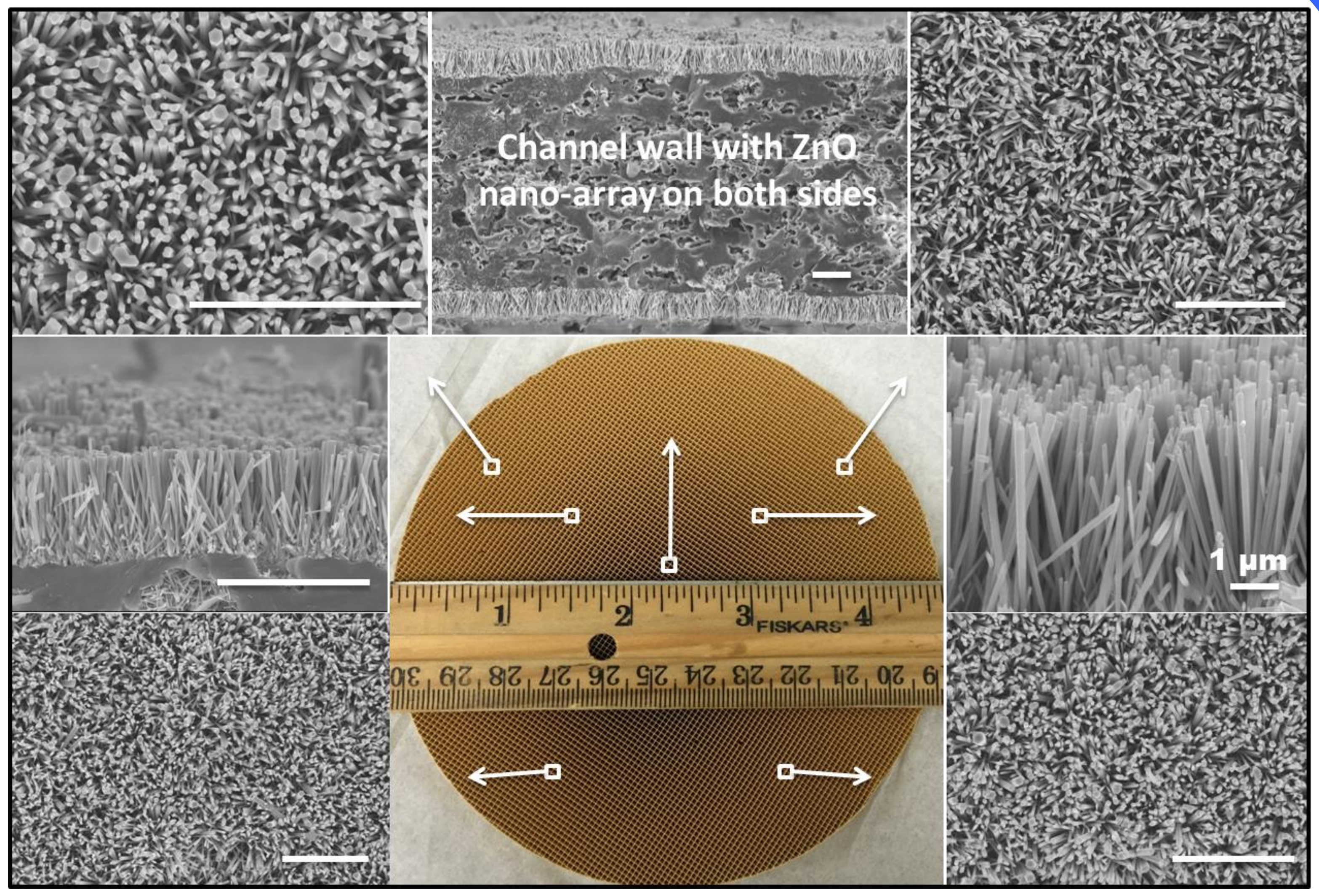

Figure 5. Lateral scale-up manufacturing using continuous flow method

Shown in **Figure 5** is uniformly distributed ZnO nanorods array on a 4.6″ wide honeycomb substrate successfully achieved via continuous flow synthesis. Uniformly distributed ZnO nano-array is grown on the surface of channel walls.

Although the CFS shows much enhanced growth rate compared to the conventional batch process, the average integration rate is far from enough for industrial relevant large-scale production. Microwave irradiation is being utilized as one of the external stimuli to significantly accelerate various types of nano-array monolith synthesis and manufacturing.

Conclusions

A low-temperature hydrothermal based continuous flow synthesis was successfully conducted to integrate uniformly distributed large-scale ZnO nanorod arrays with commercial 3D cordierite honeycomb substrate with channel length up to 5 cm. As compared to traditional batch process, the morphology of ZnO nanorod array achieved from CFS possessed longer average length, significantly enhanced distribution uniformity across the channel length, and significantly improved precursor utilization efficiency. With the assistance of microwave and sophisticate setup design, the novel continuous flow hydrothermal synthesis holds great promise towards industrial-relevant scalable manufacturing of 3-D nano-array based monolithic devices.

References

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