



第173回GMSI公開セミナー/第63回GSDMプラットフォームセミナー

## Autonomous Experimentation Applied to Carbon Nanotube Synthesis

カーボンナノチューブ合成の自律制御

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日時: 2015年7月7日(火) 10:00-11:30

場所: 東京大学工学部2号館 31A会議室(機械系会議室)

The progress of 'Nanotechnology' is the key scientific and engineering issue which has strong impact on current social design. For innovative energy system, innovative information technology, and innovative environmental system, carbon nanotubes are representative material of nanotechnology. Carbon nanotubes have an exciting array of applications which span mechanical, electrical, thermal and chemical/sensing. However, full exploitation is slowed by a lack of control over synthesis. Despite the two decades since the explosion of work in the area, progress in controlled production of nanotubes is impeded by our lack of understanding of the fundamental mechanisms of nucleation and growth. Our group has endeavored to develop a method that addresses the critical bottlenecks impeding the speed of research by taking advantage of advances in robotics, artificial intelligence, data sciences and in-situ/in-operando characterization.

Our Autonomous Research System, ARES, is capable of designing, executing and evaluating its own CNT growth experiments. Artificial intelligence module based on random tree / genetic algorithm statistical approach analyses experimentally obtained kinetic parameters (rate, time constant, etc.) and proposes new experiments to achieve user-defined objective. These are then executed by ARES automatically and without human intervention, and fed back into the AI module to ensure machine learning.

Recent experiments utilized maximum growth rate as an objective. The normalized difference between the objective and experimentally observed growth rates behaves in a fashion similar to what is typically seen in the control systems, with experimentally observed growth rate oscillating around the target. The convergence can be expressed via cumulative root mean square (RMS) of the rate difference. RMS increases initially (divergence), followed by consistent decrease after ~50 experiments, indicating convergence. That is, after some unsuccessful experimentation, ARES was better able to supply experimental conditions that achieved the objective growth rate. We take this as a clear demonstration of autonomous AI learning: convergence on the objective via closed-loop iterative experimentation without human intervention.