Controlled Synthesis of High-Entropy Alloy Nanoparticles by an Inductively Coupled Plasma Jet (ICPJ) with a Supersonic Nozzle

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Abstract: We studied the controlled synthesis of CoCrFeMnNi high-entropy alloy nanoparticles (HEA NPs) by the ICPJ process. Adjustments in reactor pressure, feed rate, plasma gas composition, and adapting a convering-diverging nozzle demonstrated a great flexibility in controlling nanoparticle structure and crystallinity.

1. Introduction

High-entropy alloys nanoparticles (HEA NPs) exhibit unique properties due to complexity in their composition and the confinement effect. These nanoparticles are of significant interest thanks to their large surface areas and superior catalytic performance with great potential for many fields such as catalysis, energy storage, and biomedical applications. The synthesis of HEA NPs has been a focus of numerous studies, yet the control over their size and microstructure in a broad range still remains a challenge. Recently, we reported continuous synthesis of HEA NPs by the ICPJ process [1]. Here our study focuses on tailoring HEA NPs size, structure, and composition in the ICPJ process by employing a kinetically and thermodynamically controlled environment.

2. Methods

The Cantor alloy (CoCrFeMnNi) was selected for the study. We designed a converging-diverging (C-D) nozzle to control the kinetics of particle growth in the plasma while operating parameters such as reactor pressure, feeding rate, and plasma gas composition were adjusted to control the thermodynamics in the particle growth. Computational Fluid Dynamics (CFD) simulations were performed to analyze the effects of those parameters on the plasma flow and temperature profiles, providing an insight into the synthesis process. Characterization of the HEA NPs produced was conducted by XRD, SEM, and TEM.

3. Results and Discussion

The results highlight a significant impact of the process conditions on the structure and composition of the nanoparticles produced. A lower feed rate decreases the vapor pressure and thus effectively delays the supersaturation of vapors, which is crucial for controlling the nucleation process. Also, reducing the reactor pressure increases the plasma jet velocity, which decreases the growth time of particles. The implementation of the C-D nozzle had the most effect by dramatically increasing plasma jet velocity and decreasing particle growth time, which were key factors in reducing both particle and crystallite sizes. Particles were synthesized with tunable average sizes ranging from 95.7 nm to 19.6 nm while crystallite sizes varied between 22.3 nm to 10.6 nm, demonstrating an ability to control the particle size and degree of crystallinity.

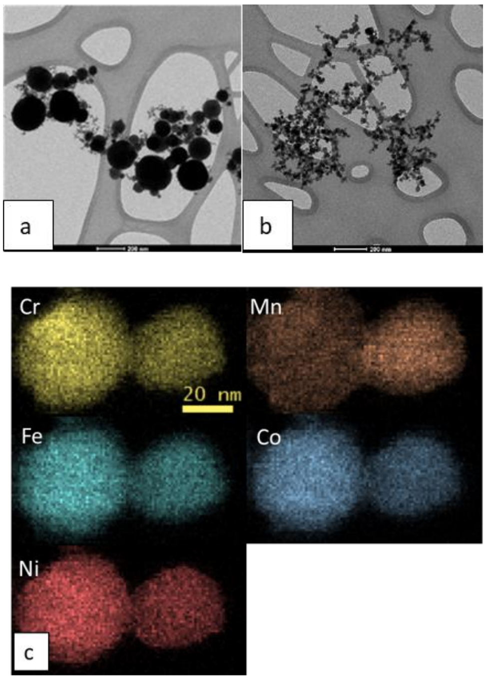

Fig. 1. TEM images of the HEA NPs produced by (a) Ar-H_2 at 66.67 kPa, 1g/min without nozzle (b) Ar-He at 26.67 kPa, 0.5 g/min with nozzle. (c) EDX elemental map of HEA NPs

4. Conclusion

This study successfully demonstrates a scalable, controlled approach to synthesize HEA NPs with tuable structural and compositional properties, emphasizing the importance of process parameters in achieving desired particle characteristics.

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References

[1] K.S. Kim et al., Nat. Commun., 15, 1450 (2024).