Oral/Invited

Fri. Aug 1, 2025 9:10 AM - 10:30 AM JST | Fri. Aug 1, 2025 12:10 AM - 1:30 AM UTC 🏛 Room B(2F The SalonThe Grand Ballroom East)

[4B01-04] Session 6

Chair:Keisuke Muramatsu(Shinshu University)

9:10 AM - 9:30 AM JST | 12:10 AM - 12:30 AM UTC

[4B01]

Synthesis of solid solution alloy nanoparticles and study of their structure transition

*Mai Thanh Nguyen[1], Tetsu Yonezawa[1] (1. Hokkaido University (Japan))

9:30 AM - 9:50 AM JST | 12:30 AM - 12:50 AM UTC

[4B02]

Synthesis and Bonding Characteristics of Copper Nanoparticle Pastes Coated with Carboxylic Acids

*MASAHITO GOTO[1], Tetsu Yonezawa[1], Hiroki Tsukamoto[1] (1. Hokkaido university (Japan))

9:50 AM - 10:10 AM JST | 12:50 AM - 1:10 AM UTC

[4B03]

[Invited] Synthesis of Quasi-Single Crystalline Nanoporous Iron Oxides using Metal Chloride as a Precursor

*Takamichi Matsuno[1], Daichi Oka[1], Kohei Takaoka[1], Atsushi Shimojima[1] (1. Waseda University (Japan))

10:10 AM - 10:30 AM JST | 1:10 AM - 1:30 AM UTC

[4B04]

[Invited] Interconnection of organic-inorganic hybrid hydroxide salt nanoparticles toward design of porous architectures

*Naoki TARUTANI[1], Kiyofumi Katagiri[1], Kei Inumaru[1] (1. Hiroshima University (Japan))

Mai Thanh Nguyen and Tetsu Yonezawa

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Synthesis of solid solution alloy nanoparticles and study of their structure transition

Solid solution alloy nanoparticles are considered as a promising candidate for a wide range of advanced applications in such as catalysis and optics. Using a co-sputter deposition technique, we have been successfully synthesized binary and ternary solid solution alloy nanoparticles composed of various metal systems including those typically miscible in the bulk or with large miscibility gaps, with composition of intermetallic compounds.[1-4] Our findings demonstrate the ability to control both particle size and tuning particle elemental composition across a broad range.

It is well established that the solid solution alloy nanoparticles can undergo changes in their crystal structure when subjected to elevated temperatures, particularly under catalytic conditions. In this current study, we focus on the structure transition behavior of binary solid solution alloy nanoparticles, specifically those with composition lying within the bulk miscibility gaps or corresponding to intermetallic compounds. We observe temperature dependent transition from solid solution alloy to intermetallic structure. A detailed discussion for a case study of AgPt nanoparticles will be presented to illustrate this transformation.

1) Nguyen, M. T., Zhang, H., Deng, L., Yonezawa, T., Tokunaga, T., *Langmuir,* **33**, 12389 (2017).

2) Deng, L., Nguyen, M. T., Shi, J., Chau, Y.-t. R., Tokunaga, T., Kudo, M., Matsumura, S., Hashimoto, N., Yonezawa, T., *Langmuir,* **36**, 3004 (2020).

3) Zhu, M., Nguyen, M. T., Chau, Y.-t. R., Deng, L., Yonezawa, T., *Langmuir,* **37**, 6096 (2021).

4) Zhu, M., Nguyen, M. T., Sim, W. J., Yonezawa, T., *Mater. Adv.*, 3, 8967 (2022).

Masahiro Goto,[1] Hiroki Tsukamoto,[2] and Tetsu Yonezawa[1]

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Synthesis and Bonding Characteristics of Copper Nanoparticle Pastes Coated with Carboxylic Acids

Copper nanoparticles (CuNPs), owing to their high surface area-to-volume ratio and elevated surface energy at the nanoscale, exhibit distinct mechanical, thermal, electrical, and chemical properties compared to bulk copper. In this study, surface modification of Cu NPs was achieved using three types of carboxylic acids, such as hexanoic acid, to enhance dispersion, suppress agglomeration, and prevent oxidation. These modified nanoparticles were then used to formulate Cu NP pastes with triethanolamine (TEA).

Copper nanoparticles were synthesized via the liquid phase chemical reduction of CuO micropowder in an organic solvent containing carboxylic acids. The resulting CuNPs were collected and re-dispersed in TEA to prepare Cu NP pastes with concentrations ranging from 80 to 90 wt% in 2 wt% increments. These pastes were applied to copper substrates and subjected to pressure sintering at 200°C under 15 MPa using a home made fixture with a commercial hot press. Post-sintering, shear strength was measured, and the fracture surfaces and nanoparticle morphology were analyzed via SEM to assess the dispersion and structural characteristics.

It was found that the type of carboxylic acid had a noticeable effect on the crystal grain size and morphology of the Cu NPs. Longer carbon chains in the carboxylic acids tended to produce smaller and more variably sized grains. In terms of bonding strength, all types of carboxylic acid coatings showed improved strength at higher NP concentrations, likely due to increased viscosity. Interestingly, while high-concentration pastes could not be formulated with dodecanoic acid, samples prepared at lower concentrations still exhibited high bonding strength, indicating a distinctive effect of this acid on particle interaction and sintering behavior.

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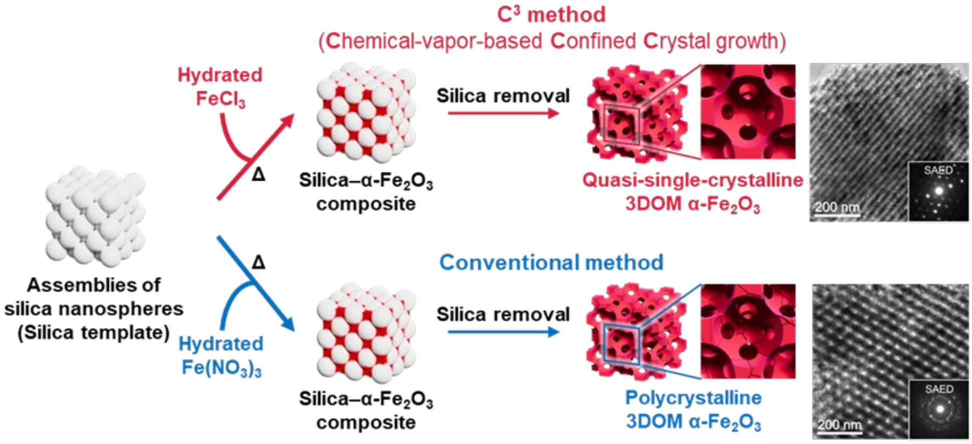
Synthesis of Quasi-Single Crystalline Nanoporous Iron Oxides using Metal Chloride as a Precursor

Ordered nanoporous metal oxides have attracted attention for their broad applications across fields, including catalysts, energy materials, and biomaterials. In addition to the compositions and porous structures, controlling the crystallite size of the pore walls is crucial for improving performance.[1] However, precise control of the nanostructures from the atomic to the nano-scale range is still challenging, because of the limitation of the conventional synthesis method.

In this study, we report the preparation of a quasi-single-crystalline nanoporous metal oxide via oxidation of a metal chloride precursor within the nanopores of silica templates (Scheme 1).[2] We focused on iron oxide, which is used in various applications such as catalysts and electrodes, and investigated its properties in an inverse opal (3DOM) structure with a high specific surface area. Iron chloride inside the template is transported in the vapor phase upon heating, enabling the nucleation and crystal growth of α-Fe_2O_3. As a result, nanoporous α-Fe_2O_3 with a larger and more uniform crystallite size than that prepared using the conventional $Fe(NO_3)_3$ hydrate precursor was achieved. The quasi-single-crystalline nanoporous α-Fe_2O_3 exhibited higher catalytic activity and thermal stability than the conventional nanoporous α-Fe_2O_3 composed of nanocrystals, as confirmed by methylene blue degradation and heating tests, respectively.



Scheme 1. Synthesis of 3DOM α-Fe_2O_3 with different crystallite sizes.

References: (1) W. Li and K. Xie, *Acc. Chem. Res.*, **56**, 374−384 (2023). (2) D. Oka, K. Takaoka, A. Shimojima, and T. Matsuno, *Chem. Mater.*, 10.1021/acs.chemmater.5c00155 (2025).

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Interconnection of organic-inorganic hybrid hydroxide salt nanoparticles toward design of porous architectures

The bottom-up assembly of nanoscale building blocks into functional architectures is an effective approach to overcome the limitations of conventional bulk material design. Among such building blocks, metal hydroxide salts have attracted attention due to their unique layered structures and chemical versatility, enabling the incorporation of diverse metal cations and coordinative species. By introducing polymerizable organic species into the hydroxide layers, organic-inorganic hybrid nanoparticles can be obtained, providing enhanced dispersibility, surface functionality, and interparticle interactions.

In recent studies, a material design platform was established that integrates nanoparticle stabilization, controlled interconnection, and three-dimensional structuring. Carboxylate-functionalized metal hydroxide salts were used to prepare stable colloidal dispersions of nanometer-scale hybrid particles with concentrations exceeding 40 wt%1). The coordinated acrylates were polymerized to induce three-dimensional interconnection, forming porous networks with tunable meso- to macroporous structures while preserving the crystallinity of the inorganic components. The process offers precise control over architecture with/without templates (Fig. 1).

Moreover, the hybrid structure plays a key role not only in shaping but also in material transformation. Heat treatment triggers simultaneous decomposition of the organic moieties and dehydration of the hydroxide layers, leading to self-reduction and conversion into various inorganic materials, including metals, metal chalcogenides, and pnictides2). This thermal conversion enables precise control of both chemical composition and nanostructure of the porous objects. Overall, these findings demonstrate that dispersion and interconnection control of organic-inorganic hybrid hydroxide salt nanoparticles provide a versatile and unified strategy for designing hierarchical porous materials with advanced functionality.

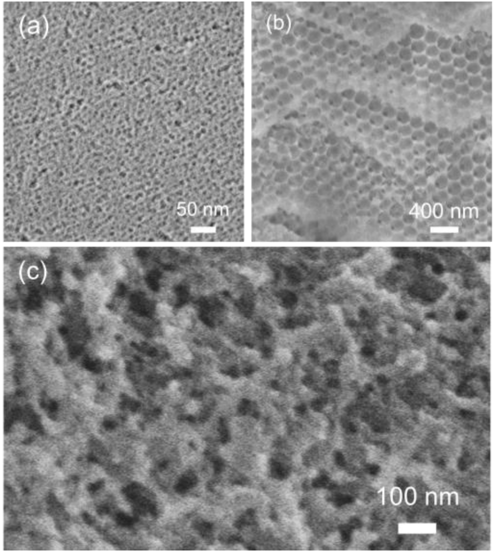

Fig. 1 Scanning electron microscope images of porous materials prepared (a)(b) with and (c) without templates

1) Tarutani, N. *et al.*, *J. Ceram. Soc. Jpn.,* **131**, 830 (2023).
2) Tarutani, N. *et al.*, *Inorg. Chem.,* **62**, 13977 (2023).