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CONCEPT

Advances in Metal 3D Printing Technology for Tailored Self-Catalytic Reactor Design

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Abstract: Metal 3D printing is revolutionizing the industry by enabling rapid and cost-effective production of complex metal 3D products with intricate geometries, attracting significant attention in recent years. Particularly, metal 3D-printed catalysts have emerged as a cutting-edge fusion technology that serves as both catalysts and reactors, referred to as self-catalytic reactors (SCRs). Therefore, this concept article aims to introduce the recent advancements in metal 3D printing technology in the catalyst and reactor field, and illustrate the various aspects for the rational design of self-catalytic reactors.

1. Introduction

In recent years, metal 3D printing technology has seen rapid advancements, moving from a nascent technology to an essential tool in numerous industries, including aerospace, automotive, healthcare, and more recently, chemical engineering.^[1,2] This technology's ability to fabricate complex geometries with fine precision, reduced waste, rapid prototyping capability, and scalability has opened unprecedented possibilities in catalyst reactor design and operation.^[3] Specifically, this technology allows for the fabrication of reactors with intricate internal geometries, enabling the production of highly efficient catalysts with high surface area, optimized reactant flow paths, and tuneable selectivity.^[4-6] Moreover, the new concept of integration of catalyst and reactor functions, covered by the term self-catalytic reactor (SCR), has been recently proposed and opened a new way of optimizing the usage of metal 3D printing in the catalytic field (Figure 1).^[7-9] However, the successful integration of metal 3D printing technology into the design and manufacturing process of catalytic reactors hinges on solving unique challenges, including materials selection, printer resolution, post-surface treatment requirements, and durability under harsh reaction conditions. In spite of these challenges, the potential benefits, such as improved catalyst performance, reactor design efficiency, and environmental sustainability, require thorough investigation and discussion.

This concept article aims to present an overview of the latest advances in the application of metal 3D printing technology to catalytic reactor design. We highlight recent advancements and unique advantages of this technology, identify the key challenges faced in its application, and discuss future research directions in this exciting field.

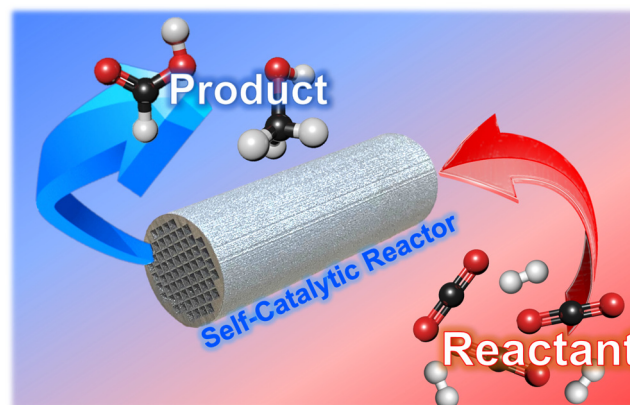


Figure 1. Metal 3D-printed self-catalytic reactor as a fusion technology of metal 3D printing technology in the catalysis field.

2. Advancements in Metal 3D Printing Technology in Catalytic Field

Metal 3D printing methods, exemplified by electron beam powder bed fusion (EB-PBF) and laser powder bed fusion (L-PBF), have been extended into the catalysis field. In the case of L-PBF, a powdered raw material is first uniformly coated onto a build plate in a thin layer using a recoater blade. The material is selectively melted with high-intensity laser energy. Next, the build platform is lowered and the blade is used to spread a new powder layer over the previous layer. By repeating melting and

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solidification layer by layer and stacking two-dimensional objects, a three-dimensional object is finally obtained.

The initial application of metal 3D printing technology to the field of catalysis has taken two distinct routes: catalyst design and reactor design. In catalyst design, metal 3D printing is primarily employed to fabricate intricate 3D porous catalyst architectures.^[4,10–12] These complex geometric structures can be transformed into macro-nano porous configurations using chemical-electrochemical dealloying methods. This transformation not only increases surface area but also enhances ion-mass transport during catalytic reactions. In 2017, Zhu et al. used a combination of direct ink writing (DIW) and chemical dealloying to fabricate nanoporous gold (Au) from an Ag-Au alloy (Figure 2a).^[4] This 3D-printed hierarchical nanoporous Au exhibited 10-fold higher electric field-driven ion transport and 105-fold higher pressure-driven mass transport than conventional nanoporous Au, resulting in a 2.2-fold enhancement in methanol oxidation.

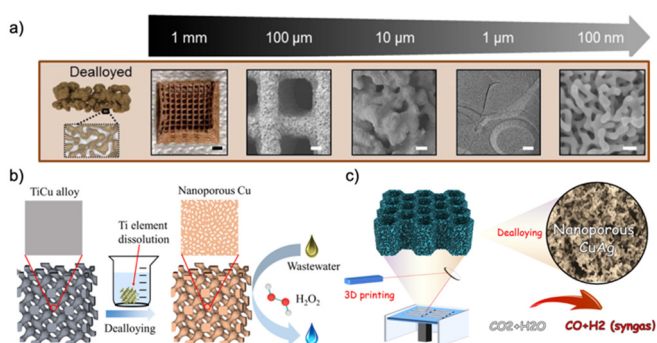


Figure 2. Dealloying of a) Ag-Au alloy for the fabrication of nanoporous Au for methanol oxidation.^[4] b) Ti-Cu alloy for the preparation of nanoporous Cu for wastewater treatment.^[11] and c) ZnCuAlAg alloy as a precursor of nanoporous CuAg for electrochemical CO₂ reduction.^[12] Reprinted with permission from ref.^[11] Copyright 2021 American Chemical Society. Reprinted with permission from ref.^[12] Copyright 2021 American Chemical Society.

Similarly, Cai et al. successfully fabricated micro-nano porous copper (Cu) for wastewater treatment by integrating selective laser melting (SLM) and chemical dealloying of a Ti-Cu alloy (Figure 2b).^[11] SLM enabled the construction of a ~90% porous Ti-Cu alloy precursor with high resolution, and subsequent chemical dealloying led to the creation of a 3D-printed micro-nano porous Cu. This micro-nano porous Cu structure was used in conjunction with a Cu/H₂O₂/visible light system for removal of rhodamine B and achieved a reaction rate of $8.24 \times 10^{-2} \text{ min}^{-1}$, which was approximately 8- and 2.5-fold higher than Cu²⁺ and Cu powder/H₂O₂/visible light systems, respectively. Additionally, some research groups have utilized these porous structures as electrocatalysts. Zhang et al. prepared a 3D-printed hierarchical nanoporous copper (3Dp-hnp-Cu) structure by dealloying an Mn-Cu alloy prepared using SLM.^[10] This 3Dp-hnp-Cu structure exhibited a 9.2-fold improvement in electrocatalytic methanol oxidation compared to conventional nanoporous copper due to facile mass transport and a larger specific surface area. Similarly, Yan et al. applied a 3D nanoporous Cu-Ag alloy to electrochemical CO₂ reduction. They used SLM to prepare a ZrCuAlAg alloy, which was then dealloyed to produce 3D

nanoporous CuAg catalysts with a high density of Ag and Cu nanodomains (Figure 2c).^[12] This catalyst demonstrated a tunable H₂/CO ratio, with syngas production rates of 140 μmol/h/cm² and high stability.

In the case of reactor design, the utilization of metal 3D printing involves the creation of complex structures to facilitate heat and mass transfer. Avril et al. used electron beam melting (EBM) to fabricate a 3D-printed static mixer with Ti, CoCr, and STS (Figure 3a).^[5] They coated it with Ni and Pt catalysts through metal cold spraying or electrodeposition. The complex geometry of the static mixer maximized heat and mass transfer in hydrogenation reactions, reduced the formation of side products, and minimized the pressure drop. Vilé et al. similarly developed a 3D-printed static mixer with STS 316L fabricated using EBM (Figure 3b).^[13] The mixer was washed coated with an alumina-based layer and a single-atom Cu-based C₃N₄ catalyst. This complex architecture effectively controlled internal fluid flow and heat transfer, resulting in high turnover frequencies (TOF) of 551 h⁻¹ and 1563 h⁻¹ for benzaldehyde and furfural reduction, respectively. Moreover, Gutmann et al. designed a microreactor using STS produced via SLM for rapid difluoromethylations using fluoroform (Figure 3c).^[14] This specially designed microreactor not only enhanced heat and mass transfer but also provided chemical and high-pressure resistance. It enabled exothermic catalytic reactions in cryogenic environments and elevated pressures.

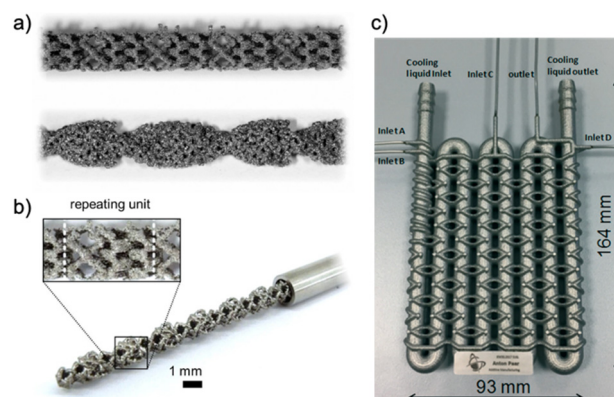


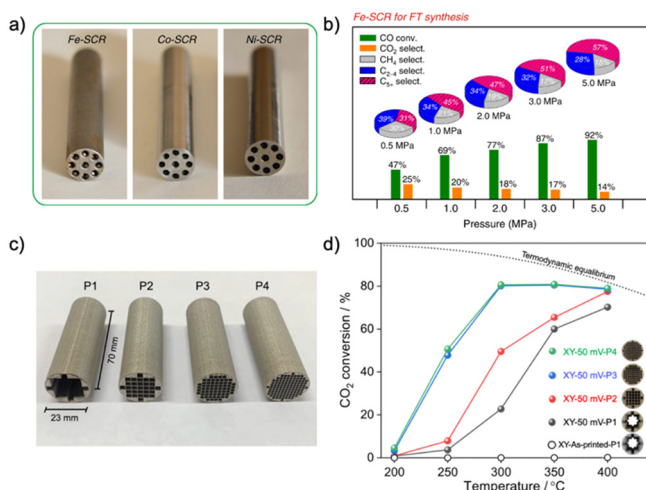
Figure 3. a) Two different complex geometric designs of 3D-printed static mixer for hydrogenation.^[5] b) Repeating micromixer reactor prepared by STS 316 for benzaldehyde and furfural reduction.^[13] c) Microreactor fabricated by STS 316L for rapid difluoromethylations using fluoroform.^[14] Reprinted with permission from ref.^[5] Copyright 2017 The Royal Society of Chemistry.

As a post-functionalization approach for 3D-printed metallic catalytic flow reactors, Mori et al. recently proposed the utilization of metal organic frameworks (MOFs) as a modifier. The Cu-based 3D-printed reactors were immersed into an acid solution containing an organic linker for the synthesis of MOFs, where Cu²⁺ ions dissolved in situ were assembled to form MOF crystals on the surface of the reactor.^[15] This further allows carbonization, deposition of metal nanoparticles, and modification with organic molecules, enabling H₂ production from liquid-phase hydrogen storage materials.

As a next step of utilization of metal 3D printing in catalytic field, a novel concept known as the self-catalytic reactor (SCR) has emerged, which integrates both catalyst and reactor functions.

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The catalytic active sites in an SCR are generated within the reactor itself through self-oxidation or dissolution, simplifying post-treatment processes and enhancing reusability.^[7,9] This integrated approach not only optimizes reactor capacity utilization but also streamlines catalyst development, potentially accelerating the development of new commercial catalysts. Wei et al. utilized selective laser sintering (SLS) to fabricate an SCR with Fe, Co, and Ni for harsh catalytic reactions such as Fischer-Tropsch (FT) reactions, CO₂ hydrogenation, and methane dry reforming, demonstrating high performance and reusability (Figure 4a and b).^[7] Moreover, Wang et al. expanded the application of a Ni-SCR in CO_x methanation. Simple calcination transformed the Ni-SCR into an active catalyst with conversion rates of 35.2% for CO and 39.6% for CO₂ at 450 °C and 350 °C, respectively.^[8] Kim et al. created Ni-SCRs with four different pattern shapes using SLM for CO₂ methanation and applied electrochemical methods to expose active Ni sites (Figure 4c and



d).^[9] After the appropriate electrochemical treatment, the Ni-SCR was transformed into a highly active catalyst, achieving 80% CO₂ conversion in the P4 pattern with high stability and reusability.

Figure 4. a) Optical photograph of Fe-, Co-, and Ni-SCR and b) Catalytic performance of calcined Fe-SCR in the Fischer-Tropsch reaction.^[7] c) Optical photograph of Ni-SCR with four different patterns and d) Catalytic performance of electrochemically treated Ni-SCR in the CO₂ methanation reaction.^[9]

3. Customized Self-Catalytic Reactor Design using Metal 3D Printing

3.1 Materials and building conditions

The initial step in designing an SCR involves the selection of suitable materials. The choice of appropriate metal elements should align with the targeted catalytic application. For instance, Fischer-Tropsch (FT) reactions benefit from Fe or Co alloys, CO₂ hydrogenation thrives with Ni or Fe alloys, and methanol oxidation finds efficacy in Cu and Au alloys.^[7,10,16,17] Furthermore, the selection of alloying elements is closely related to most surface treatment processes for SCR, so that careful consideration is required.

The building conditions for an SCR also significantly impact its design and performance optimization. Presently, most SCR are produced using SLM and EBM due to their high resolution, quality, and scalability. In the SLM and EBM processes, building conditions such as scan strategy, laser power, and scan speed exert a profound influence on the crystallographic attributes of the components. This leads to variations in crystal texture, grain size, and dislocation density due to differing thermal gradients during the construction process.^[18,19] These attributes play a vital role in surface treatment, effectively acting as interfaces, and can yield significantly different outcomes. Kim et al. employed two different scan strategies, X and XY, for the fabrication of a Ni-SCR.^[9] The inverse pole figure (IPF) image showed that the different scan strategies resulted in significantly different crystal texture (Figure 5a). The XY scan Ni-SCR showed 1.4 to 1.7 times larger grain size and approximately 1.4 times more geometrically necessary dislocations (GND). This divergence influenced the growth of the Ni(OH)₂ layer during electrochemical treatment and affected CO₂ methanation performance (Figure 5b). Hence, the establishment of databases for each material, coupled with a consideration of the building conditions, takes precedence for the proper design of SCR.

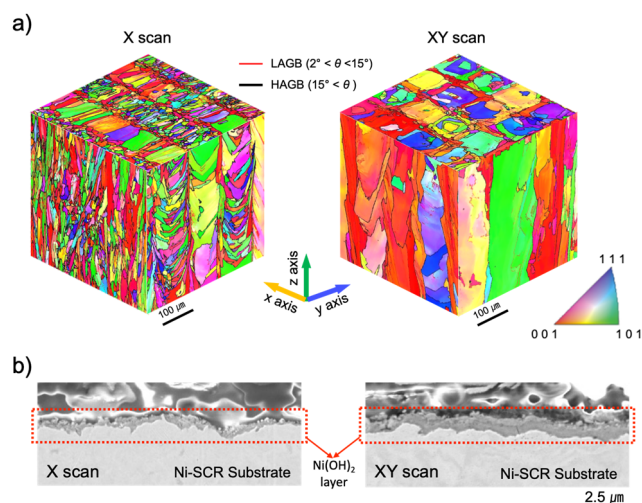


Figure 5. a) 3D visualization of inverse pole figure images of X and XY scan Ni-SCR. b) Cross-sectional view of X and XY scan Ni-SCR after electrochemical selective dissolution.^[9]

3.2 Surface functionalization

Although metal 3D printing has enabled the fabrication of catalytic reactors with intricate geometries, the surface area of as-printed SCR remains limited, often around several square meters per gram ($\text{m}^2 \text{g}^{-1}$), and is covered with an oxide layer formed during printing.^[7] Therefore, subsequent surface functionalization is necessary to expose active sites and fully unlock the potential activity of SCR. One common method is chemical dealloying, which involves creating a galvanic cell between noble and active metals in an acidic or alkaline electrolyte. This process leaves a nanoporous structure with exposed noble metal sites on the surface, which are the active sites for catalytic reactions. Chemical dealloying has a great advantage in its simplicity without applying an external power source, whereas it has limitations such as the need to satisfy the parting limit, which is the minimum fraction of the active metal for

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dealloying to proceed, and a relatively long process time.^[20] To overcome these limitations, electrochemical treatment offers a promising alternative. This method can transform the SCR surface into an active catalyst within a few hours. Moreover, the selective enrichment of one of the alloy elements and tuneable microstructure can be achieved by a simple adjustment of treatment parameters, i.e., current density and potential.^[9,21] Oxidation and hydrothermal reactions are also promising methods that convert the SCR surface to being enriched by metal oxide, which is a support or precursor of catalytic active sites.^[8]

Most catalysts suffer from deactivation due to segregation, aggregation, and poisoning, which can hinder their long-term performance and recyclability.^[22] The concept of an SCR revolves around generating catalytic active sites within the reactor itself. Thus, appropriate post-treatments, often referred to as 'refresh' processes, can effectively regenerate these active sites. For example, a simple immersion of Ni-SCR in a 50 wt.% NaOH solution for just 10 minutes can lead to a continuous improvement of activity, demonstrating the effectiveness of the refresh treatment.^[9]

3.3 Geometric structure

The geometric configuration of the reactor is a pivotal determinant that profoundly influences the performance of an SCR due to its impact on the fluid dynamics of the reactant. Firstly, its impact on the heat-mass transfer: An increase in the cell density of the reactor proportionally increased the thermal conductivity of the reactor, leading to a more uniform heat distribution at higher cell densities, both radially and axially.^[9,23] Besides, the intricate porous and tortuous structure of the SCR facilitated more favourable diffusion of reactants, leading to superior catalytic activity compared to a planar surface.^[4] Secondly, its influence extends to the flow regimes within the SCR. The tortuous nature of the inner structure transforms laminar flow into oscillatory flow, consequently prolonging reactant-contact time (Figure 6).^[24,25] This not only augments catalytic activity but

also enables tunability of selectivity by promoting secondary reactions.

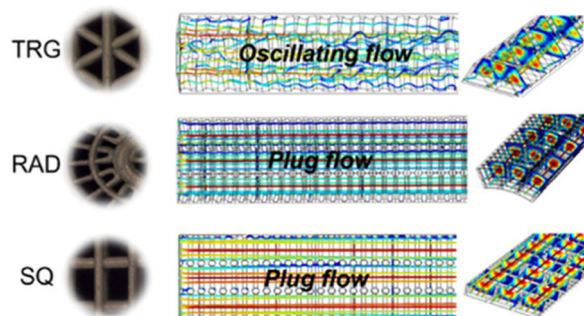


Figure 6. Transformation of flow regimes according to the geometric structure of reactor.^[26]

Nevertheless, it is imperative to note that excessive cell density or reduced pore size can lead to undesirable phenomena such as backflow and elevated pressure drops, thereby undermining catalytic performance.^[24,27] Consequently, the utilization of computational fluid dynamics (CFD) is indispensable for optimizing the reactor structure by modelling the hydrodynamics of the SCR. CFD allows simulating the fluid dynamics and is of great help in understanding the associated heat and mass transfer inside the SCR, facilitating optimization of the geometric structure. For instance, Mori et al. utilized CFD to elucidate the effect of pore size in a MOF-functionalized Cu-SCR on liquid-phase catalytic H₂ production. The CFD study indicated that a decrease in pore size from 2.0 mm to 1.5 mm caused a pressure drop (Δp) approximately 3 times greater, resulting in a negative effect on the catalytic activity by reducing the surface velocity.^[15] Moreover, Li et al. designed an optimum CuNi alloy monolithic catalyst for a reverse water-gas shift (RWGS) reaction combined with CFD method. Two different unit cell sizes of triply periodic minimal surface (TPMS) structure and honeycomb monolith were designed to investigate the effect of the geometric structure on the catalytic performance. The CFD results showed that the TPMS structure significantly improved mass and heat transfer

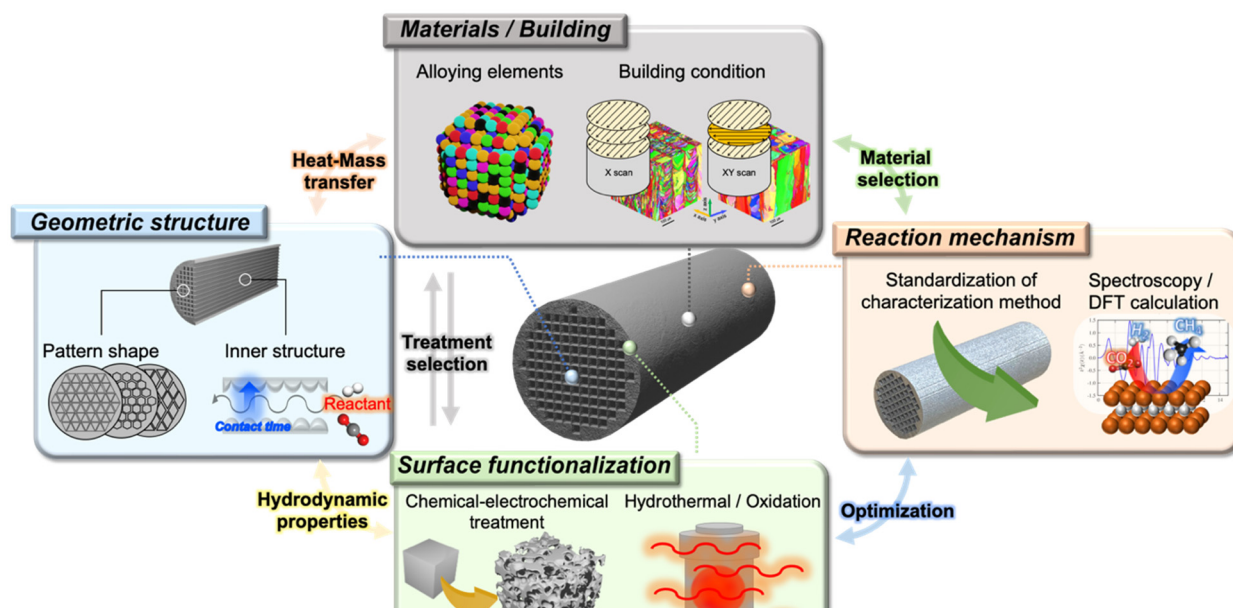


Figure 7. Rational design of self-catalytic reactor

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with a consistent turbulent flow in the 3D microchannels, and thereby exhibited 1.3 to 1.6 times higher catalytic activity than a honeycomb monolith.^[28]

3.4 Mechanistic study

Mechanistic study of SCRs is correlated with the selection of material and surface treatment.^[29] Multiple microscopic techniques, i.e., SEM, TEM, and confocal microscopy, can be used for analysis of the morphology of SCRs. Since the topology and microstructure of SCRs are changed during the process of building and surface treatment, both macro and nanoscopic analyses are important for precisely unveiling the reaction mechanism of SCRs. Spectroscopic analyses, i.e., XPS, XAS, Raman, and DRIFT, are also powerful tools for mechanistic study due to the fact that these techniques provide information on the local structure and surface properties, which are the reaction sites of catalytic reaction. Moreover, DFT calculation elucidates reaction pathways and provides a deeper understanding of the catalyst reaction. However, because of the large size of the reactor, there are still difficulties in the application of existing characterization methods to SCRs, and the regulation of a customized method is required for a proper mechanistic study of SCR.

4. Challenges and Future Outlook

Metal 3D-printed SCRs are a newly emerged research field, and thereby there is a lack of comprehensive information on SCRs. Further research is essential to establish a robust SCR design database containing materials, surface functionalization, geometrical structure, and mechanistic study results (Figure 7). This database would enable the creation of various scaled-up SCR shapes, i.e., heat exchangers and high-density reactors, for improved cost-effectiveness and industrial application with maximization of the advantages of SCRs. Despite these challenges, SCRs are anticipated to be a technology that explores potential future advancements in both 3D printing technology and catalytic reactor design.

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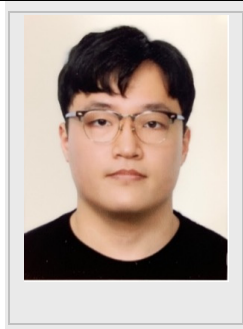
Keywords: Metal 3D printing • Catalyst • Reactor • Self-catalytic reactor

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Kohsuke Mori received his PhD degree from the Graduate School of Engineering Science at Osaka University under the supervision of Prof. K. Kaneda in 2003. In 2004, he joined Prof. T. Don, Tilley's group at University of California, Berkeley as a postdoctoral fellow. He moved to his current faculty in 2005 and then became an associate professor in 2009. His current research interests focus on establishment of clean and environmentally friendly carbon neutral hydrogen energy process based on metal and alloy nanoparticle catalysts and hybrid photocatalysts.



Takayoshi Nakano has been a professor at Osaka University since 2008 and a distinguished professor since 2017. He received his Ph.D. degree from Osaka University in 1996. He has been the director at Anisotropic Design & Additive Manufacturing Research Center, Osaka University since 2020, the president of Japanese Institute of Additive Manufacturing since 2022 and a member of the Science Council of Japan since 2023. In addition, he is a fellow of International Union of Societies for Biomaterials Science & Engineering. He was the president of Japanese Society for Bone Morphometry in 2017 and the Japan Institute of Metals and Materials in 2021 and 2022. He aims to establish a new academic field, "Materials Science based on Anisotropy".

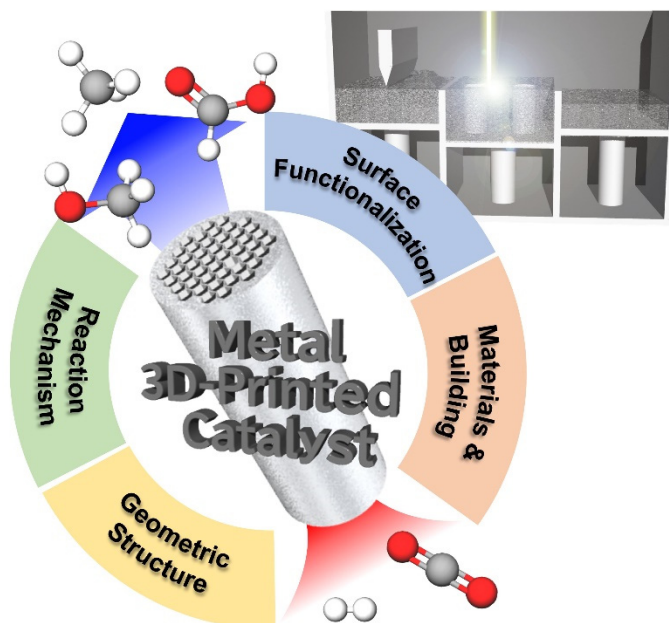


Hiromi Yamashita has been a professor at Osaka University since 2004. He received his PhD degree from Kyoto University in 1987. He was an assistant professor at Tohoku University, and an associate professor at Osaka Prefecture University. He was also a visiting research fellow at the California Institute of Technology and an invited professor of Université Pierre-et-Marie-Curie. He has been the president of the Catalysis Society of Japan, the president of the Asia-Pacific Association of Catalysis Societies, and the editor of Applied Catalysis B. His research interests include the design of nanostructured catalysts for sustainable energy and environmental uses.



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Metal 3D printing has attracted recent attention as a cutting-edge fusion technology in the catalyst field due to advantages such as design flexibility, variety of material selection, and ease of mass production. Additionally, catalytic efficiency can be enhanced by adjusting surface functionalization and geometric structural design. Metal 3D-printed catalysts are expected to provide a new direction for catalysis research by integrating existing individual catalyst and reactor research, thereby shortening the realization of a carbon-neutral society.