Materials Transactions

MT-T2023002

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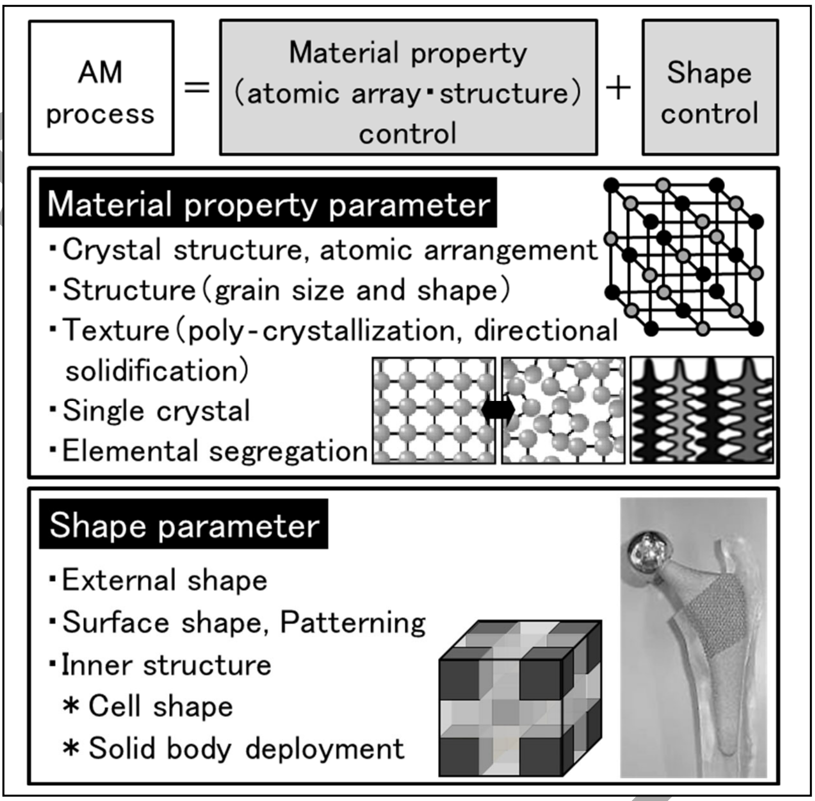
Doi: https://doi.org/10.2320/matertrans.MT-T2023002

Received: May 24, 2023
Accepted: July 14, 2023
Published: August 4, 2023



Graphical Abstract

Review − Microstructure Control and Function Expression Using Metal 3D Additive Manufacturing in the Digital Age[*1]

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[*1] This Paper was Originally Published in Japanese in Journal of Japan Thermal Spray Society 58 (2021) 121-128. The title was changed due to the addition of "Review−"

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Abstract

3D Additive Manufacturing is the heaven-sent child for Internet of Things (IoT) in the digital age, and is a process that can be used for customized design and production. In particular, metal Additive Manufacturing enables not only the fabrication of complex shapes but also the control of crystal orientation at the atomic level by locally melting and solidifying the metal material, thereby realizing high functionality of the product through simultaneous design of shape and materials properties. Therefore, it is expected to be applied in various social infrastructure fields including medical, energy-related, aerospace, and automotive, and also as a means of adding high value. In this review article, the new manufacturing concept that can be realized by metal additive manufacturing is introduced.

Keywords: *powder bed fusion, crystallographic texture, anisotropy, materials integration (MI) system, cross-ministerial strategic innovation promotion (SIP) program*

1. Introduction

The power of monozukuri that drives the manufacturing industry in Japan continues to significantly influence the global economy. Low-variety/mass-production processes have become a commodity; however, manufacturing systems are transforming to achieve mass customization in the form of high-variety/low-volume production and high-variety/mass-production. This accelerated development of high-value-added products has significantly changed the global manufacturing landscape[1-4]. As the 4th Industrial Revolution approaches, represented by "Society 5.0" in Japan, and as digitalization grows in the form of the Internet of Things (IoT), artificial intelligence (AI), big-data construction/analysis, digital transformation (DX), cyber-physical systems (CPS), and materials integration (MI) systems, great expectations have been placed on three dimensional (3D) additive manufacturing (AM), which can be considered the offspring of the IoT. Furthermore, computer simulations (forward problem/inverse design analysis), 3D/4D design, material development, material manufacturing processes, surface treatment technologies, processing/joining technologies, and quality control systems are being constructed as whole systems to serve as new manufacturing platforms that support the key technologies of AM that correspond to digitalization.

In this review paper, we focus on the metal AM, which will support manufacturing in the future, and introduces the Osaka

University Anisotropic Design & Additive Manufacturing Research Center affiliated with graduate school of engineering, Osaka university (referred to as Osaka University AM Center). Furthermore, we present social trends in the field, such as national projects related to metal AM, functional design by complex shape designs and by microstructure control at the atomic level enabled via metal AM accompanied by DX.

2. Osaka University Metal AM Center

In December 2014, the Osaka University AM Center was established (Fig. 1), equipped with powder bed fusion (PBF) metal AM equipment that uses electron or laser beams as heat sources, and various tools that enable shape design and quality control, including in-process monitoring$^{1)}$. The center also implements computational simulations to predict the temperature and stress fields, material structure, and atomic arrangement of the manufactured material via the metal AM process, while also developing tools that enable these processes. Recently, research and development has been conducted on an MI system that utilizes inverse design analysis, which enables the search for the optimum modeling conditions for metal AM process parameters using machine learning as an AI method to optimize the shape and function of the required molded objects$^{5)}$. Using MI systems leads to the effective utilization of metal AM and high added value of various functions, such as mechanical properties, corrosion and oxidation resistance, surface functionalization, and biocompatibility of molded materials[6-18]. The Osaka University Metal AM Center has two electron beam (Arcam Q10 and a domestic machine) and two laser beam (EOS M290) metal AM machines; the center conducts research and development and actively supports companies, research institutions, and other universities from its neutral standpoint as a university[1].

Figure 2 shows a conceptual diagram of anisotropic materials science, which forms the basis of the design concept and theory of the Osaka University Metal AM Center. The design policy of the center is to improve material functionality using a hierarchical structure based on isotropy /anisotropy[1]. In contrast to many artificial materials, e.g., existing metallic materials, designed to exhibit isotropic functions, most natural materials have an anisotropic structure such that functionality is achieved in the required direction in 3D, i.e., they have anisotropic structures in multiscale[19].

Figure 3 shows the anisotropic microstructure of living bones[20,21]. Bones are mainly constructed from a coherent relationship between type I collagen and apatite crystallites[22]; however, the *c*-axis orientation of the apatite crystallites along the collagen fibers changes, reflected in the *in vivo* stress distribution. Collagen and apatite crystallites exhibit a preferential *c*-axis alignment to the direction of the maximum principal stress vector, resulting in high strength in the orientation direction. The crystallographic texture in living bones is a good example of high functionality in a required direction; healthy and normal anisotropy is typically lost in diseased or regenerated bones and some genetically mutated mice[22-26]. Furthermore, osteocalcin is an important protein that controls the crystallographic consistency of collagen and apatite[27].

Anisotropic materials science can be defined as a "science related to the research and development of materials and elucidation of the mechanism for exhibiting the ultimate high-performance properties in the required direction." Metal AM is the ultimate means of freely producing objects that include such anisotropy[28,29]. Therefore, metal AM is one of the most suitable processes for creating individually customized substitutes for living bones[1,29].

3. National Projects Related to Metal Additive Manufacturing in Japan

Metal AM has attracted significant research attention domestically and internationally, with various related large-scale projects promoted in Japan. For example, the Cross-Ministerial Strategic Innovation Promotion (SIP) Program 1st Phase/Innovative Design and Production Technology ((Program Director: Dr. Naoya Sasaki; Funding (Management) Agency: NEDO), which started in 2014, involves the research and development of high value-added design using metal AM equipment and high functionality through microstructure control of metallic materials[30)]. The Monozukuri Revolution Program centered on 3D modeling technology by the Ministry of Economy, Trade and Industry (Technical Development of Next Generation Type 3D Printer for Industry), which also started in 2014, began developing next-generation domestic industrial metal AM equipment. Furthermore, the Technology Research Association for Future AM (TRAFAM) (President: Professor Hideki Kyogoku, Kinki University) continues to promote the construction of world-class next-generation industrial 3D printers and ultra-precision 3D modeling systems[31)].

The Cross-Ministerial SIP 2nd Phase/Materials Integration for Revolutionary Design System of Structural Materials[5)] (Program Director: Dr. Yoshinao Mishima; Funding (Management) Agency: JST), which started in 2018, aims to utilize the technical base of MI systems developed in Japan to construct the first MI development system that supports inverse design MI, which designs materials and processes based on desired performance. Targeting powder and metal AM materials, the project aims to utilize existing materials databases and build a database that supports new processes and evaluation technologies. The project aims to realize an inverse design MI system that fuses materials and information engineering and yields a material revolution that reduces the development period and cost of social implementation. Across three domains (A, B, and C), 13 teams from 44 institutions, including industry, academia, and government, were working together.

The three domains are as follows. Domain A, “Establishment of the inverse design MI basis for advanced structural materials and processing,” aims to establish an MI development system to realize Society 5.0, proposes the required structure and properties of materials to fulfill their desired performance, and enables the potential processing of such materials. Domain B, “Applications of the Inverse Design MI to Actual Structural Materials (Carbon fiber reinforced plastics: CFRP),” aims to develop technologies to improve the property and productivity of CFRP, which are becoming widely used as materials for lightweight structure, using MI development and lead the world in the development of transport equipment such as aircrafts. Domain C, “Applications of the inverse design MI to actual structural materials (3D powder processing),” aims to realize innovative materials and processes using MI development, with a focus on powders of heat-resistant alloys with intense development competition and ceramics which are super high temperature heat-resistant materials for next-generation transportation and energy equipment, to strengthen industrial competitiveness in Japan.

The A2 (“Processing design”), C1 (“Development of AM process for Ni-based alloys”), and C4 (“Development of powder manufacturing process and basic technologies for high performance TiAl based alloy turbine blades”) teams are based at the Osaka University Metal AM Center and lead the work on the social implementation of the MI system with mutual organic collaboration. The Osaka University Metal AM Center oversees the A2 team’s research on MI system technologies to accelerate the development of Ni-based super alloys, Ti-based alloys, and super heat-resistant ceramics, which are of crucial importance for aerospace and energy research, while also developing modules and workflows necessary for predicting the performances of target materials and processing. By utilizing the inverse problem analyses, they aim to establish a method to suggest appropriate materials and optimal processing conditions for a given desired performance. The author (Takayoshi Nakano) is the leader of Domain C; the C1 team considers the AM process for Ni-based alloys, which is an advanced process that can lead to innovations in the shape of parts and materials properties and is expected to be applied to combustion burners for hydrogen gas turbines. However, AM requires complex and wide-range parameter optimization. In their research, the C1 team aims to improve the durability of combustion burners by fabricating new Ni-based alloys found by the MI system for AM. The C4 team aims to develop superior low-pressure turbine blades using powder processes of metal injection molding and AM and build a sophisticated MI system for inverse problems in collaboration with other universities and industries. The MI system consists of a property prediction module microstructure design module, and process design module based on experiments and theoretical calculations and successfully allows the design of novel alloys to meet the required mechanical properties and geometries for both MIM and AM processes. The outcomes of this project will enhance industrial strength in Japan.

The C1 team applied laser beam-based metal AM to Ni-based alloys developed by the MI system; their method is an advanced process that could yield innovations in both the shape of parts and material properties and could be applied to combustion burners for power generation gas turbines that are exposed to high temperatures and have complicated flow paths. However, optimizing the complex and diverse parameters required by this laser metal AM process is difficult. The C1 team is searching for conditions in cyberspace using the MI system while also pursuing the optimization of conditions and demonstration of the unique functions of 3D AM components. In other words, they are working with the MI system for AM to verify the cyberspace process conditions in physical space and conduct demonstrations, including developing new Ni-based alloys; the Osaka University AM Center is building a basic database to build the MI infrastructure, optimizing the laser metal AM processes, and developing heat-resistant new alloys. The MI system will be integrated into the National Institute for Materials Science

(NIMS), and a consortium-type Japan-wide operation system is being constructed$^{32)}$.

4. Control of Shape and Material properties Using Metal Additive Manufacturing

AM is often considered for modeling 3D objects from simple to complex shapes. The main purpose of modeling metallic and ceramic materials using polymers and binders as raw materials is to control the shape of the material. Meanwhile, even with a molded object that has only undergone shape control, when a specific part is selectively melted/solidified by a heat source such as a laser or an electron-beam, temperature distribution effects, such as the migration velocity of the solid-liquid interface during solidification and the thermal gradient, induce cell growth with a preferred orientation and dendrite growth. Further, layer-by-layer modeling due to the influence of epitaxial growth and cyclic thermal profiles change the microstructure in a complex manner$^{7,33)}$. Hence, the PBF and directed energy deposition (DED) methods, which directly melt raw metal materials, control shape parameters as well as material properties, such as material structure and atomic arrangement. According to the often-used solidification map$^{34)}$, material properties, ranging from amorphous, polycrystal, columnar, and single crystal, can be controlled by controlling compositional supercooling and nucleation/growth conditions.

Figure 4 summarizes the material property (material structure and atomic arrangement) and shape parameters that can be controlled by metal AM. The material property parameters of metallic materials are directly linked to the mechanical properties and functionality of the modeled object; hence, methods for designing material properties become the deciding factor for the high added value of manufacturing using metal AM. Controlling functions while considering isotropy/anisotropy provides high functionality in a specified direction and can lead to the formation of specific material structures and expression of higher functions$^{1,4,11)}$.

Parameters involved in controlling the shape and material of a molded object are usually expressed in terms of energy density per unit volume (E), where E is a function of power (P), beam scanning speed (v), scanning interval (w), and layer thickness (h), as in Eq. (1), where E has units of J/m^3.

$$E = \frac{P}{v \cdot w \cdot h} \qquad (1)$$

E helps select process parameters for producing an optimal modeled object. However, E is essentially the amount of energy input to a unit volume; energy loss due to heat dissipation to the surroundings is not considered. The shape and material structure design of a modeled object requires controlling the heat distribution based on heat conduction and transfer; the shape of the molten pool formed by scanning the heat source and controlling the temperature distribution around the object, including the thermal gradient, are particularly important. Therefore, optimizing the melting conditions using a thermal simulation that considers heat dissipation enables the identification of non-melted, optimally melted, and excessively melted conditions, and the optimization of the shape parameters. Additionally, material property parameters can be optimized by optimizing the shape of the molten pool and thermal gradient based on the temperature distribution and fully utilizing various computer simulation methods, such as phase field simulation[35,36].

4.1. Control of shape parameters

In metal AM, the shape of a structure is designed in 3D-CAD, where any three-dimensional shape can be controlled. Optimizing the outer and inner shapes makes it possible to express the desired functional characteristics, including isotropy/anisotropy.

Figure 5 shows Young's modulus of a modeled object, in which 27 cubic elements (3×3×3) are combined arbitrarily along each side and powder/solid portions are selectively arranged, predicted by design and computer simulation[37]. The structure is expected to exhibit a triaxial anisotropic Young's modulus for each axis. Triaxial anisotropy can be achieved in real molded objects, with the expression of isotropy/anisotropy by such internal structure control determined by the number of supports parallel to the load as well as point/line/surface contact[37]. Adding such an internal structure directly controls the macroscopic mechanical properties of the structure, making it an extremely useful method of utilizing the characteristics of metal AM.

4.2. Control of material properties

Metal AM excels at free shape control as outlined in Section 4.1, and the unique directionality of the heat flux in the molten pool, cyclic melting/solidification, and thermal profile enable controlling material properties such as microstructure and atomic arrangement. A notable feature unique to metal AM is texture control, including single crystallization$^{6-18)}$. Direction control and single crystallization achieve anisotropic mechanical properties, such as Young's modulus, and enable the selective control of physical property values according to the application, even for the same material. Conventional monocrystal production methods are lengthy and cannot obtain sufficient shapes and sizes, thus limiting their commercialization. However, metal AM has yielded increased expectations for realizing large monocrystal products.

Single crystallization enables the creation of bone implants capable of suppressing stress shielding$^{2,4)}$. A β-type Ti alloy with a bcc structure exhibits a relatively low Young's modulus, even in a polycrystalline state, but single crystallization results in an anisotropic Young's modulus that depends on crystal orientation. As shown in Fig. 6, Young's modulus represents the minimum value along <001> direction[38,39]. The elastic stiffness constant (c') depends on valence electron density (e/a) (Fig. 6(a)), so Young's modulus E_{001} and its anisotropic E_{111}/E_{001} depend on e/a; as e/a decreases and approaches the value of 4, E_{111}/E_{001} increases and E_{001} decreases (Fig. 6(b)).

The Ti-15Mo-5Zr-3Al (mass%) alloy, with a small e/a of 4.10 and approved by the ISO (ISO 5832-14)[40], exhibits a low Young's modulus of approximately 85 GPa in a polycrystalline state. Furthermore, single crystallization results in E_{100} decreasing to 44.4 GPa[37], which is low compared to Young's modulus of cortical bone (~30 GPa); stress shielding is expected to be suppressed when <001> is placed parallel to the long axis of the long bone[2].

Applying the laser beam method and metal AM to this alloy enables selective shaping of the crystal growth orientation using scan strategy control. As shown in Fig. 7, the crystallographic orientation in the modeled object depends on the scan strategies X and XY; in either case, <001> can be preferentially oriented with a low Young's modulus in a specific direction in the modeled object[6]. Crystallographic orientation control is determined by the movement direction of the solid–liquid interface during solidification into the molten pool, stability of the smooth surface, and the priority of the crystal growth orientation. In this case, the <001> preferred crystal orientation exhibited a low Young's modulus of approximately 70 GPa, while Young's modulus of the <011> preferred crystal orientation was approximately 100 GPa; metal AM enabled the creation of objects with material anisotropy. Further increasing the integration of crystallographic orientation and optimal composition control that considers the evaporation of light elements is expected to result in Young's modulus approaching the theoretical value of single crystal and achieving a value similar to that of bone[6)].

5. Unique Material Structure Formation and High-Order Functionality Using Metal Additive Manufacturing

As outlined in Section 4.2, metal AM can be used to vary the crystallographic orientation from site to site depending on the scanning strategy[6)]. Therefore, unlike other processes, the material properties of each part of a product can be changed. Furthermore, controlling the shape of the molten pool and aligning the two crystal orientations enables forming a unique material structure, such as a layered structure with a fine periodicity of 100 μm. For example, Fig. 8 shows the atomic arrangement and mechanical properties of the SUS316L austenitic stainless-steel alloy and the anodic polarization curve in a 0.9 mass% aqueous NaCl solution[11)]. The modeled object forms a layered structure comprising two layers (main layer and sublayer) with different crystal orientations. The unique layered structure of SUS316L leads to a decrease in strain transmissibility at the interface because the stress transfer coefficient at the interface decreases from the value of 1, leading to increased strength[11)]. Furthermore, the quenching effect of laser beam metal AM imparts a corrosion resistance that significantly exceeds that of conventional materials by eliminating MnS-based precipitates and other causes of pitting corrosion, as shown in Fig. 8(c). These observations are also currently being confirmed in various other stainless-steel grades.

In metal AM, attention has recently focused on a new class of materials: high-entropy alloys (HEAs) comprising five or more elements. Strong solid-solution hardening is expected due to the high-entropy effect, but conventional melting/solidification methods exhibit strong segregation and do not exhibit ideal solid-solution strengthening. AM can achieve rapid cooling of approximately $\sim 10^7$ K/s when using a laser as a heat source; thus, the segregation prevention effect is recognized, as shown in Fig. 9. As a result, the expression of functionalities unique to metal AM based on microstructure control is ever-expanding, such as imparting high strength and shape by an ideal forced solid solution[13)].

6. Conclusion

Metal AM technology can control complex external shapes and their internal structure, microstructure, and atomic arrangement as well as design functions determined by these aspects. Metal AM can also control the microstructure and atomic arrangement of materials with customized material properties for each part, which is unique to metal AM. The simultaneous control of complex shapes and material properties according to each part can be considered a new high-value-added manufacturing process only achievable in the DX age. Additionally, metal AM technologies are expected to be widely used in the future for modeling bulk materials as well as a surface treatment technology involving atomic arrangement and microstructure control; thus, it has the potential to develop into a new surface control technology deeply related to thermal spraying technologies. Hence, custom-shaped objects can be created based on new ideas that control functions according to the bulk and surface parts of products that have been thus far designed based on their shape. Metal AM technologies are expected to further develop and expand into new markets, incorporating new product designs and development concepts unique to metal AM, including its concepts of isotropy and anisotropy.

Acknowledgments

This research was partly supported by the Strategic Innovation Promotion (SIP) / "Materials Integration" for Revolutionary Design System of Structural Materials (Yoshinao Mishima, PD) (management corporation: JST), Japan Society for the Promotion of Science Grant-in-Aid for Scientific Research (KAKENHI) (S) (JP25220912, JP18H052540).

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List of Captions

Fig. 1. Overview of Osaka University Anisotropic Design & Additive Manufacturing Research Center (Osaka University AM Center).

Fig. 2. Conceptual diagram of isotropy and anisotropy. The Osaka University AM Center considers anisotropy as its design concept and aims to “construct anisotropic materials science”.

Fig. 3. Orientation of unique collagen/apatite crystallites in cortical bone. The *c*-axes of collagen and apatite crystallites are self-organized in almost parallel to form a texture according to the bone location. Long and vertebral bones show uniaxial orientation, the skull shows two-dimensional orientation, and the mandible shows a complex orientation depending on the state of masticatory stress. Modified from the references[20, 21].

Fig. 4. Shape and material property (structure/atomic arrangement) parameters that can be controlled using metal AM.

Fig. 5. Young’s modulus simulation result of powder/solid composite. Freely selecting the number of solids of the 27 cubic elements and their positions enables the creation of more than

100 million combinations and a wide range of Young's modulus values. The composite exhibits isotropic/anisotropic Young's modulus values depending on the number of columns in each loading direction. This figure is an example of triaxial anisotropy.

Fig. 6. (a) Change in elastic stiffness constant (c') with valence electron density (e/a) and (b) associated change in Young's modulus anisotropy in β-type Ti-alloy single-like crystals. A low Young's modulus is achieved along the crystal orientation <001>, which has a low elastic modulus. (c) Stress shielding by bone implants can be suppressed, in which <001> is aligned with the bone axis. Modified from references[37, 38].

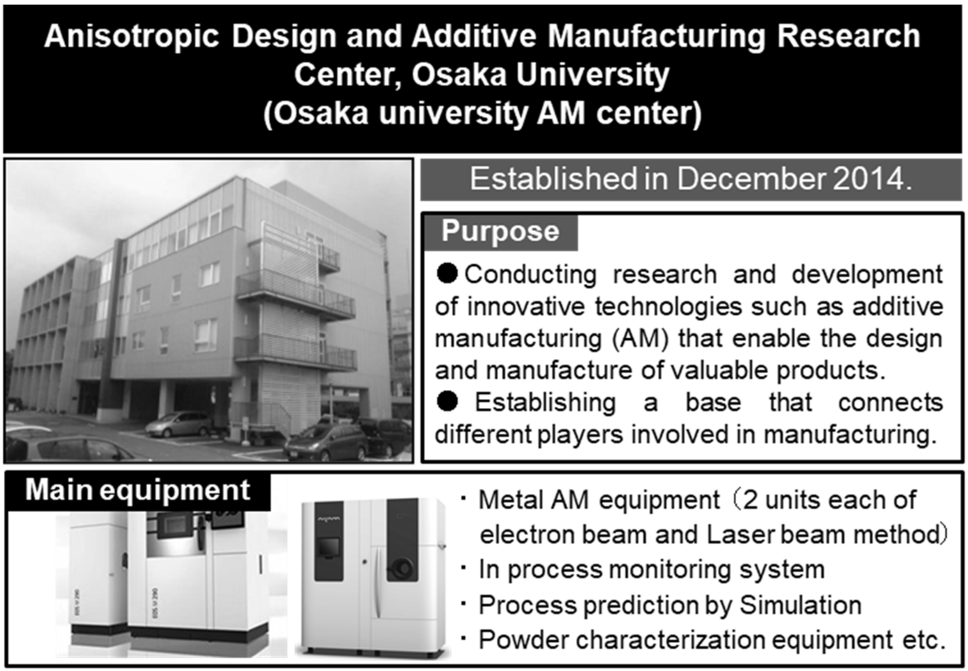
Fig. 7. Anisotropic atomic arrangement control for two scanning strategies (X scan and XY scan) for medical Ti-15Mo-5Zr-3Al alloy using laser beam metal AM. A low elastic modulus, established by the <001> orientation, is suitable for *in vivo* bone plates. Modified from reference[6].

Fig. 8. Excellent mechanical and corrosion properties of SUS316L. (a) Formation of a unique crystallographic lamellar microstructure and imparting of high (b) yield stress and (c) corrosion resistance to SUS316L austenitic stainless steel using

laser metal AM. Modified from references[11, 17].

Fig. 9. Suppletion of elemental segregation in multi-element high-entropy alloy (HEA). (a) Cast material of HEA and (b) comparison of segregation using laser AM, which can be suppressed by ultra-rapid cooling with laser AM. Modified from reference[13].

Figures



Anisotropic Design and Additive Manufacturing Research Center, Osaka University
(Osaka university AM center)

Established in December 2014.

Purpose

- ● Conducting research and development of innovative technologies such as additive manufacturing (AM) that enable the design and manufacture of valuable products.
- ● Establishing a base that connects different players involved in manufacturing.

Main equipment

- ・Metal AM equipment（2 units each of electron beam and Laser beam method）
- ・In process monitoring system
- ・Process prediction by Simulation
- ・Powder characterization equipment etc.

Fig. 1. Overview of Osaka University Anisotropic Design & Additive Manufacturing Research Center (Osaka University AM Center).

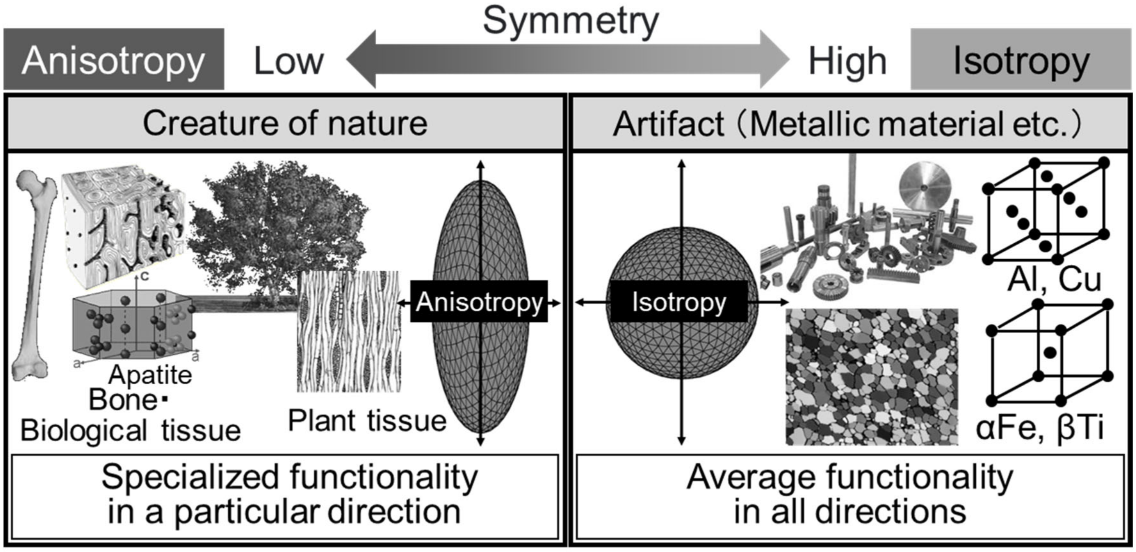


Fig. 2. Conceptual diagram of isotropy and anisotropy. The Osaka University AM Center considers anisotropy as its design concept and aims to “construct anisotropic materials science”.

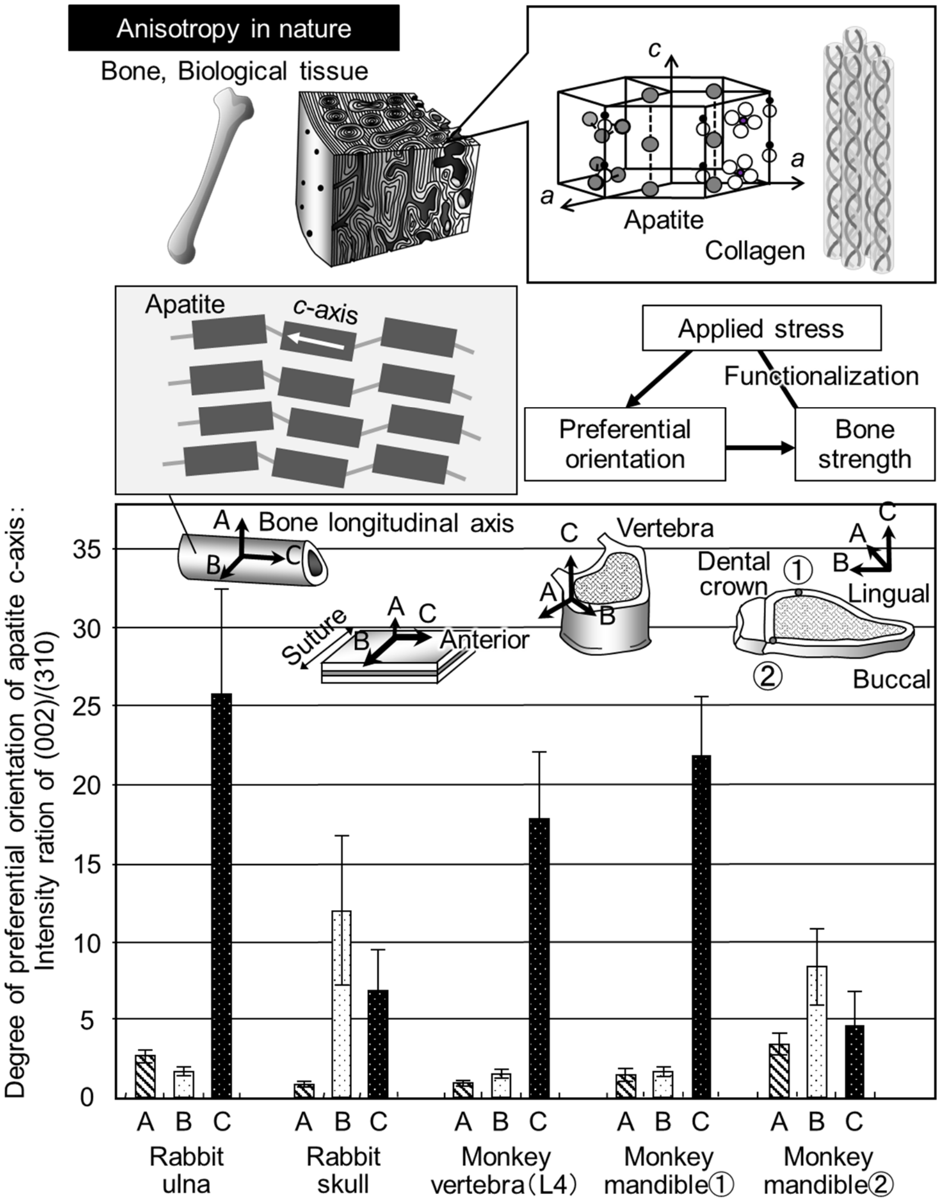


Fig. 3. Orientation of unique collagen/apatite crystallites in cortical bone. The *c*-axes of collagen and apatite crystallites are self-organized in almost parallel to form a texture according to the bone location. Long and vertebral bones show uniaxial orientation, the skull shows two-dimensional orientation, and the mandible shows a complex orientation depending on the state of masticatory stress. Modified from the references[20, 21].

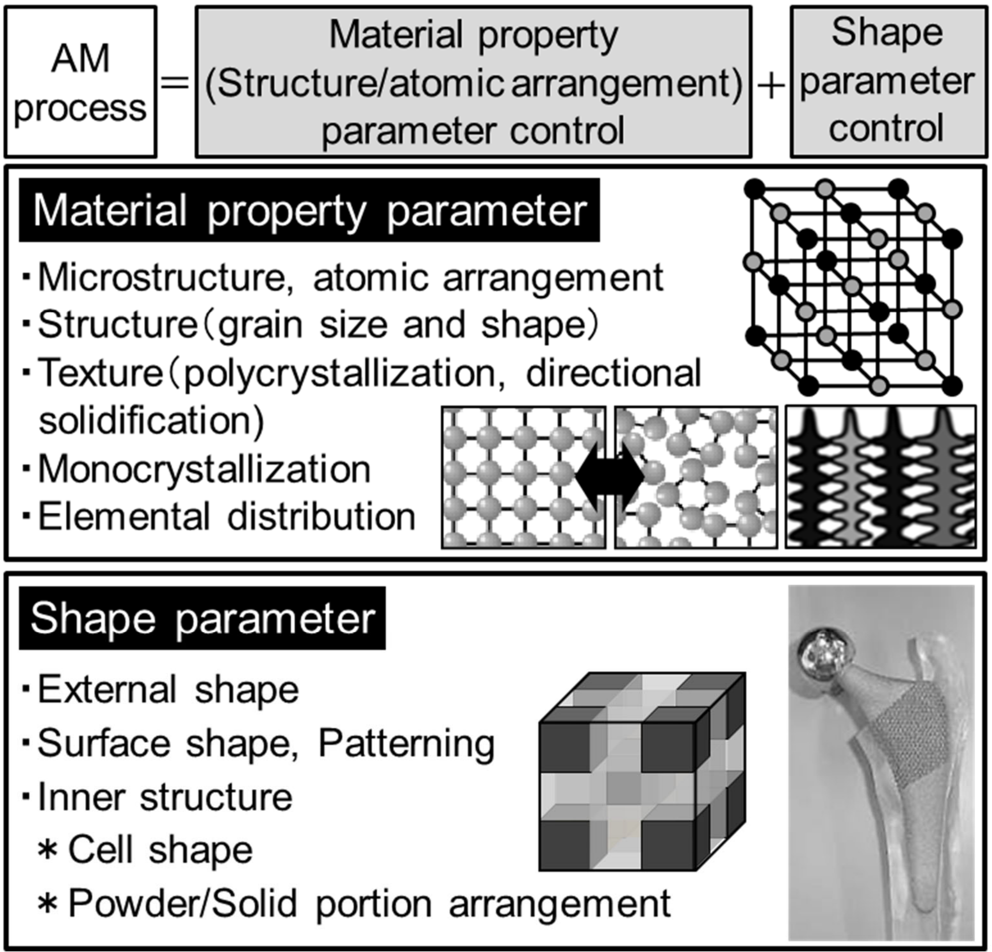


Fig. 4. Shape and material property (structure/atomic arrangement) parameters that can be controlled using metal AM.

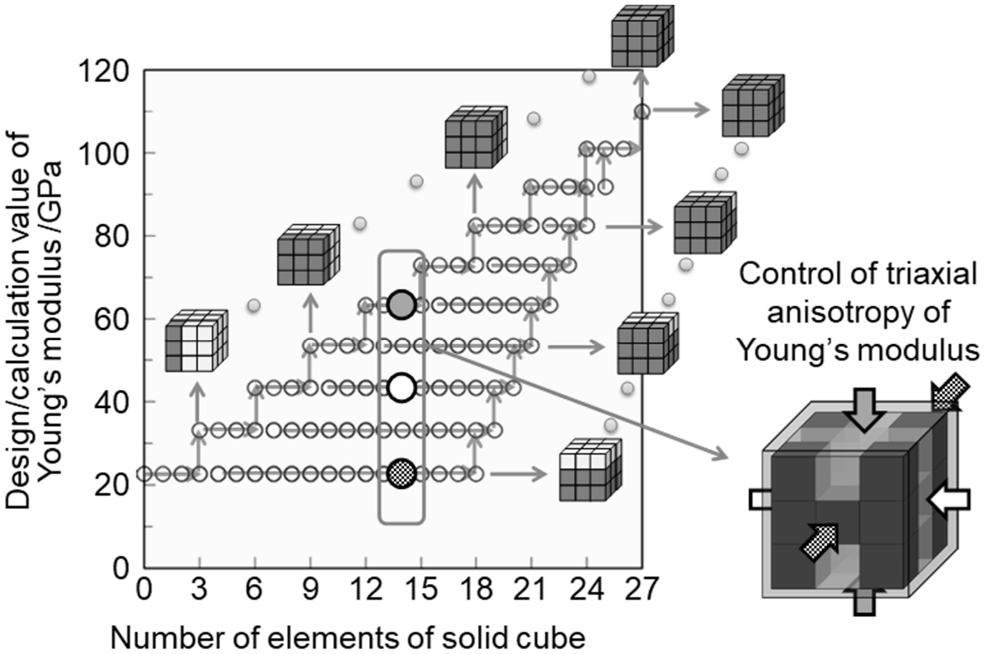


Fig. 5. Young's modulus simulation result of powder/solid composite. Freely selecting the number of solids of the 27 cubic elements and their positions enables the creation of more than 100 million combinations and a wide range of Young's modulus values. The composite exhibits isotropic/anisotropic Young's modulus values depending on the number of columns in each loading direction. This figure is an example of triaxial anisotropy.

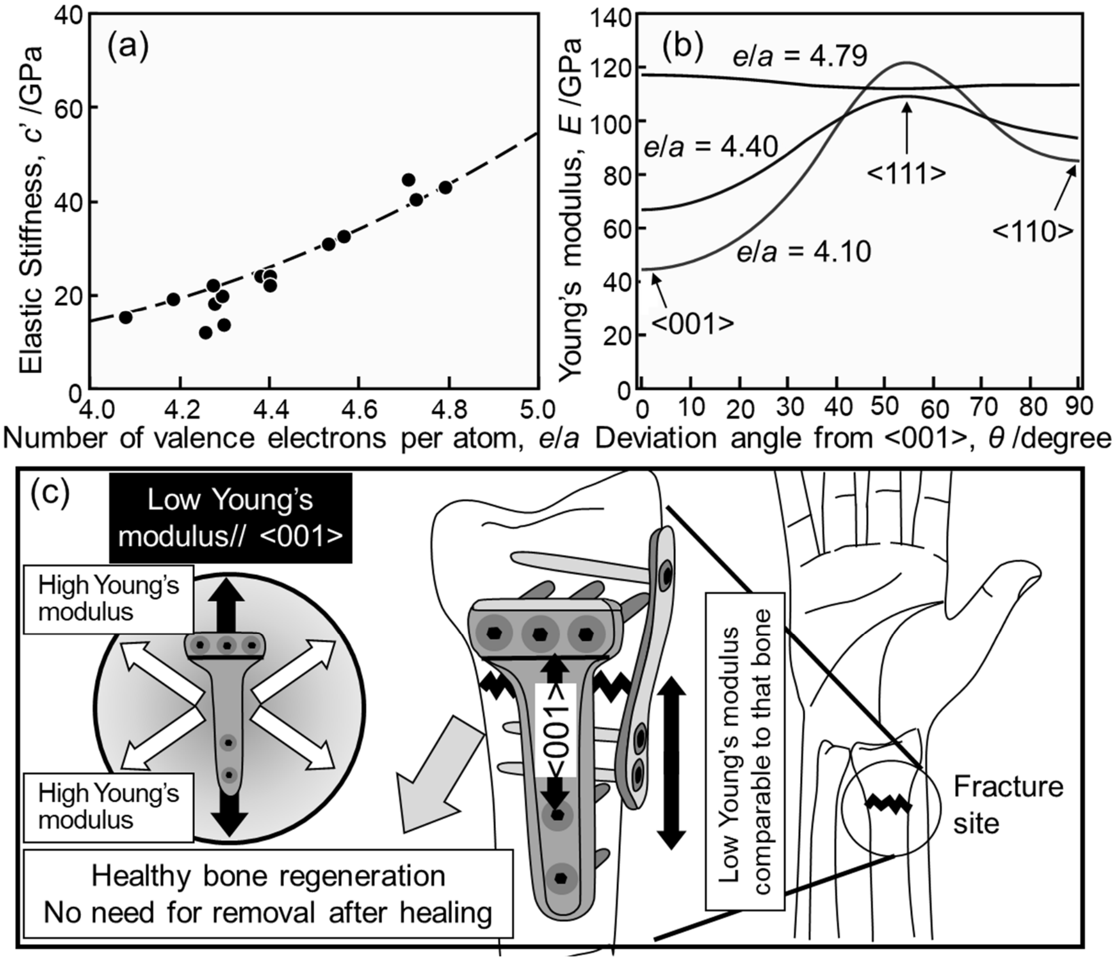


Fig. 6. (a) Change in elastic stiffness constant (c') with valence electron density (e/a) and (b) associated change in Young's modulus anisotropy in β-type Ti-alloy single-like crystals. A low Young's modulus is achieved along the crystal orientation <001>, which has a low elastic modulus. (c) Stress shielding by bone implants can be suppressed, in which <001> is aligned with the bone axis. Modified from references[37, 38].

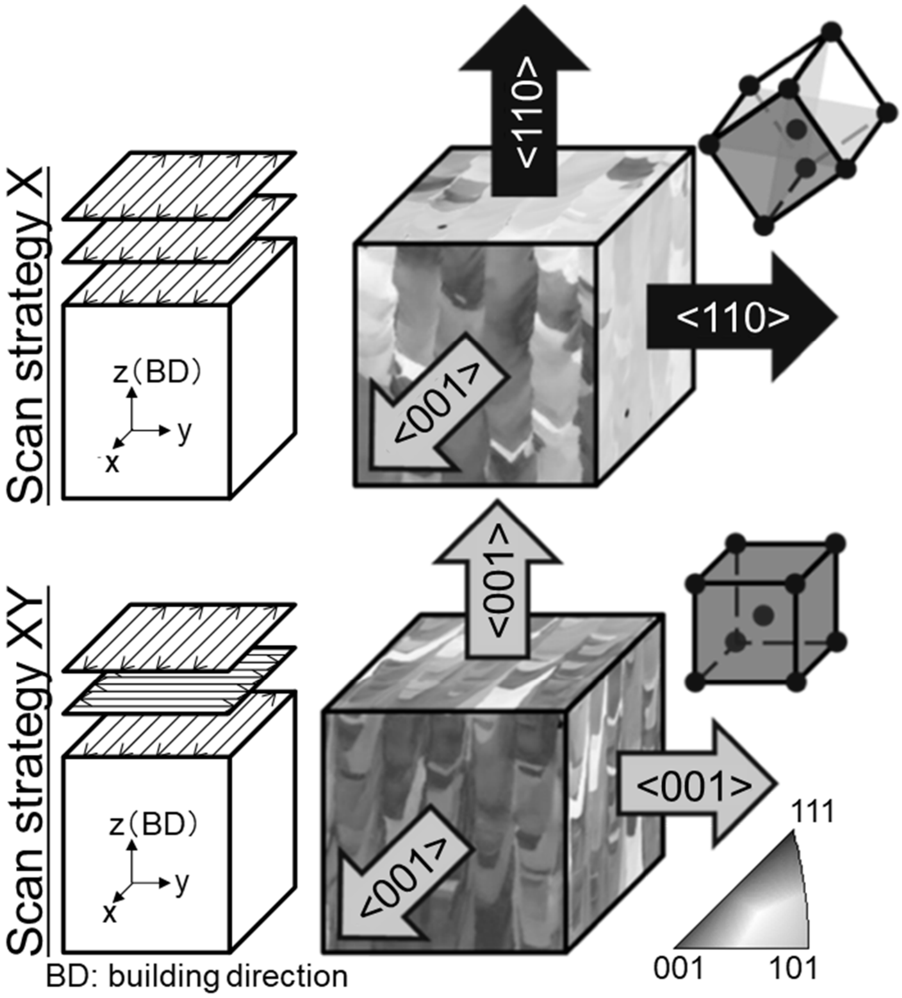


Fig. 7. Anisotropic atomic arrangement control for two scanning strategies (X scan and XY scan) for medical Ti-15Mo-5Zr-3Al alloy using laser beam metal AM. A low elastic modulus, established by the <001> orientation, is suitable for *in vivo* bone plates. Modified from reference6).

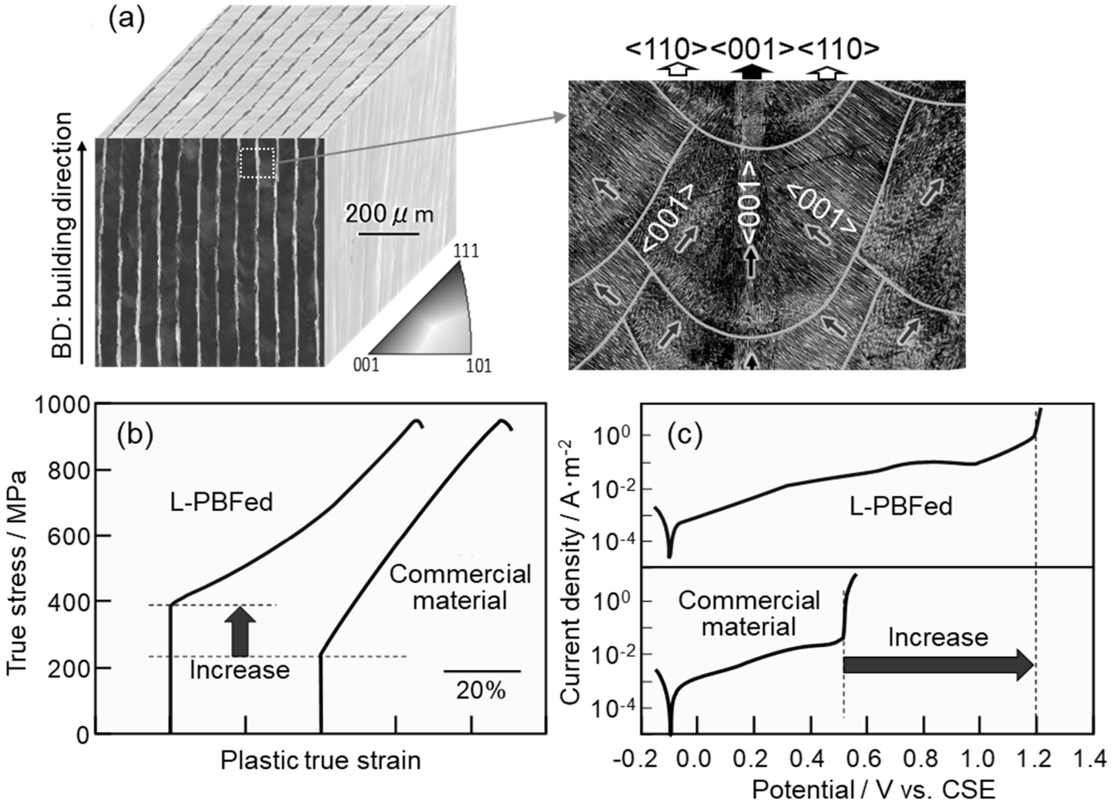


Fig. 8. Excellent mechanical and corrosion properties of SUS316L. (a) Formation of a unique crystallographic lamellar microstructure and imparting of high (b) yield stress and (c) corrosion resistance to SUS316L austenitic stainless steel using laser metal AM. Modified from references11, 17).

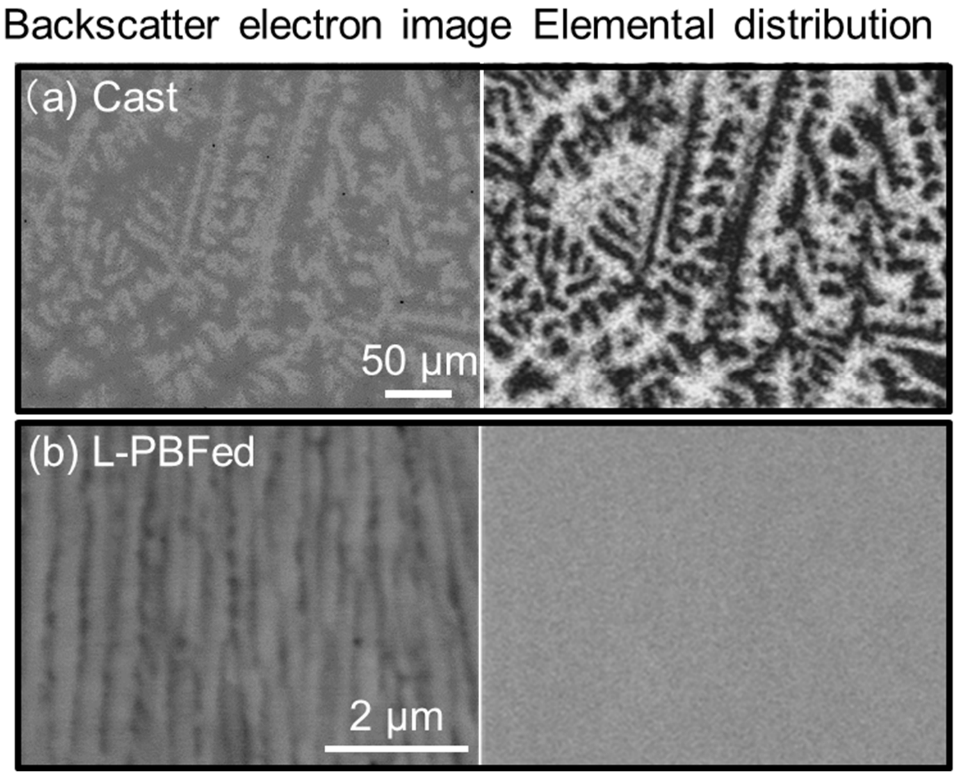


Fig. 9. Suppletion of elemental segregation in multi-element high-entropy alloy (HEA). (a) Cast material of HEA and (b) comparison of segregation using laser AM, which can be suppressed by ultra-rapid cooling with laser AM. Modified from reference[13]).