



A review on advances of high-throughput experimental technology for titanium alloys

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Abstract: Ti alloys, as leading lightweight and high-strength metallic materials, exhibit significant application potential in aerospace, marine engineering, biomedical, and other industries. However, the lack of fundamental understanding of the microstructure–property relationship results in prolonged research and development (R&D) cycles, hindering the optimization of the performance of Ti alloys. Recently, the advent of high-throughput experimental (HTE) technology has shown promise in facilitating the efficient and demand-driven development of next-generation Ti alloys. This work reviews the latest advancements in HTE technology for Ti alloys. The high-throughput preparation (HTP) techniques commonly used in the fabrication of Ti alloys are addressed, including diffusion multiple, additive manufacturing (AM), vapor deposition and others. The current applications of high-throughput characterization (HTC) techniques in Ti alloys are shown. Finally, the research achievements in HTE technology for Ti alloys are summarized and the challenges faced in their industrial application are discussed.

Key words: titanium alloys; high-throughput; microstructure; mechanical properties

1 Introduction

Ti and its alloys have become key metallic materials in aerospace, marine engineering, and biomedicine due to their low density, high specific strength, excellent corrosion resistance, heat resistance, and biocompatibility [1–6]. As service conditions become increasingly demanding, the performance requirements for Ti alloys are also heightened, driving the development of next-generation Ti alloys [7–9]. This poses a significant challenge, as the traditional “trial and error” approach to material design is limited by time, manpower, and resources, severely hindering the advancement of high-performance Ti alloy.

To address this challenge, the U.S. government proposed the Materials Genome Initiative (MGI) in

2011, which focuses on high-throughput computing, high-throughput experimental (HTE) technology, and the application of material big data technology [7]. HTE technology facilitates the efficient preparation and characterization of a large number of samples in a short period. Its fundamental principle is to transform the traditional sequential iterative research process into an efficient parallel process, achieving qualitative changes in material research efficiency through quantitative changes. The successful application of HTE technology is not isolated; it requires integration and development with computational simulation and material databases. This integrated strategy not only improves the efficiency of materials research and development (R&D) but also revolutionizes the material design philosophy towards “design on demand”. HTE techniques play a crucial role in this

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revolution by providing abundant experimental data for material simulation and calculation, enriching material databases, and optimizing calculation models. In summary, HTE techniques offer the fundamental experimental basis for the entire MGI, thereby bridging simulation and final material output.

In this review, we introduce the HTE technologies used, or potentially used, in Ti alloys, including high-throughput preparation (HTP) and high-throughput characterization (HTC). We emphasize that HTP primarily enables the composition gradient and process condition gradient of Ti alloys, while HTC involves the rapid screening of microstructures and corresponding mechanical properties. Specifically, Ti alloy samples with different composition gradients can be prepared by diffusion multiple, additive manufacturing (AM), and vapor deposition. Meanwhile, deformation and heat treatment processes are precisely controlled using double-cone compression, gradient rolling, and gradient heat treatment technologies. Additionally, HTC methods such as nanoindentation and synchrotron radiation X-ray diffraction (XRD) are introduced to comprehensively evaluate the microstructure and mechanical properties of Ti alloys. At the end of the review, we present the future vision of HTP and HTC for Ti alloys.

2 High-throughput preparation techniques

HTP technology is an advanced experimental design method that significantly enhances experimental efficiency and accuracy by simultaneously processing a large number of samples or data. This technology is not only applicable to fields such as biology, chemistry, and medicine, but also has been widely applied in the study of Ti alloys and other non-ferrous metals in recent years. In the R&D of Ti alloys, HTP technology accelerates material screening, optimizes process parameters, and facilitates a deep understanding of material properties. Table 1 summarizes the applications of these technologies in Ti alloys across multiple dimensions.

2.1 Acquiring component gradient

Subtle changes in composition can lead to significant variations in the phase composition and

microstructure of Ti alloys. Within a wide compositional space, numerous possible combinations exist, and conventional methods can no longer meet the needs for rapid screening of Ti alloy compositions on a large scale. HTP preparation technologies capable of obtaining composition gradients have become essential tools for Ti alloy composition design. They include the long-established diffusion multiple techniques, as well as more recently optimized methods such as additive manufacturing (AM), vapor deposition, and other synthesis technologies combined with high-throughput (HT) concepts. The application of these technologies accelerates the composition design of Ti alloys and reduces development costs.

2.1.1 Diffusion multiple

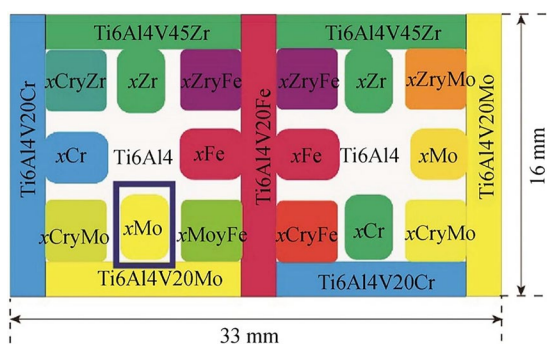
Diffusion defines the pathway for mass transport in solids and is critically related to most thermodynamic processes in alloys, including recovery, recrystallization, homogenization, and solid-state phase transformation. Traditional binary diffusion couples and ternary diffusion multiples have long been used to determine diffusion coefficients and phase diagrams [8,9]. With technological advancements, the diffusion multiple method has evolved, focusing on composition gradients and the resultant multi-element alloys or intermetallic compounds through long-term annealing [10–12]. This provides a powerful tool for studying the phase diagrams, kinetics, and composition–structure–property relationships of bulk alloys.

The precise composition design of Ti alloys is challenging because subtle adjustments in composition can significantly impact microstructure and properties. The diffusion multiple method can achieve a continuous distribution of alloy components over a wide range, providing abundant microstructure and property data for the precise control of Ti alloy composition. ZHAO [13,14] used thermal interdiffusion to produce complete single-phase compositions in binary and ternary systems and mapped detailed composition–structure–property relationships through microscale property measurements, providing scientific evidence for Ti alloy design and optimization. WU et al [15] successfully designed Ti64–xMo alloys using diffusion multiple techniques (Fig. 1) and thermodynamic databases, analyzing their phase composition and properties. Specifically, after aging

Table 1 Comparison of HTP synthesis methods

| HTP method | Number of samples | Contiguity | Gradient condition range scale | Required time | Compatible HTC | |
|--|-----------------------------|--------------------------|--------------------------------|------------------------|---|--|
| Getting elemental composition gradient | Diffusion multiples | Single sample | Continuous | Microscale | Depends on temperature, elemental species (often >24 h) | SEM, EDS, EBSD, nanoindentation, Micro-XRF, EPMA |
| | Additive manufacturing | Single or series samples | Continuous or discrete | Microscale, macroscale | Depends on numbers, size, and thickness (often <12 h) | All conventional characterization methods |
| | Vapor deposition | Single or series samples | Continuous or discrete | Microscale, mesoscale | Depending on thickness (often <10 h) | SEM, EDS, EBSD, nanoindentation |
| | Spark plasma sintering | Single sample | Continuous | Microscale | Depends on temperature, elemental species (often >24 h) | SEM, EDS, EBSD, nanoindentation, Micro-area XRD, Micro-XRF |
| | Gradient continuous casting | Single sample | Continuous | Macroscale | Depends on size, elemental species | All conventional characterization methods |
| Obtaining process condition gradient | Double-cone compression | Single sample | Continuous | Microscale | Depends on test temperature, sample size (often <1 h) | SEM, EDS, EBSD, TEM, nanoindentation |
| | Gradient rolling | Single sample | Continuous | Microscale | Depends on test temperature, sample size (often <1 h) | SEM, EDS, EBSD, TEM nanoindentation |
| | Gradient heat treatment | Single sample | Continuous | Microscale | Depends on heat treatment time (often <10 h) | SEM, EDS, EBSD, TEM, nanoindentation |

Abbreviation: EDS–Energy dispersive X-ray spectroscopy; XRF–X-ray fluorescence; OM–Optical microscopy; NA–Not applicable; TEM–Transmission electron microscope; EBSD–Electron back scatter diffraction; SEM–Scanning electron microscopy; XRD–X-ray diffraction; EPMA–Electron probe microanalysis

**Fig. 1** Diffusion multiple section diagram of Ti₆Al₄V–Ti₆Al₄V₄₅Zr, Ti₆Al₄V₂₀Mo, Ti₆Al₄V₂₀Fe and Ti₆Al₄V₂₀Cr [15]

treatment at 600 °C, the Ti₆Al₄–6Mo alloy exhibited high strength and moderate plasticity, validating the effectiveness of the alloy design.

DING et al [16] developed low-cost, high-strength Ti–Al–Cr alloys through diffusion couple

experiments and CALPHAD calculations, with Ti–6Al–10.9Cr alloy showing the best mechanical properties. This work demonstrated the power of diffusion multiple techniques in alloy design, especially for Ti alloys. LING et al [17] successfully established a mechanical properties database for Ti–Nb–Zr–Mo and Ti–Nb–Zr–Ta alloys using diffusion couple technology and advanced testing methods. This result provides strong data support for the application of Ti alloys in the medical field, showcasing the potential of diffusion multiple techniques in alloy performance evaluation. WEN et al [18] prepared a Ti–Nb–Zr–Hf diffusion couple after annealing at 1273 K for 25 h, exploring the composition–mechanical property relationships over a wide composition range using EPMA and nanoindentation techniques. They also determined the interdiffusion coefficients of the alloys, established a mechanical properties

database as a function of composition, and created mechanical and diffusion characteristic maps, providing critical information for biomedical and aerospace applications. In terms of alloy performance optimization, DING et al [19] employed HT technology to design an ultrahigh strength Ti alloy with excellent toughness, providing a new direction for the development of Ti-based alloys. ZHU et al [20] studied the alloying effects of Mo and Cr on the properties of Ti alloys and predicted the microstructural characteristics of the alloys using machine learning (ML) techniques, significantly improving the efficiency of component screening, and the fitting results are shown in Fig. 2.

Additionally, WU et al [21] used diffusion

couple technology to thoroughly investigate the relationship between the microstructure and hardness of Ti6Al4V alloy. GAO et al [22] revealed the effect of Fe content on the hardness of T20C alloy by designing diffusion couples with a gradient in Fe content, providing an important reference for alloy composition optimization. WU et al [23], based on the pseudo-spinodal mechanism, identified the composition range corresponding to the ultrafine grains of TM- x Mo- y V alloy. The designed TM-6Mo-3V alloy has a yield strength of 1411 MPa and an elongation of 6.5%. All these studies provide a theoretical basis for alloy performance optimization and highlight an effective method for designing high-performance Ti alloys.

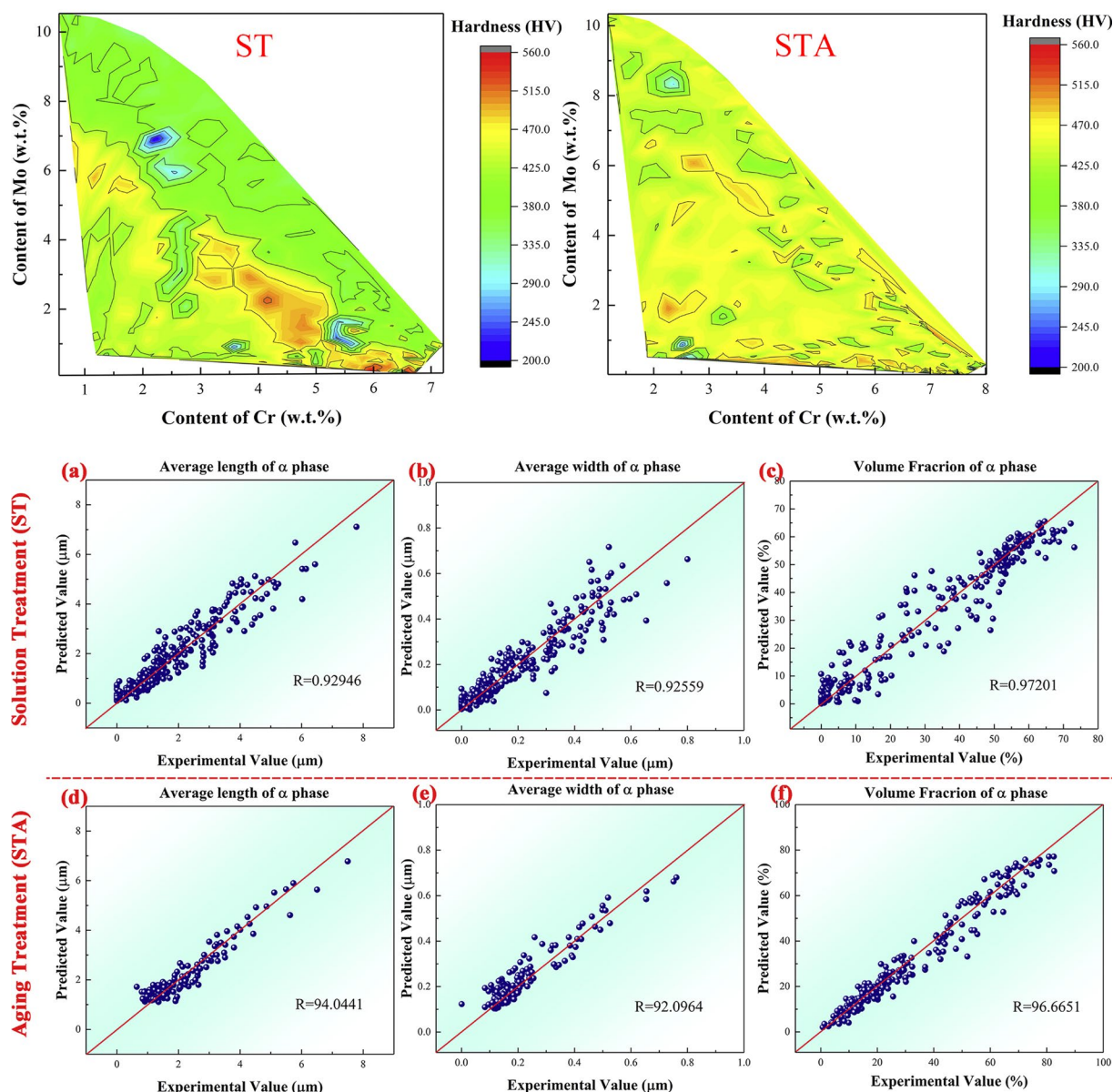


Fig. 2 Correlation coefficient between experimental and predicted values for solution treatment (a–c) and aging treatment (d–f): (a, d) Average length of α phase; (b, e) Average width of α phase; (c, f) Volume fraction of α phase [20]

In brief, the diffusion multiple technology has become a key technology for the design and innovation of Ti alloys due to its unique advantages. This technology not only optimizes compositional gradient design of Ti alloys but also combines advanced testing techniques to build a mechanical properties database, providing the important references for the application of Ti alloys.

2.1.2 Additive manufacturing (AM)

Additive manufacturing (AM), with its high-precision processing capabilities, is suitable for preparing samples with complex structures or compositional gradients and different microstructures [24,25]. By utilizing laser metal deposition [26] and laser beam melting [27], HTP of metallic materials can be effectively achieved.

GONG et al [28] utilized laser-engineered net shaping (LENSTM) technology and modified the motion control file to change the powder flow rates at specific layers to pre-determined powder flow rate set points, enabling the HT preparation of TiNi alloys with compositional gradients. The alloy composition ranged from pure Ti to Ti-12wt.%Ni, and HT spherical indentation testing was employed to efficiently establish the structure-property relationships, as shown in Fig. 3. In addition, they applied the same method to high-throughput fabricate Ti-Mn alloys with compositional gradients (with Mn content varying from 0 to 12% along the axial direction of the cylinder), and a large sample database was established, correlating Mn content with the α - β microstructure [29].

MENG et al [30] developed a pulsed laser

melting water-cooled copper mold technology to efficiently manufacture Ti-6Al-4V alloy samples and used three-dimensional simulation to explore the relationship among melt pool heat transfer, fluid flow, and process parameters, providing theoretical guidance for laser manufacturing. ZHANG et al [31] combined DED with ML to develop a HT technology for revealing the relationships among the composition, microstructure, and mechanical properties of Ti-Al-V alloys, as shown in Fig. 4. By preparing 144 sets of ternary alloys and evaluating their microstructure, hardness, and yield strength, they accelerated the design process of Ti alloys based on the Ti-Al-V system using the obtained quantitative data.

Compared to the diffusion multiple method, AM has the following significant advantages for sample preparation:

(1) High time efficiency: AM significantly shortens the sample preparation time, eliminating the need for a prolonged diffusion process.

(2) Strong design flexibility: AM can directly produce samples with compositional gradients and can be used for mold manufacturing, demonstrating strong design capabilities.

(3) Simple requirements for the preparation of samples: Unlike the ultra-high precision fitting required for bulk samples in diffusion couples, AM has simpler requirements for samples, reducing preparation difficulty.

However, AM also has the following disadvantages:

(1) Post-processing requirements: AM samples

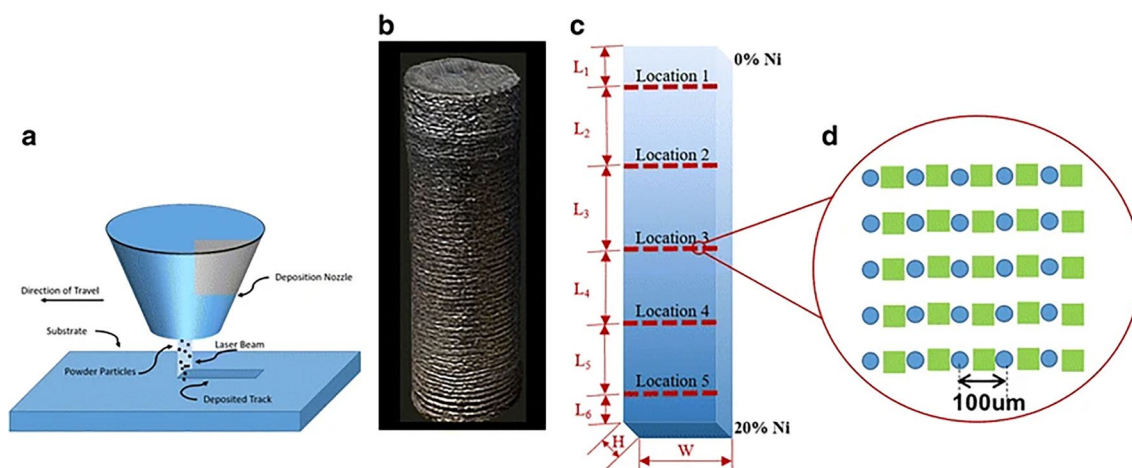


Fig. 3 HT preparation of TiNi alloys with compositional gradients [28] (Reproduced with permission from Springer Nature Ltd.)

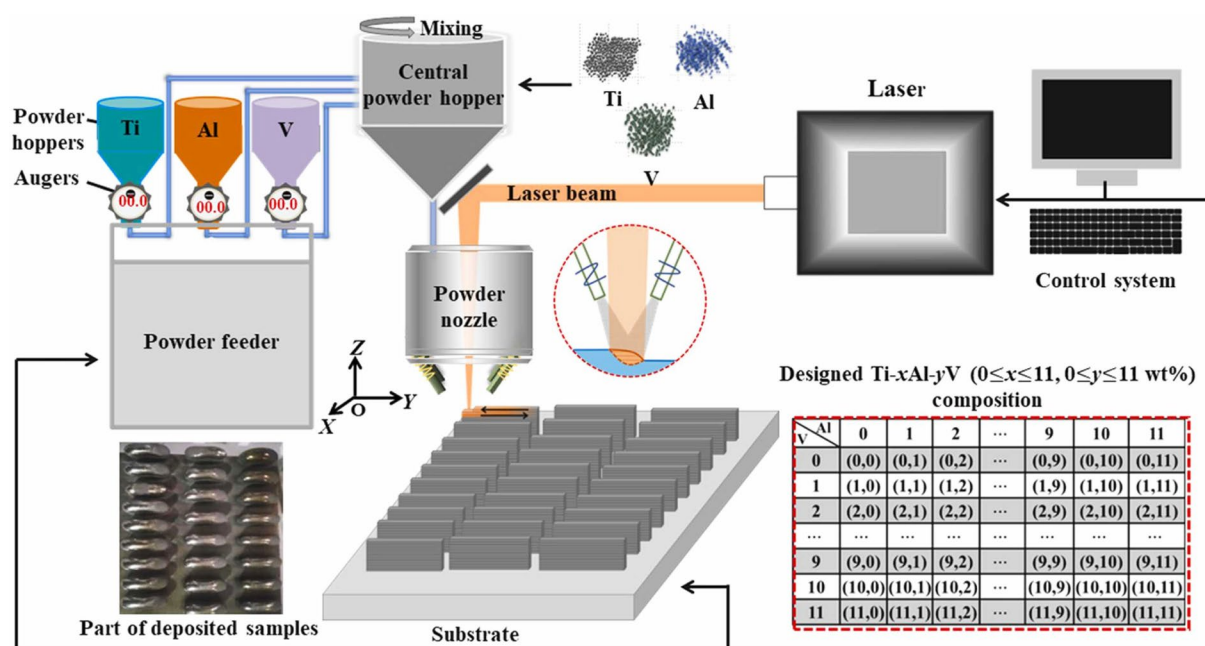


Fig. 4 Schematic diagram of composition adjustment of Ti-xAl-yV ($0 \leq x \leq 11, 0 \leq y \leq 11$, wt.%) alloy prepared by HT method of DED [31] (Reproduced with permission from Elsevier Ltd.)

may require post-processing to improve the surface quality. However, current post-processing techniques are still in need of improvement, which may affect the consistency of sample quality.

(2) Material and size limitations: AM is constrained by the materials and sizes of workpieces. Some materials may not be suitable for AM, and it may not be applicable for the production of large gradient samples.

(3) Internal quality risks: During the process of AM, internal defects such as micro-voids may occur, which can impact the overall quality and performance of the samples.

2.1.3 Vapor deposition

Compared to bulk materials, thin film materials more easily achieve in-plane compositional gradients. This facilitates HTC of the microstructure and properties of materials, enabling rapid phase diagram mapping and aiding researchers in screening for material compositions with superior properties. Traditional material growth methods are usually slow and time-consuming, typically allowing for the preparation and characterization of only one material at a time. This limits the rapid exploration of the composition and properties of solid materials. Consequently, HT growth methods for thin film materials have emerged, such as co-deposition and physical

masking techniques.

The co-deposition method utilizes changes in the relative angles and positions of the deposition sources and the substrate to deposit multiple alloy compositions on the same substrate, preparing material samples with a gradient distribution of alloy compositions. The physical masking method can be divided into continuous masking and discrete masking. The former controls the deposition rate of the sample by controlling the substrate stage and then prepares compositionally controlled multi-alloy combination samples using coating and time-shifted masking techniques. The latter combines coating and masking techniques to obtain different discrete composition samples using continuous masking.

XIANG et al [32] developed a combinatorial material preparation technique that combined thin film deposition and discrete masking, which has been applied to the parallel synthesis of spatially addressable solid-state material libraries. The HT material growth methods mentioned above have been widely used in research to screen compositions for optimizing material properties of thin film since then. Initially, these methods were mainly focused on screening materials for applications such as high critical temperature superconductors and phosphors [33–35].

Currently, HT material growth methods have been applied in the research of Ti and its alloys. YAN et al [36] used a combinatorial co-sputtering deposition and physical masking method to prepare Ti–Nb–Zr ternary alloys. By optimizing the composition of the materials, they screened out a Ti₃₄Zr₅₂Nb₁₄ alloy with low elastic modulus and excellent corrosion resistance. PEI et al [37] employed a three-target magnetron sputter co-deposition HT method to prepare a continuous composition spread (Nb–Ti)_{1–x–y}Si_xCr_y alloy thin film, resulting in a combinatorial materials library of (Nb–Ti)_{1–x–y}Si_xCr_y covering (9.34–32.31)Si–(10.43–20.27)Cr (at.%). A schematic diagram of this method is shown in Fig. 5.

HASAN et al [38] synthesized a compositionally gradient Ni–Ti–Cu–V thin film library on thermally oxidized Si wafers using the magnetron co-deposition. They determined composition-related phase transition temperatures and microstructures using the HT wavelength dispersive spectroscopy, synchrotron X-ray diffraction, and temperature-dependent resistance

measurements. MIYAGAWA et al [39] used sputtering to prepare Ti–Cu combinatorial thin films with different compositions and measured the mechanical properties and distributions using nanoindentation. They combined the neural network potential and ab initio molecular dynamics simulations to study the mechanisms of mechanical properties from an atomic-scale perspective.

In recent years, the development of artificial intelligence and automation technologies has provided a shortcut for accelerating material preparation. Automated material laboratories utilize automation and machine learning (ML) methods to drive the rapid synthesis and screening of materials. Currently, autonomous laboratories are using modular robotic platforms to conduct automated experiments, combined with ML and active learning to plan and interpret experimental results [40,41]. They also analyze the reasons for synthesis failures and provide solutions to improve current material screening and synthesis design techniques. However, current automated material laboratories still use serial experimental methods, resulting in

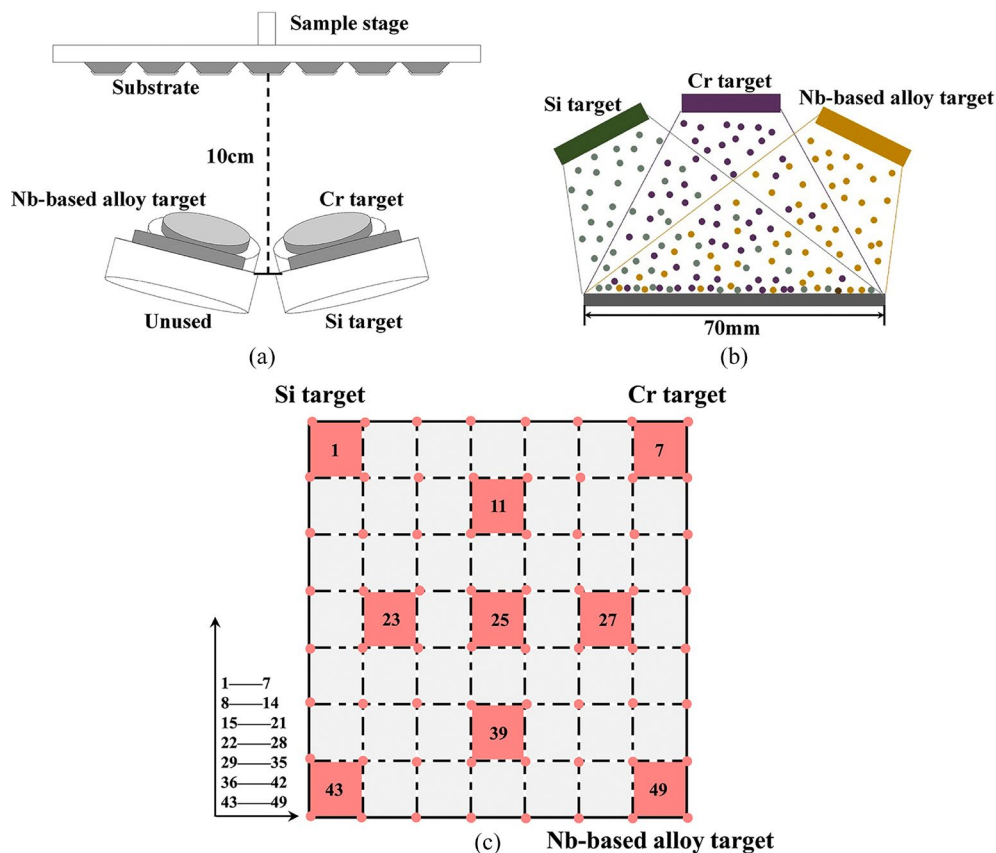


Fig. 5 Schematic diagram of co-deposition by three target magnetron sputtering: (a) Three-dimensional view of co-deposition; (b) Front view and deposition process; (c) Schematic diagram of position of matrix on sample table [37] (Reproduced with permission from Elsevier Ltd.)

low experimental throughput. Applying HT material growth methods to autonomous laboratories is a feasible approach to enhance the efficiency of material research.

2.1.4 Other preparation methods

In the field of material preparation, diffusion multiples, additive manufacturing (AM), and vapor deposition have highlighted their core value. However, research should not focus on emerging technologies only; in-depth exploration and optimization of classical techniques are equally crucial. By fine-tuning and enhancing these classical techniques, the application scope of alloy materials can be further expanded.

ZHAO et al [42] designed 19 combinatorial alloy compositions, using customized honeycomb array cells made of Ni or Ti foil to vacuum encapsulate powder mixtures, and consolidated them at 1050 °C and 120 MPa for 10 h via hot isostatic pressing (HIP). The schematic diagram of this method is shown in Fig. 6. The resulting alloys were free of macroscopic defects, with actual compositions consistent with nominal values, and their phase composition and microstructure were similar to those produced by traditional bulk alloy processes.

HE et al [43] developed a HTP method for the high-performance thermoelectric materials, preparing gradient bulk samples using spark plasma sintering and annealing, providing effective guidance for the development of new thermoelectric materials. LI et al [44] and ZHANG et al [45] proposed a HTP technique using gradient continuous casting, producing various alloy samples with gradient variations and quickly obtaining performance data by combining plastic processing and characterization methods. This technique is expected to be applied to the HTP of Ti alloys, high-entropy alloys, steels, and other new materials, promoting the rapid screening of high-performance new alloy materials. DAI et al [46,47] used a spiral HT gradient continuous casting process (Fig. 7) to produce new biodegradable Zn- x Cu- y Ti ($x=0.001$ – 2.72 and $y=0.03$ – 1.21 , wt.%) alloys and Zn- x Cu- y Ti- z Mo alloys ($x=0.02$ – 0.10 , $y=1.22$ – 1.80 , and $z=0.04$ – 0.06 , wt.%), and evaluated their as-cast and rolled microstructures, mechanical properties, and corrosion performance. In vivo experiments demonstrated that these alloys are suitable candidates for medical implants. This study not only made significant progress in the development of biodegradable alloys but also

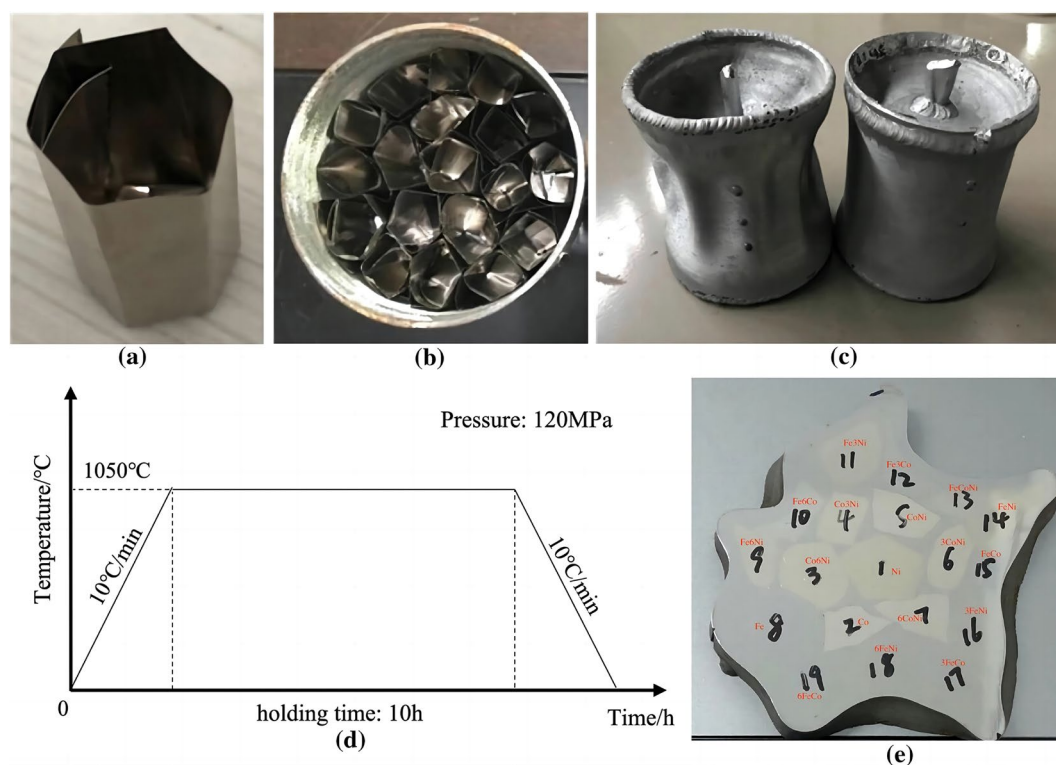


Fig. 6 Schematic diagram of HIP method: (a) Thin foil column; (b) Honeycomb array sleeve; (c) Post-pressing sleeve; (d) Process flow; (e) Post-pressing polishing section [42] (Reproduced with permission from Springer Nature Ltd.)

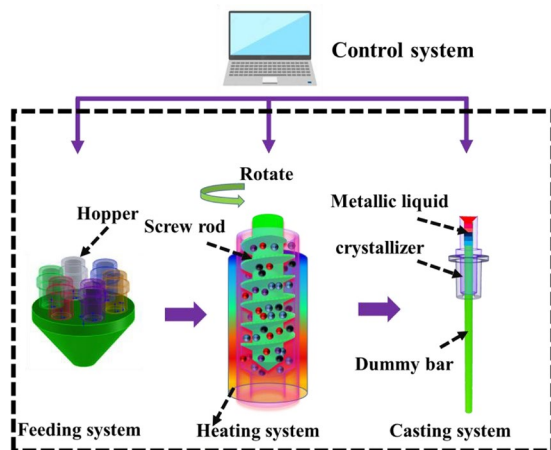


Fig. 7 Schematic diagram of spiral HT gradient continuous casting [47] (Reproduced with permission from Elsevier Ltd.)

proposed an efficient production method, marking a key step forward in biomedical engineering.

2.2 Acquiring deformation condition gradient

Plastic deformation is crucial in the production of Ti alloys, significantly influencing the shaping and performance of Ti alloy workpieces. However, the current plastic deformation process for Ti alloys

suffers from inefficiencies, including multiple processing passes and poor stability, which results in high production costs and low material yield, thereby limiting their broader application. Additionally, the complex deformation mechanisms of Ti alloys complicate the design of process parameters needed to achieve the desired microstructure. HT deformation testing technology offers a solution to these challenges. Techniques such as double-cone compression and gradient rolling can produce strain and microstructure gradients within a single sample, allowing for the rapid establishment of relationships between deformation conditions and microstructure during forging and rolling. This approach provides innovative methods for the rapid design and optimization of Ti alloy processing techniques.

2.2.1 Double-cone compression

The double-cone compression test is an HT compression deformation technique that produces gradient strain and corresponding microstructure variations within a single sample. By uniaxially compressing a double-cone sample, as shown in Fig. 8(a), the microstructure varies continuously

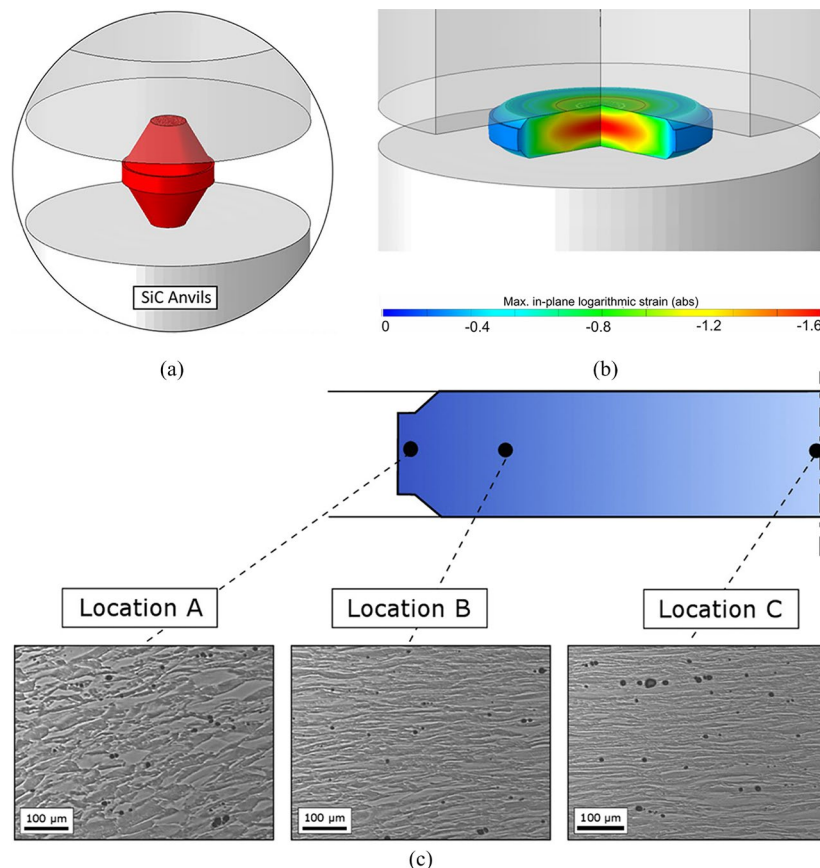


Fig. 8 Double-cone compression test: (a) Sample geometric characteristics; (b) Strain distribution obtained by FEM; (c) Continuously changing microstructures [48] (Reproduced with permission from Elsevier Ltd.)

along the radial direction of the sample's cross-section (Fig. 8(c)) [48]. This heterogeneity arises from the controlled and continuously varying strain field due to the unique geometric dimensions of the double-cone sample. Since direct measurement of the radial strain gradient during the compression test is not feasible, finite element modeling (FEM) is typically employed to determine the equivalent strain distribution, as illustrated in Fig. 8(b). Compared to the traditional cylindrical sample compression tests, the double-cone compression test achieves continuous changes in the strain field and microstructure, eliminating the need for multiple samples with different deformation conditions for repeated tests. This approach shortens development time and reduces costs, providing a more economical and efficient experimental method for studying microstructure evolution during the compression (forging) process of Ti alloys and revealing their deformation mechanisms.

The application of double-cone compression test in Ti alloys primarily focuses on studying hot compression (forging) deformation behavior, as most Ti alloy ingots undergo initial processing through hot forging. By using the double-cone compression test to investigate microstructure evolution during the hot forging process, the relationship between deformation conditions and microstructure can be rapidly established. This approach offers data support and a theoretical basis for the design and optimization of Ti alloy forging processes, benefiting energy consumption reduction and material yield improvement. CALVERT et al [49] used large double-cone samples to rapidly assess the effectiveness of the FAST-forge process on the Ti-5553 alloy, finding that the resulting microstructure was similar to that achieved by traditional β forging, demonstrating its industrial-scale application potential. LIM et al [50] utilized double-cone samples to study the effects of strain and strain rate on β annealing of the Ti-55511 alloy at supercritical deformation temperatures. They discovered that deformation conditions influenced mechanisms such as dynamic recovery (DRV), dynamic recrystallization (DRX), and texture evolution, which subsequently affected static recrystallization during annealing. They also investigated the effects of strain and deformation rate on the

spheroidization of lamellar microstructures, crystallographic texture, and boundary dislocations, establishing a simple processing design map to determine the relationship among microstructure, texture evolution, and reduction during hot compression at 850 °C [51].

The double-cone sample can reveal the variation of thermal deformation mechanisms with strain and assist researchers in constructing deformation mechanism maps. Inspired by Ashby plots, these maps visually reflect the deformation mechanisms occurring in alloys under different deformation conditions, aiding in the design of process parameters for target microstructures [52,53]. Double-cone compression technology was utilized to investigate the thermal deformation behavior of various Ti alloys [54–57]. The continuously varying microstructural results were used to reveal the evolution of deformation mechanisms. Based on graphical analysis combined with characterization techniques such as EBSD and TEM, critical points of different deformation mechanisms were identified. By comparing these discrete critical points with strain distribution maps obtained from FEM, the critical strain was quantitatively analyzed at which deformation mechanisms changed. Furthermore, hot processing maps were constructed by using dynamic material models based on hot deformation stress–strain data [58]. Overlaying deformation mechanism maps with hot processing maps allowed for quantitative analysis of how deformation conditions affect deformation mechanisms. Deformation mechanism maps for TC18, TA16, TA16/SiC_p alloys, and TiZrV medium entropy alloys are shown in Figs. 9(a–d).

2.2.2 Gradient rolling

Double-cone technology facilitates the rapid acquisition of microstructural evolution during the forging of Ti alloys and reveals their deformation mechanisms. For rolling processes, gradient rolling techniques have also been proposed. These techniques alter the geometric characteristics of samples to achieve a gradient distribution of microstructure and strain field during rolling. A “wedge-shaped” sample, suitable for HT research in rolling processes, is introduced. As shown in Fig. 10, this wedge-shaped sample exhibits varying heights from front to back, resulting in different and continuously changing strains when passed through the rollers. According to the principle of constant

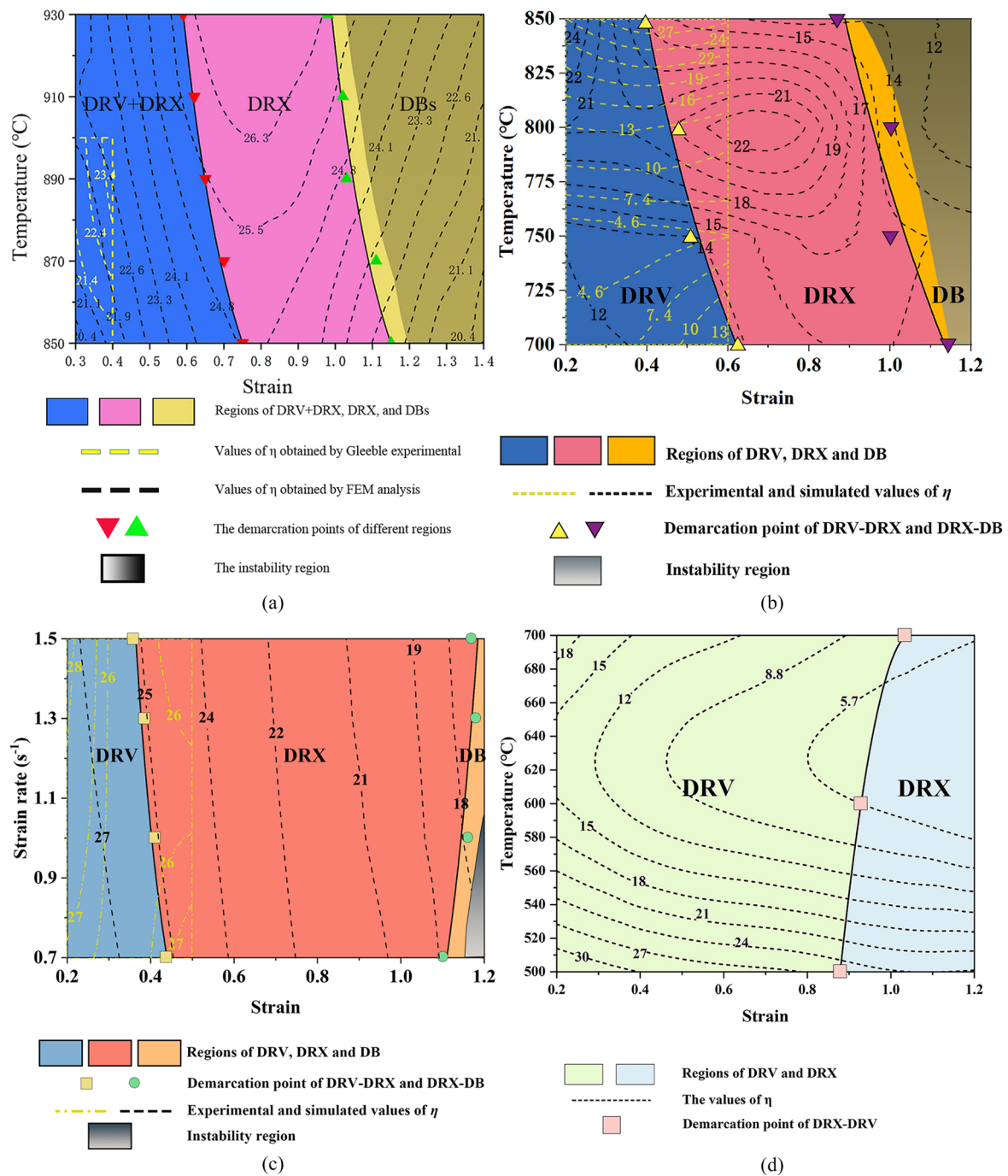


Fig. 9 Deformation mechanism diagram of several Ti alloys and medium entropy alloys: (a) TC18; (b) TA16; (c) TA16/SiCp; (d) TiZrV [54–57]

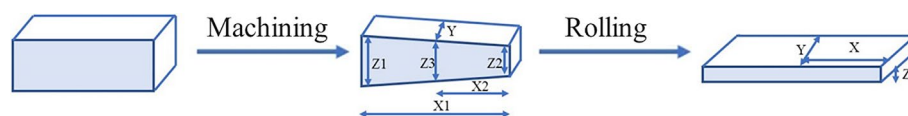


Fig. 10 Schematic diagram of HTP of rolled samples with different reductions [59]

volume in plastic deformation, the strain magnitude in each part can be derived, and FEM is used to verify and validate the calculated results against simulated outcomes. This approach validates the

computed and simulated results to determine the strains in each part and provides continuous variations in properties and microstructure under different deformation parameters [59].

Using the principle of constant volume in plastic deformation, the reduction ΔZ at distance X from the exit can be calculated as follows [59]:

$$\Delta Z = 1 - \frac{Z}{\sqrt{2XZ\tan\theta + Z_2^2}} \quad (1)$$

The results from this equation are validated against computer simulations, as shown in Fig. 11 (FEM results of a wedge-shaped sample rolling experiment) [60]. This allows for the determination of strains in each part. Systematic characterization of the alloy sample can then be conducted to rapidly establish the relationship between deformation parameters and microstructure.

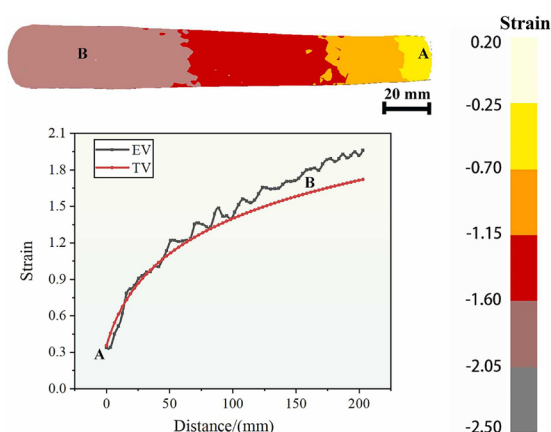


Fig. 11 FEM results of wedge sample rolling [60]

HT rolling techniques using wedge-shaped samples have been applied to research on the rolling process of various Ti alloys. ZHU et al [60] employed HT rolling technology with wedge-shaped samples to apply continuous gradient strains to rare-earth-doped α -Ti during hot rolling, facilitating the evaluation of changes in microstructure, texture, and hardness characteristics as strain increased from low to high levels. XU et al [61] developed an HTP method using wedge-shaped hot rolling for the Ti-55511 near- β Ti alloy. They obtained samples with a gradient true strain distribution ranging from 0 to 1.1 and annealed samples with gradient strain distributions to achieve spheroidization of α phases, allowing for rapid assessment of the spheroidization fraction of α phases under different deformation conditions.

Gradient rolling techniques have also been applied to other non-ferrous metals and their alloys. CHEN et al [59] rolled wedge-shaped samples of Ag-6Cu-1Zn-0.5Ni alloy to obtain continuous

variations in reductions ranging from 33% to 80%, and rapidly characterized the microstructure and properties of the alloy. JIN et al [62], using gradient rolling, achieved a transition from initial to deformed microstructure within the same sample of Mg-Al-Zn alloy, studying the influence of Mg17Al12 second-phase particles on twin-induced recrystallization behavior.

2.3 Acquiring temperature gradient

Heat treatment is a critical process for controlling the microstructure and properties of Ti alloys. Traditional alloy heat treatment methods typically employ conventional heating techniques, generating only a single temperature zone. To study heat treatment processes, multiple parallel experiments are often required, each under different conditions, followed by characterization of the microstructure and properties of the samples. This approach incurs significant time and economic costs. Gradient heat treatment technology, however, uses specialized experimental equipment to create continuous temperature distributions in samples. This method achieves gradient distribution of microstructures, enabling extensive and rapid acquisition of data on properties and microstructure through high-throughput characterization (HTC) techniques.

The phase transformations of Ti alloys during heating and cooling make their heat treatment more complex compared to other non-ferrous metals. Gradient heat treatment technology allows for faster and more accurate acquisition of data on heat treatment parameters, microstructure, and properties of Ti alloys. The Jominy end-quench test, originally developed to determine the hardenability of steels, is now applicable to high-throughput studies of Ti alloy heat treatment processes, as depicted in Fig. 12 [63].

XU et al [64] utilized the end-quench technique to create a continuous temperature gradient from 600 to 700 °C in the Ti-5553 alloy, investigating the evolution of microstructure and mechanical properties under different heat treatment conditions. LAPIN and MAREK [65] used the Jominy end-quench test to study the effects of continuous cooling on solid-state phase transformations in the Ti-47Al-8Ta (at.%) alloy. They found that with increasing distance from the

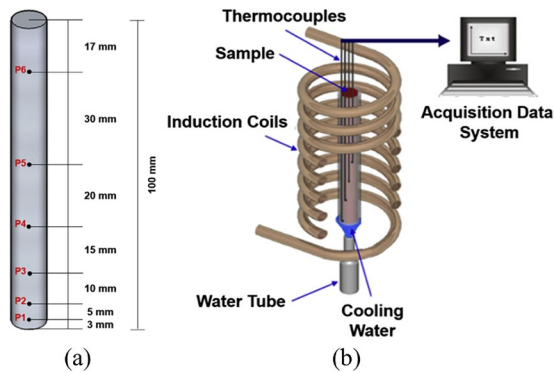


Fig. 12 Schematic diagram of Jominy end quench test: (a) Sample and locations of thermocouple; (b) Experimental equipment [63] (Reproduced with permission from Elsevier Ltd.)

water-quenched surface, the microstructure transitioned from $\gamma_M(\text{TiAl}) + \alpha$ (Ti matrix solid solution) to blocky γ_M , and then to lamellar $\gamma(\text{TiAl}) + \alpha_2(\text{Ti}_3\text{Al})$ and γ_M phases.

High-frequency induction heating, electro-pulsing treatment, and other methods can also generate temperature gradients within a single sample. JIAN et al [66] successfully prepared gradient structures using high-frequency induction quenching to improve the mechanical properties of the Ti-6Al-4V alloy. After high-frequency induction quenching and aging treatment, the microstructure changed from surface α' -martensite with fine α_s lamellae to a bimodal microstructure at the center, as shown in Fig. 13.

FAN et al [67] used electropulsing treatment to create axial gradient structures in TC21 Ti alloy, followed by step-quenching to control the precipitation behavior of α phases, as shown in Fig. 14. This approach improved both the strength and ductility of the alloy simultaneously.

Additionally, researchers have utilized the non-uniformity of temperature fields within heating furnaces to achieve gradient heat treatment on

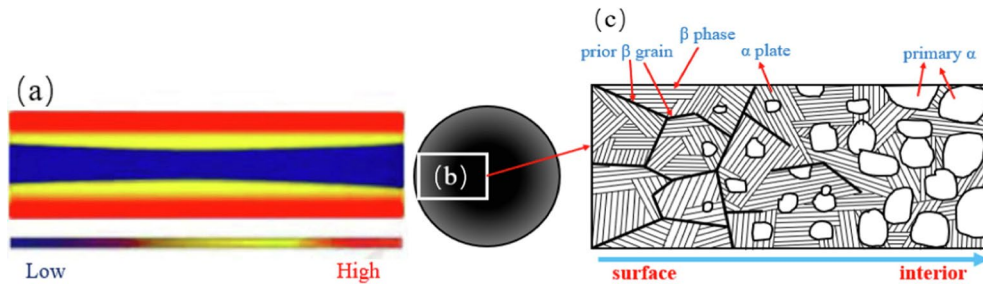


Fig. 13 High-frequency induction quenching of Ti-6Al-4V alloy: (a) Schematic of thermal distribution during induction heat treatment; (b, c) Schematic of microstructural gradient from surface to interior [66] (Reproduced with permission from Elsevier Ltd.)

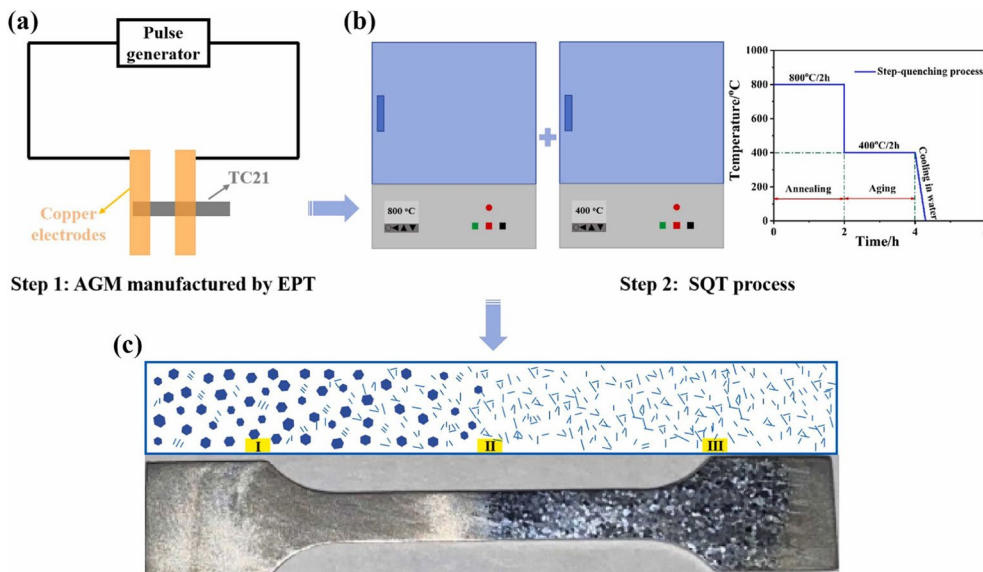


Fig. 14 Schematic diagram of preparation: (a) Electropulsing treatment; (b) Step-quenching; (c) Microstructure diagram and its distribution in tensile sample [67] (Reproduced with permission from Elsevier Ltd.)

individual samples. ZHAO et al [68] conducted aging heat treatment on a 15 cm-long Inconel 718 alloy rod by placing it into a tubular heat treatment furnace with one end exposed to the external environment, establishing a gradient temperature field ranging from 605 to 825 °C. Temperature variations in different regions of the sample were monitored using thermocouples. This method has also been applied to Ti alloys. WANG et al [69] performed continuous temperature gradient solution treatment on long rod samples in a tubular furnace at temperatures ranging from 746 to 909 °C (with the β transformation temperature of (845 ± 5) °C). This rapid optimization aimed to improve the microstructure of the metastable β Ti alloy (Ti–6.8Mo–3.9Al–2.8Cr–2Nb–1.2V–1Zr–1Sn), achieving high strength and ductility.

The gradient heat treatment methods mentioned above all generate gradient temperature fields within individual samples. However, the challenge lies in the limited temperature span achieved within a single sample, resulting in limited data on microstructure and properties. WEI et al [70] developed an efficient multi-temperature field heat treatment method using microwave radiation as the heating source. They utilized the selective heating capability of microwaves to create 100 temperature zones simultaneously, allowing for heat treatment of 100 individual bulk samples. This method integrates multiple rapid characterization techniques such as HT X-ray diffraction (XRD), HT scanning electron microscopy (SEM), and HT hardness testing. It enables the acquisition of a large amount of characterization data in a short time, which can be uploaded to a HT materials database, providing data support for subsequent research.

Current gradient heat treatment technologies focus on generating temperature gradients within individual samples or creating numerous temperature zones simultaneously across multiple samples. However, most reports overlook subsequent HTC and rapid data analysis. Combining gradient heat treatment techniques with HT microstructure and property characterization technologies allows for parallel processing of samples, thereby enhancing the efficiency of experimental research stages. After acquiring extensive microstructure data, computer image recognition technology can be employed to extract

existing microstructure features. By training on these data, relationships between microstructure morphology parameters and heat treatment parameters can be quickly learned, facilitating rapid prediction and selection of specific microstructures and corresponding heat treatment parameters.

For titanium alloys, even minor alterations in microstructure can significantly impact their mechanical properties. The effect of elemental composition on microstructure mainly occurs through changes in lattice parameters, elemental enrichment at interfaces, and similar factors, rather than the method used to achieve a composition gradient. During deformation processes, gradient distributions of strain fields and their corresponding microstructures can be captured in a single sample using high-throughput preparation techniques, such as biconical table technology and gradient rolling technology. Similarly, during heat treatment, gradient temperature fields and the resulting microstructure can also be observed within a single sample. This allows certain microstructural parameters (e.g., sphericity, aspect ratio, and grain size) to be correlated with process parameters through modeling.

For subsequent mechanical property testing, although existing specialized equipment can perform microzone testing, the resulting data often reflect localized properties rather than the true macroscopic behavior of the material. The most common approach used by researchers today is to reproduce large-size samples under specific processing conditions to verify and map the relationships among processing, microstructure, and mechanical properties. However, high-throughput preparation methods primarily provide extensive microstructural data without directly linking processing to performance. Instead, empirical and semi-empirical methods are typically employed to relate this microstructural data to overall material properties.

Looking forward, the development of more rapid and precise high-throughput characterization techniques, along with specialized equipment, is anticipated. These advancements would facilitate the direct establishment of microstructure–property relationships in small-sized high-throughput samples, enabling a more comprehensive understanding how processing influences material performance.

3 High-throughput characterization techniques

HTC techniques have demonstrated excellent potential in the application to Ti alloys, particularly for microstructural observation and mechanical property testing. These techniques typically feature high spatial resolution, large characterization scales, fast acquisition speed, high stability, and scalability. Leveraging automated equipment and software, HTC enables efficient and precise characterization of numerous Ti alloy samples, significantly enhancing research efficiency.

HTC technology not only provides high-definition imaging of microstructure of Ti alloys, revealing intricate structural features, but also accurately measures their mechanical properties, quantifying key performance parameters such as strength and toughness. This comprehensive analysis from micro to macro levels offers robust data support for performance optimization and mechanism research of Ti alloy materials. Researchers can quickly screen high-performance candidate materials and further investigate the intrinsic relationships between their performance and structure, thereby promoting the innovative development of Ti alloy materials.

In summary, HTC technology plays an indispensable role in the scientific research of Ti alloys. Its efficient, precise, and comprehensive characterization capabilities provide strong technical support for the development and application of Ti alloy materials. Several methods already applied or showing promise in the HTC of Ti alloys are summarized in Table 2.

3.1 Microstructure

When discussing the mechanical and physical properties of structural materials, the microstructure of precipitates, the matrix, and interfaces plays a crucial role. Therefore, detailed characterization of the composition (phase) of the matrix alloy, the morphology, distribution and content of the reinforcing phase and compounds, and the interface structure (composition) is particularly important. However, existing methods for characterizing the microstructure of materials, such as X-ray, ultraviolet, and infrared spectroscopy, as well as optical and electron microscopy techniques, each

have their advantages but are limited by factors such as light flux density, spatial resolution, and the capability to fully characterize large-sized samples and cross-scale material structures [71,72]. These constraints hinder their effectiveness in meeting contemporary demands for HT micro-regional testing, rapid R&D processes, and efficient detection techniques in materials science.

To overcome these limitations, scientists have developed numerous HT techniques for characterizing structural materials. For instance, synchrotron radiation sources, which can achieve high-brightness micro-focusing over the entire spectrum range from infrared to hard X-rays, possess excellent characteristics such as high collimation, full spectrum coverage, high polarization, and high purity. Thus, they can meet the brightness and spatial resolution requirements for HT material samples, making them ideal for HTC of alloy materials. Across-scale span original-position statistical-distribution analysis (OPA) is a HT spatial mapping characterization technique that quantitatively characterizes parameters such as segregation, porosity, and inclusion distribution within materials. This method enables cross-scale analysis, effectively reflecting the microstructural state on a macroscopic scale. The Spark-OPA technology has matured into a commercially viable application. Since the development of its first equipment in 2002, the technology has advanced significantly, with the current systems now in their fourth generation, as shown in the Fig. 15 [73]. In addition, traditional microstructural characterization tools like SEM have been adapted for the high-throughput characterization. JU et al [74] developed a single-beam HT SEM (Fig. 16) that can simultaneously collect secondary electron (SE) and backscattered electron (BSE) signals with nanoscale resolution. This enables the identification and differentiation of various phases and allows collection and analysis of extensive length-scale data. As a result, the experimental data can be linked across scales, from centimeter-level to nanoscale, and even down to the atomic level.

ZHAO et al [75] utilize an HT micro-area XRD technique to investigate the constituent phase structures of high-temperature alloy samples with 106 diverse components, which cannot be achieved by conventional characterization methods. This

Table 2 Comparison of HTC methods in Ti alloy

| HTC method | Sample preparation | Maximum spatial resolution | Acquisition time/s | Operational simplicity | Data processing | Scalability & versatility |
|------------------------|--------------------------|--|--------------------|----------------------------------|-------------------------------------|--|
| Chemical composition | EDS | Conventional grinding and polishing, surface carbon sprayed | ~1 μm | ~10 ⁻³ | Automated acquisition | Software package, analysis requires user input Integrated with SEM |
| | Mico-XRF | Conventional grinding and polishing, usually greater than 1 mm thick | ~300 nm | ~10 | Automated acquisition | Software package, analysis requires user input Integrated with XRD |
| Crystal structure | XRD, including micro-XRD | Conventional grinding and polishing, usually less than 5 mm thick | ~50 μm | 10 ² –10 ³ | Automated acquisition | Raw data, analysis with other software Integrated synchrotron light source |
| | EBSD | Electrolytic polishing | ~100 nm | ~10 ⁻³ | Automated acquisition | Software package, analysis requires user input Integration with in-situ heated and energized experimental platforms |
| Micro-structure | OM | Conventional grinding and polishing | ~1 μm | ~1 | Manual focus, automated acquisition | Combined with other software (image analysis) Integration with in-situ heated and energized experimental platforms |
| | SEM | Conventional grinding and polishing | ~1 nm | ~10 | Automated acquisition | Combined with other software (image analysis) Integration with in-situ heated and energized experimental platforms |
| Elastic modulus | Nanoindentation | Special surface roughness | ~100 nm | ~10 | Automated acquisition | Massive parallel data – |
| | Ultrasound spectroscopy | Conventional grinding and polishing | ~10 μm | ~10 | Automated acquisition | Software package, analysis requires user input Integrated with SEM |
| Hardness | Nanoindentation | Special surface roughness | ~100 nm | ~10 | Automated acquisition | Massive parallel data – |
| Strength and ductility | Nanoindentation | Special surface roughness | ~100 nm | ~10 | Automated acquisition | Massive parallel data – |
| | HT tensile testing rig | Conventional grinding and polishing | – | 10 ² –10 ³ | Automated acquisition | Massive parallel data Promising integration with DIC |
| Creep | Small punch test | Conventional grinding and polishing | – | – | Automated acquisition | – – |
| Fracture toughness | Small punch test | Conventional grinding and polishing, usually less than 1 mm thick | – | ~10 ² | Automated acquisition | Raw data, analysis with other software – |

DIC–Digital image correlation

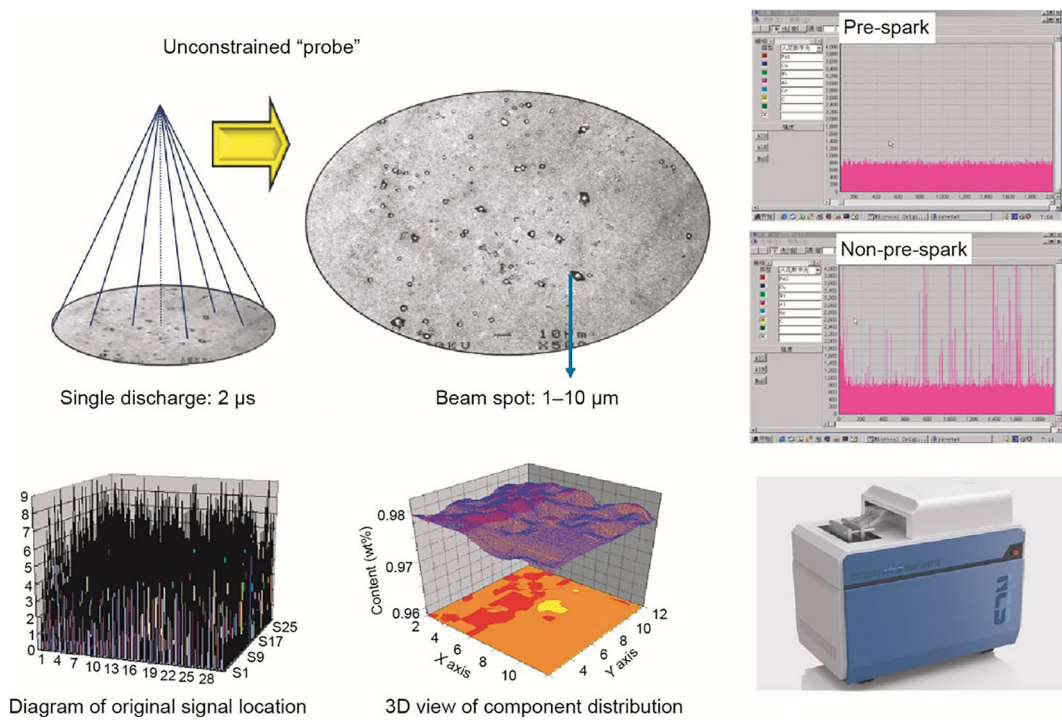


Fig. 15 Principle and device of Spark-OPA technology [73]



Fig. 16 System components in Navigator-100 high-throughput SEM [74]

technique not only successfully revealed the phase structures of various alloy components, but also generated a vast amount of experimental data, providing a rich resource for subsequent in-depth analysis. MALINOV et al [76] studied the properties of three Ti alloys, Ti–6Al–4V, Ti–6Al–

2Sn–4Zr–2Mo–0.08Si, and β 21s, at room and high temperatures. According to room temperature experiments, the effects of heat treatments on microstructures were explored, and distinct phase combinations at room temperature were found. In situ synchrotron radiation X-ray diffraction was

used to study the surface transformation of Ti alloys, tracking oxidation kinetics and phase transitions at high temperatures. Additionally, they elucidated the coefficient of thermal expansion of the hexagonal close-packed (HCP) α -phase and the effect of oxygen on lattice parameters. These results are significant for understanding the surface transformation under extreme conditions. KOJIMA et al [77] used synchrotron radiation X-ray laminography to measure carbon fibre (30 wt.%) reinforced polyamide 6 specimens with sharp notches to study the internal fatigue damage behavior of samples. SANDGREN et al [78] utilized high-energy synchrotron radiation X-ray microchromatography to observe in situ an initial 20 μm crack extension in additively manufactured Ti–6Al–4V materials. To gain insights into fatigue crack growth (FCG) microstructure, they analyzed the fracture surfaces using both 2D and 3D tomography reconstructions techniques, coupled with SEM. Notably, the obtained data exhibited consistency with conventional FCG test data, affirming the efficacy of combining X-ray microchromatography and SEM in studying fatigue crack extension behavior. SU et al [79] extensively characterized the microstructure and residual strain of induction-quenched gears in an HT fashion. Their approach utilized the scattered neutron source technology, coupled with Bragg-edge transmission imaging, to derive 2D maps that investigate the spatial distribution of both the microstructure and residual strain, as shown in Fig. 17.

WEI et al [70] conducted an experimental study on parallel heat treatment, leveraging an HT micro-area XRD and SEM. Their methodology allowed for efficient and rapid characterization of the microstructures of diverse materials with varying compositions. Through this approach, they successfully established a correlation between tempering temperatures and material microstructure and properties, providing valuable insights into the effects of heat treatment on material properties. ZHANG et al [31] employed a combination of SEM and DIC techniques to distinguish the microstructure of alloys based on the grayscale properties of α and β phases. They used a macro-programming language to efficiently determine the α -layer width and α -phase volume fraction in DED Ti–xAl–yV alloys. Their findings indicate that the α -phase volume fraction primarily influences the hardness,

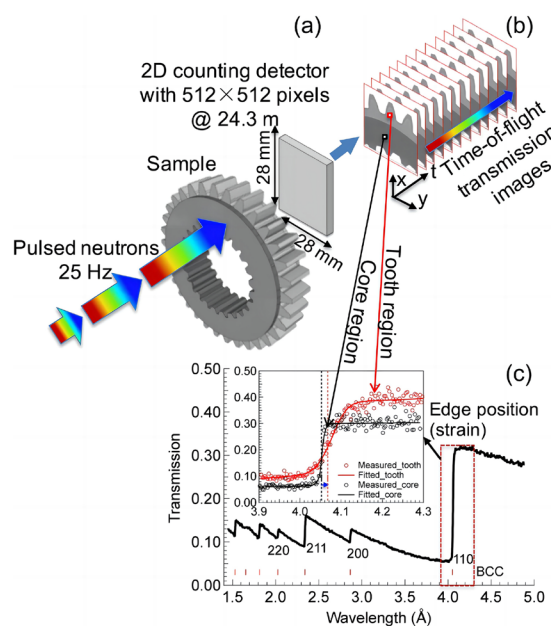


Fig. 17 Schematic of neutron Bragg edge transmission imaging method [79]

while the yield strength exhibits greater sensitivity to the α -layer width. ZHANG et al [80] introduced a multidimensional characterization technique for the microstructural analysis of the TC4 alloy. By efficiently acquiring and visualizing etched SEM images, the 3D surface profile highlights the distinction between the α and β phases, enabling phase identification through advanced analysis techniques (Fig. 18). With a minimum detection limit of 0.5 μm in centimeter scale samples, the proposed approach proficiently characterizes cracks, pits, and contaminants, thus providing a streamlined method for identifying material defects.

3.2 Mechanical property

The evaluation of mechanical properties typically encompasses indices such as hardness, impact toughness, yield strength, creep, and fatigue. Traditional testing methods generally assess the mechanical properties of a single sample or at a single scale, which is insufficient for simultaneous experiments on multiple samples or scales. Therefore, advanced characterization methods are needed for mechanical properties, particularly for the intricate compositions of Ti alloys that necessitate further advancements in HTC technologies.

Traditional hardness testing is a destructive method. The HTC method of fluid microprobe

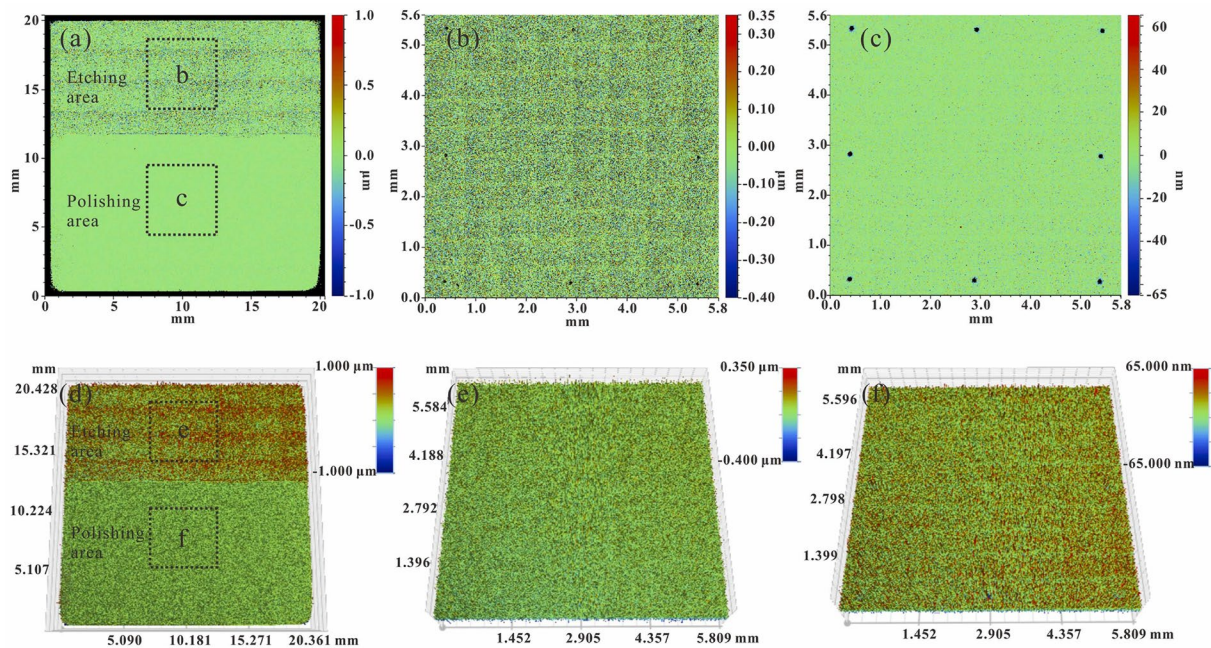


Fig. 18 2D surface profiles (a–c) with corresponding 3D surface profile morphologies (d–f) [80] (Reproduced with permission from Elsevier Ltd.)

mechanics, based on the principle of isostatic pressure, uses high-pressure fluid loading to nondestructively characterize the hardness characteristics of metal materials [73]. FENG et al [81] investigated the deformation and microstructural distribution of high-chromium white cast iron after cold isostatic pressing, revealing its close relationship with elastic modulus, equivalent modulus, and hardness (Fig. 19). PREISLER et al [82] used field-assisted sintering technology to prepare layered samples of Ti alloys with 24 different chemical compositions. The elastic properties of these layers, specifically the shear modulus (G) and Young's (E) were accurately measured through resonant ultrasound spectroscopy. HUANG et al [83] conducted HTC of the ultimate tensile strength and fracture elongation of 316L samples under various process parameters using a HT tensile sample platform, obtaining a large amount of mechanical performance data for different samples.

As micro-region mechanical characterization technology continues to evolve, it has become increasingly pivotal as a microscopic mirror of macroscopic properties. Consequently, HT micro-region mechanical property testing has emerged as a focal point in current research and has widespread adoption. For over a century,

indentation technology has been a cornerstone in material testing [84]. In traditional indentation testing, a hard indenter presses onto the surface of a soft sample under predefined conditions, and the resulting residual indentation is measured to determine microhardness. With advancements in high-resolution equipment, modern instrumented indentation techniques now allow for continuous monitoring of load and displacement throughout the indentation process. GAO et al [85] combined nanoindentation and EPMA to directly investigate the microzone composition and performance of novel Ti–Mo–Al–Zr–Cr–Sn alloys under multiple indentations. They tested eight element combinations, evaluating hardness (H) and elastic modulus (E). Using principal components analysis and equivalent theory, they mapped alloy–property relationships, enabling rapid determination of high- E and high- H compositions and guiding alloy optimization. WANG et al [86] synthesized thin films of Ti–Al₂O₃ functionally graded materials (FGMs) using magnetron sputtering. Through the nanoindentation analysis, they revealed the relationship among gradient morphology, indentation depth, and material hardness and modulus, providing a rapid method for researching and designing new FGMs. The nanoindentation technique is not only limited to determining the

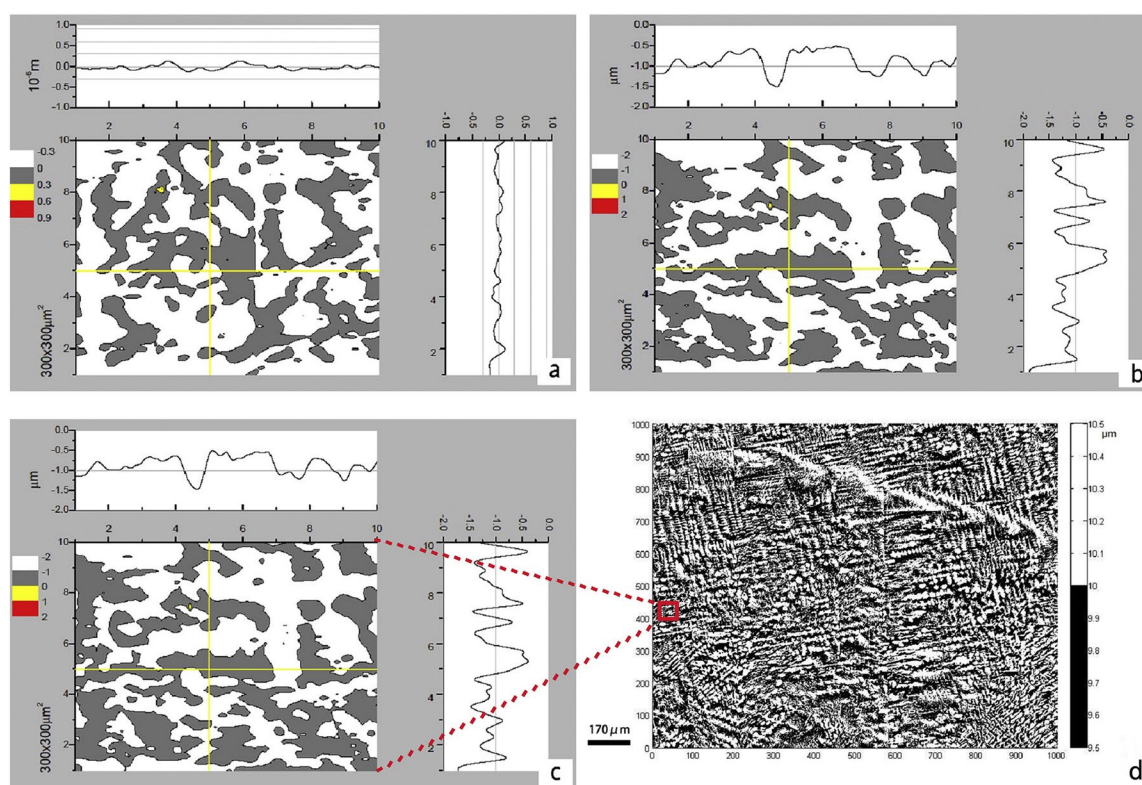


Fig. 19 Contour map of isostatic pressing [81] (Reproduced with permission from Elsevier Ltd.)

hardness and elasticity modulus but can also characterizes multidimensional material mechanical parameters such as yield strength [87], hardening index [88], fracture toughness [89], creep or stress relaxation [90], and residual stress [91]. Micro–nano indentation testing systems under service-like environments should have greater application potential and provide possibilities for new discoveries in material performance in multi-dimensional environmental spaces. Firstly, combined with in-situ characterization methods, micro–nano indentation testing systems provide technical support for revealing microzone deformation and damage mechanisms of materials. Secondly, they can assist in the establishment of an ideal loading environment. For instance, the high vacuum of an SEM chamber mitigates oxidation at high temperatures and icing at low temperatures, ensuring more accurate material assessments.

Micro-regions HTC technology significantly accelerates the materials development process because it enables mechanical properties to be tested on large samples quickly. Although the relationship between material microstructure and mechanical properties is complex, influenced by factors such as microstructure and deformation

history, data from miniature samples and thin film samples are still valuable for revealing the overall trend of this relationship. Based on the composition–structure–performance database of these miniature samples and thin film samples, the performance trends of bulk materials can be predicted and verified by preparing corresponding individual materials to precisely determine the performance parameter range of the materials. Additionally, through extensive experiments, the correlation characteristics between miniature samples, thin film samples, and large materials can be gradually established, thereby enhancing the applicability and accuracy of combinatorial material chip technology in structural material research.

4 Summary and prospect

4.1 Summary

Ti alloys face significant challenges in the industrial production, including low production efficiency, high costs, and difficulties in controlling the stability of microstructure and performance, which hinder their widespread adoption. Traditional serial R&D models, which involve preparing and researching a single sample with one composition

and process, result in dispersed data and inefficiencies. In contrast, parallel R&D models can simultaneously generate large and continuous data sets, significantly improving research efficiency and reducing costs.

HTE methods start from material design, screening target alloy compositions, optimizing processing and heat treatment parameters, and conducting rapid characterization to achieve the desired material properties.

(1) Composition screening and optimization

HTP techniques can create single samples with composition gradients or simultaneously prepare numerous samples with different compositions. The diffusion multiple technique, a well-established HTP method, achieves compositional gradient changes in a single sample using diffusion theory. Recent research combining preparation methods such as additive manufacturing (AM) and vapor deposition with HT concepts has enriched HTP methods, offering more avenues for Ti alloy composition screening and optimization.

(2) Processing and heat treatment process optimization

Double-cone compression technology and gradient rolling technology are currently common HT deformation experimental techniques used to explore forging and rolling processes, respectively. These HT deformation experimental techniques are utilized to study the evolution of microstructure and deformation mechanisms in Ti alloys during the deformation process. The obtained structure–process relationships can guide the establishment of deformation mechanism maps, accelerate the design optimization of Ti alloy forming processes, and provide data support for establishing a Ti alloy deformation process database.

(3) Optimization of heat treatment conditions

Gradient heat treatment techniques can achieve continuously varying temperature fields in a single sample or perform parallel heat treatments on multiple samples to achieve numerous independent temperature zones. Combined with HTC techniques, these methods facilitate rapid data collection. Techniques such as end quenching, high-frequency induction heating, and electrical pulse treatment have been applied in Ti alloy research. Additionally, new gradient heat treatment methods such as microwave heating, already applied in other alloys, hold potential for future Ti alloy research.

(4) Rapid characterization of properties and microstructure

The collection of microstructure and performance data generated by HTP techniques relies on HTC techniques. Large-scale research facilities, such as synchrotron light sources and spallation neutron sources, facilitate the HTC of Ti alloy microstructures. Electron microscopy techniques have been improved based on HT concepts to automate and intelligently collect microstructural images quickly. For the material performance, HT performance testing techniques such as nanoindentation have been applied in Ti alloy research. Combining HTP techniques such as diffusion multiples and in-situ characterization methods can quickly establish composition–structure–performance relationships. Additionally, HT mechanical performance testing techniques, such as fluid microprobe and HT tensile testing, are expected to be increasingly applied in Ti alloy research.

4.2 Prospect

The application of HT research methods in Ti alloys has significantly accelerated the R&D process, injecting new vitality into material science. However, despite continuous technological progress and increasingly diversified markets, the field of Ti alloys still faces numerous challenges and opportunities. HT research methods, as efficient and systematic R&D tools, have considerable untapped potential. Future research on Ti alloys using HT methods will focus on the following aspects:

(1) Microstructure and property prediction

Utilizing HT computational techniques, such as first-principles calculations and molecular dynamics simulations, can efficiently predict the microstructure and macroscopic performance of Ti alloys. These methods facilitate the quick screening of potential high-performance alloys and reveal the intrinsic impact mechanisms of alloy elements on microstructure and performance, providing a solid theoretical foundation for the customized design of Ti alloys.

(2) Multi-scale simulation and experimental validation

The performance of Ti alloys is influenced by factors at multiple scales, including atomic, nano, and micron scales. Future HT research will emphasize the deep integration of multiscale

simulation and experimental validation. This cross-scale research approach will help comprehensively understand the multiscale behavior of Ti alloys, providing more reliable theoretical support and guidance for alloy design. Experimental validation will ensure the accuracy and reliability of simulation results, forming a solid experimental foundation for Ti alloy R&D.

(3) Intelligent material design and simulation

With the rapid development of artificial intelligence technology, future HT research will increasingly focus on intelligent material design and simulation. Data-driven material design will become a crucial branch of HT research. By leveraging ML and big data technology, HT data analysis can extract potential patterns from massive experimental data, constructing complex relationship models among material composition, process, and performance more accurately. These models will provide scientific theoretical guidance for Ti alloy design, significantly shortening the R&D cycle and accelerating the transformation of new materials from laboratory to market.

(4) Innovative applications of intelligent experimental platforms

The development of intelligent experimental platforms will strongly support HT research. These platforms will use advanced technologies such as robotic operation and real-time data acquisition and analysis systems to achieve automation and intelligence in the experimental process. This will greatly improve experimental efficiency, reduce the impact of human factors on experimental results, and enhance the precision, reliability, and efficiency of HT research.

(5) Interdisciplinary integration and collaborative innovation

The R&D of Ti alloys involves multiple disciplines, such as materials science, chemistry, physics, and mechanics. Future HT research will emphasize cross-disciplinary integration and collaborative innovation. By integrating resources and advantages from different disciplines, we can jointly tackle the challenges in Ti alloy R&D and promote higher-level development. Cross-disciplinary integration will also expand the application fields and markets for Ti alloys.

(6) Green and sustainable Ti alloy R&D

With the global emphasis on environmental protection and sustainable development, future Ti

alloy R&D will focus more on green and sustainable concepts. By optimizing the preparation processes of Ti alloys, reducing the energy consumption and emissions, and improving material utilization rates, we can achieve green and sustainable Ti alloy R&D. This will help promote the sustainable development of the Ti alloy industry, creating a better future for society.

In summary, extensively utilizing HT research methodologies in Ti alloy development will significantly bolster R&D efficiency and precision. Accurate forecasting of alloy traits, beginning with microstructure and property prediction, will lay a solid foundation for customized Ti alloy design. Multi-scale simulation and experimental validation will provide in-depth insights, enabling the identification of high-performing alloys with robust theoretical underpinnings. Intelligent material design and simulation, coupled with innovative applications of intelligent experimental platforms, will expedite the R&D process, minimizing human error and enhancing data integrity. Interdisciplinary integration and collaborative innovation will break down disciplinary silos, offering holistic solutions for Ti alloy R&D. Moreover, green and sustainable Ti alloy R&D strategies will ensure the industry's long-term sustainability.

This comprehensive approach will steer the Ti alloy industry towards greater efficiency, precision, and environmental friendliness. It will foster Ti alloy applications across aerospace, marine, biomedical, and other domains, providing robust support for technological advancements and industrial progress.

CRediT authorship contribution statement

Ke-chao ZHOU: Investigation, Methodology, Writing – Original draft, Writing – Review & editing; **Xiu-ye YANG:** Investigation, Writing – Original draft, Writing – Review & editing; **Yi-xin AN:** Data curation, Conceptualization, Writing – Review & editing; **Jun-yang HE:** Supervision, Writing – Review & editing; **Bing-feng WANG:** Writing – Review & editing; **Xiao-yong ZHANG:** Funding acquisition, Supervision, Writing – Review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported

in this paper.

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钛合金高通量实验技术进展综述

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摘 要: 钛合金作为典型的轻质高强金属材料, 在航空航天、海洋工程、生物医学等诸多领域展现出巨大的应用潜力。然而, 由于对钛合金微观结构与力学性能之间关系的认知不足, 导致研发周期拉长, 进而阻碍了其性能的进一步优化。近期, 高通量实验技术的兴起为钛合金新一代材料的快速开发带来了曙光。该综述回顾了钛合金高通量实验技术的最新研究成果。聚焦于钛合金制造中常用的高通量制备技术, 包括扩散多元节、增材制造、气相沉积等; 探讨了高通量表征技术在钛合金领域的当前应用状况; 最后, 总结了钛合金高通量制备和表征技术的研究成果, 并讨论了其在工业应用中面临的主要挑战。

关键词: 钛合金; 高通量; 微观结构; 力学性能

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