



Thermodynamically stable hR20 structure of the Al_3Er phase studied by varying solidification rate and polymorphic transformation

Jianzhu Wang¹, Kunyuan Gao¹, Xiangyuan Xiong¹, Jing Ning¹, Xiaolan Wu¹, Shengping Wen¹, Wu Wei¹, Hui Huang¹, Zuoren Nie¹, Dejing Zhou²

Keywords:

Al_3Er phase, polymorphic transformation, thermodynamic stability, critical cooling rate, autocatalytic nucleation

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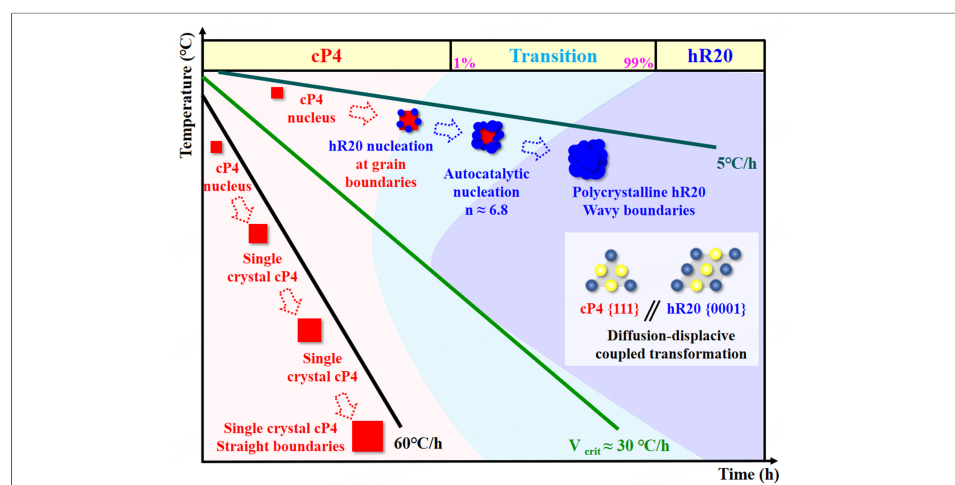
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Abstract

In this study, the crystal structure evolution and microstructure of the Al_3Er phase in an Al-30 wt% Er alloy solidified at different cooling rates were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). The XRD and EBSD results show that as the cooling rate decreases, the Al_3Er phase transforms from the cP4 structure (60 °C/h) to a mixture of cP4 and hR20 structures (50-40 °C/h), and finally to the hR20 structure (30-5 °C/h). The cP4-structured Al_3Er obtained at 60 °C/h is single-crystalline with an average grain size of 211 μm and exhibits straight boundaries, whereas the hR20-structured Al_3Er obtained at 5 °C/h is polycrystalline with an average grain size of 92 μm and displays wavy boundaries. TEM analysis shows the orientation relationship between the two structures: $[1\bar{2}1]_{\text{cP4}} // [1\bar{1}00]_{\text{hR20}}$, $(111)_{\text{cP4}} // (0006)_{\text{hR20}}$. These findings confirm that the stable structure of the Al_3Er phase is hR20. Classical nucleation theory calculation excludes the possibility of direct precipitation of the hR20-structured Al_3Er from the liquid, indicating that it forms through a polymorphic transformation of the cP4-structured Al_3Er .

¹College of Materials Science and Engineering, State Key Laboratory of Materials Low-Carbon Recycling, Beijing University of Technology, Beijing 100124, China.

²Yin Bang Clad Material Co. Ltd., Jiangsu Key Laboratory for Clad Materials, Wuxi 214145, Jiangsu, China.

Correspondence to: Prof. Kunyuan Gao, College of Materials Science and Engineering, State Key Laboratory of Materials Low-Carbon Recycling, Beijing University of Technology, Beijing 100124, China. E-mail: gaokunyuan@bjut.edu.cn

Johnson-Mehl-Avrami-Kolmogorov kinetic analysis yields a critical cooling rate of 36 °C/h for this transformation, consistent with the experimental observations. The high Avrami exponent ($n = 6.8$) reveals a diffusion-displacive coupled phase transformation mechanism of polymorphic transformation. This work provides a theoretical basis for understanding the structural evolution of the Al_3Er phase and for designing Al-Er alloys.

INTRODUCTION

Er-containing aluminum alloys have attracted considerable attention because of the excellent physical properties of the Al_3Er intermetallic compound, including its low density, high melting point and high specific stiffness^[1-3], and have been accepted as a potential high-temperature material. In these alloys, Er exists predominantly in the form of the Al_3Er phase. According to the Al-Er binary phase diagram^[4,5], a eutectic reaction occurs on the Al-rich side at 649 °C, and the maximum solid solubility of Er in α -Al is only 0.0461 at.%. Consequently, excess Er readily precipitates as nano-scale Al_3Er dispersoids during solidification or subsequent heat treatment. The equilibrium Al_3Er phase possesses a cP4 crystal structure and forms over a wide composition range in the Al-Er binary system. The cP4-structured Al_3Er compound has been widely employed as a strengthening phase in aluminum alloys^[6-11]. This phase is coherent with the Al matrix and contributes significantly to enhancing mechanical properties and thermal stability of the alloys. The stability of the Al_3Er phase plays a crucial role in determining the microstructure and overall performance of the alloys. Therefore, a thorough understanding of the formation, evolution and stability of Al_3Er is essential for the design and optimization of high-performance Er-containing aluminum alloys.

The thermodynamically stable structure of Al_3Er has long been reported to be the ordered cubic L_{1_2} (Cu_3Au -type, cP4) structure with space group $\text{Pm}\bar{3}\text{m}$, and this has generally been accepted as the equilibrium crystal structure of Al_3Er . This structure exhibits an ABC stacking sequence with a periodicity of three atomic layers, in which Er atoms occupy the cube corners and Al atoms occupy the face-centered positions^[12]. The lattice constant of cP4-structured Al_3Er is reported to be 0.4207–0.4231 nm^[13-15], corresponding to a lattice mismatch of approximately 4.0% with the Al matrix. The current Al-Er binary phase diagrams^[4,5] list the cP4 structure as the thermodynamically stable structure of Al_3Er , and many high-resolution transmission electron microscopy (HRTEM) studies have reported that nano-scale Al_3Er precipitates in Al-alloys adopt this cP4 structure^[3,16,17]. Furthermore, geometric factor calculations^[12,18] and first-principles studies comparing the L_{1_2} , $\text{D}_{0_{19}}$, $\text{D}_{0_{22}}$ and $\text{D}_{0_{23}}$ structures at 0 K identified the cP4 structure as the ground state, with the lowest formation energy of -40.1 kJ/mol^[19]. These calculations, however, did not consider the hR20 structure.

More recent evidence has challenged this long-held view, suggesting that the hR20 structure represents the thermodynamically stable structure of Al_3Er . The hR20 structure belongs to the space group $\text{R}\bar{3}\text{m}$, with lattice constants $a = 0.6025$ nm and $c = 3.5675$ nm^[20]. It exhibits an ABACBCBABCACBCA stacking sequence with a periodicity of 15 atomic layers, and is regarded as an intermediate structure between the cubic and hexagonal crystal systems, exhibiting rhombohedral symmetry. Meyer^[20] demonstrated that slow cooling (4 °C/h) of a hypereutectic Al-20 wt% Er alloy produced hR20-structured Al_3Er , whereas fast cooling (85 °C/h) resulted in the formation of the cP4 structure. In addition, the cP4 structure of Al_3Er transformed into the hR20 structure upon prolonged annealing at 400–1,040 °C^[20,21], indicating a structural phase transformation. Moreover, the addition of 0.2–0.4 wt% Si and Ge to the Al-20 wt% Er alloy resulted in the formation of hR20-structured Al_3Er , which remained stable at room temperature^[20]. First-principles calculations further suggested that the hR20 structure was the most stable phase, with a calculated formation energy of -40.5 kJ/mol using the frozen potentials^[22]. Our previous work^[23] investigated the thermodynamic stability of the Al_3Er phase with various structures using standard potentials and found that the formation energy was -39.7 kJ/mol for the cP4-structured Al_3Er and

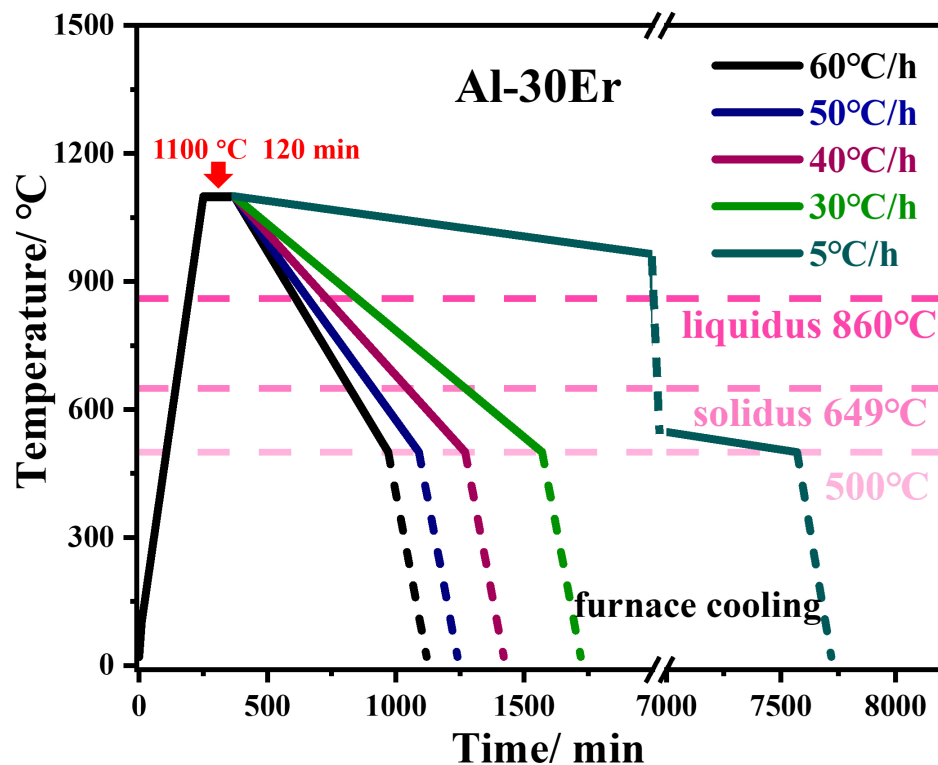


Figure 1. Processing scheme of the Al-30 wt% Er alloy.

-39.8 kJ/mol for the hR20-structured Al_3Er . Despite the small difference in the formation energies between the two structures, the hR20 structure is predicted to be thermodynamically more stable than the cP4 structure, which challenges the currently accepted understanding. Therefore, more detailed work is needed to resolve this discrepancy regarding the thermodynamically stable structure of Al_3Er and to understand the kinetic pathway of the transformation from cP4-structured Al_3Er to hR20-structured Al_3Er under different solidification conditions. In addition, the transformation pathway has not yet been reported, and there is still no experimental evidence or theoretical explanation for how the hR20-structured Al_3Er phase forms. Addressing these issues is essential for understanding the structural stability of the Al_3Er phase and for optimizing the solidification and heat-treatment processes of Al-Er alloys.

MATERIALS AND METHODS

A hypereutectic Al-30Er (in wt%) alloy was provided by Hunan Rare Earth Metal Materials Research Institute Co., Ltd. The Al_3Er crystal samples were prepared by cutting the alloy blocks into pieces to fit in a rectangular alumina crucible with dimensions of $30 \times 20 \times 17 \text{ mm}^3$. The crucible was then sealed in a quartz tube under an argon atmosphere with a pressure of 10^{-3} Pa . The alloy was melted in a KSL-1400X muffle furnace (Hefei Kejing Material Technology Co., Ltd., China) at $1,100^\circ\text{C}$ for 120 min, and subsequently cooled to 500°C (below the eutectic temperature) at varying rates of 60, 50, 40, 30, and 5°C/h , followed by furnace cooling to room temperature. A processing scheme is shown in Figure 1.

The crystal structure of the Al_3Er phase was determined using a D8 Advance X-ray diffractometer (Bruker Corporation, Germany) with Cu K α radiation at 40 kV, 30 mA and a scanning speed of $4^\circ/\text{min}$. The high-angle diffraction peaks were selected for slower scanning with a step size of 0.02° and a dwell time of 2 s per step. The Pseudo-Voigt and Pearson VII functions were applied to fit the diffraction peaks and determine precise peak positions^[24]. For micro-X-ray diffraction (XRD) analysis, the Al_3Er phase was examined using a

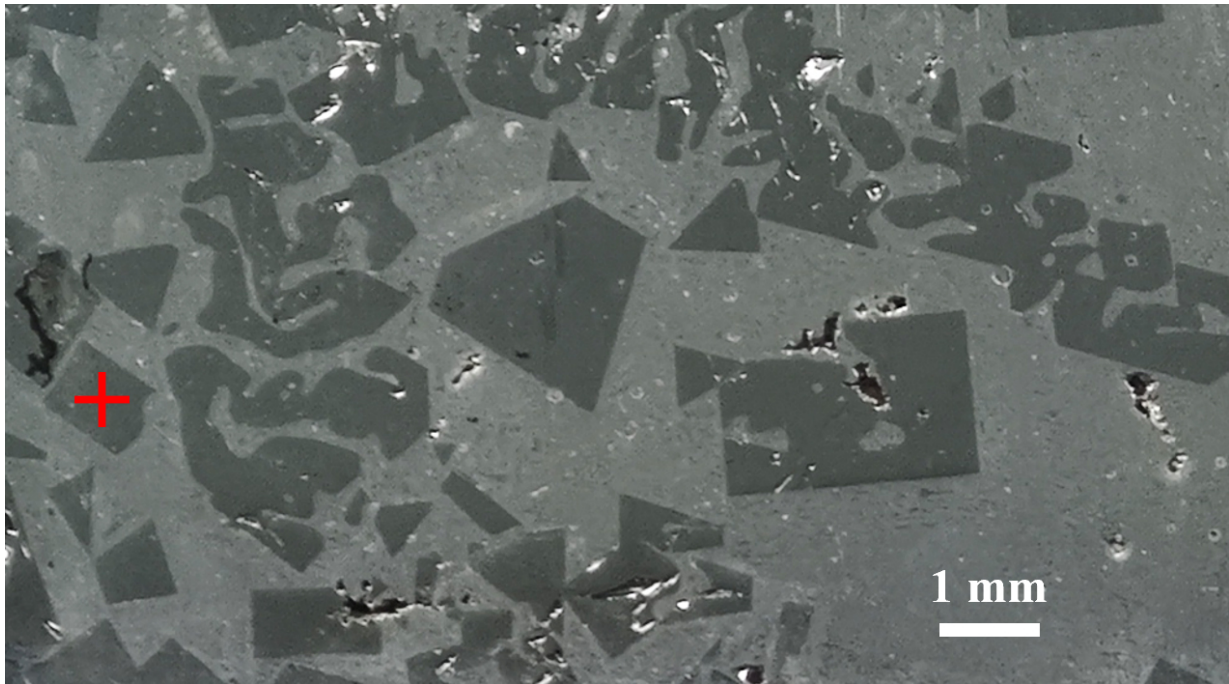


Figure 2. Optical micrograph of the sample cooled at 60 °C/h. The red cross marks the X-ray beam position in bulk-sensitive micro-XRD. XRD: X-ray diffraction.

D8 Advance X-ray diffractometer equipped with a General Area Detector Diffraction System (GADDS) (Bruker Corporation, Germany) and Co K α radiation operated at 40 kV and 30 mA, with a 0.3 mm collimator aperture. The X-ray beam was focused on the Al₃Er crystal, and the goniometer rotation angle φ was varied from 0 to 360°.

Scanning electron microscopy (SEM) samples were prepared by mechanical polishing and observations were conducted using an Field Electron and Ion Company (FEI) Helios NanoLab 600i scanning electron microscope (Thermo Fisher Scientific Inc., America). Electron backscatter diffraction (EBSD) samples were prepared by mechanical polishing, followed by vibratory polishing for 6 h using a 0.02 μm SiO₂ suspension. EBSD observations were performed on an Oxford NordlysMax3 electron backscatter diffraction detector (Oxford Instruments, United Kingdom). Transmission electron microscopy (TEM) samples were prepared by site-specific lift-out using a focused ion beam (FIB) in a Helios 4000i dual-beam scanning electron microscope (Thermo Fisher Scientific Inc., America). TEM observations were conducted in a JEOL 2100F transmission electron microscope (JEOL Ltd., Japan) operated at 200 kV.

RESULTS

Phase structure of the samples cooled at different rates

When the Al-30Er alloy is cooled from the liquid state to room temperature at different rates, millimeter-sized primary Al₃Er phase blocks are observed on the surface of the samples. A typical surface image of the sample prepared at a cooling rate of 60 °C/h is shown in [Figure 2](#). The red cross marks the X-ray beam position in bulk-sensitive micro-XRD. The dark contrast is the proeutectic Al₃Er phase. [Figure 3](#) shows the XRD patterns of the samples cooled at different rates. It can be seen that only Al and Al₃Er phases are present, and the crystal structure of the Al₃Er phase differs depending on the cooling rate. At a cooling rate of 60 °C/h, only the cP4-structured Al₃Er phase is formed. When the cooling rate decreases to 50 and 40 °C/h, the hR20-structured Al₃Er emerges, coexisting with the cP4-structured Al₃Er phase. At a slower cooling rate of 30 °C/h (and 5 °C/h), only the hR20-structured Al₃Er phase is observed. These results indicate that as the cooling rate decreases, the solidification process approaches equilibrium solidification, and the proportion of

the hR20-structured Al_3Er phase increases progressively until it becomes the sole phase.

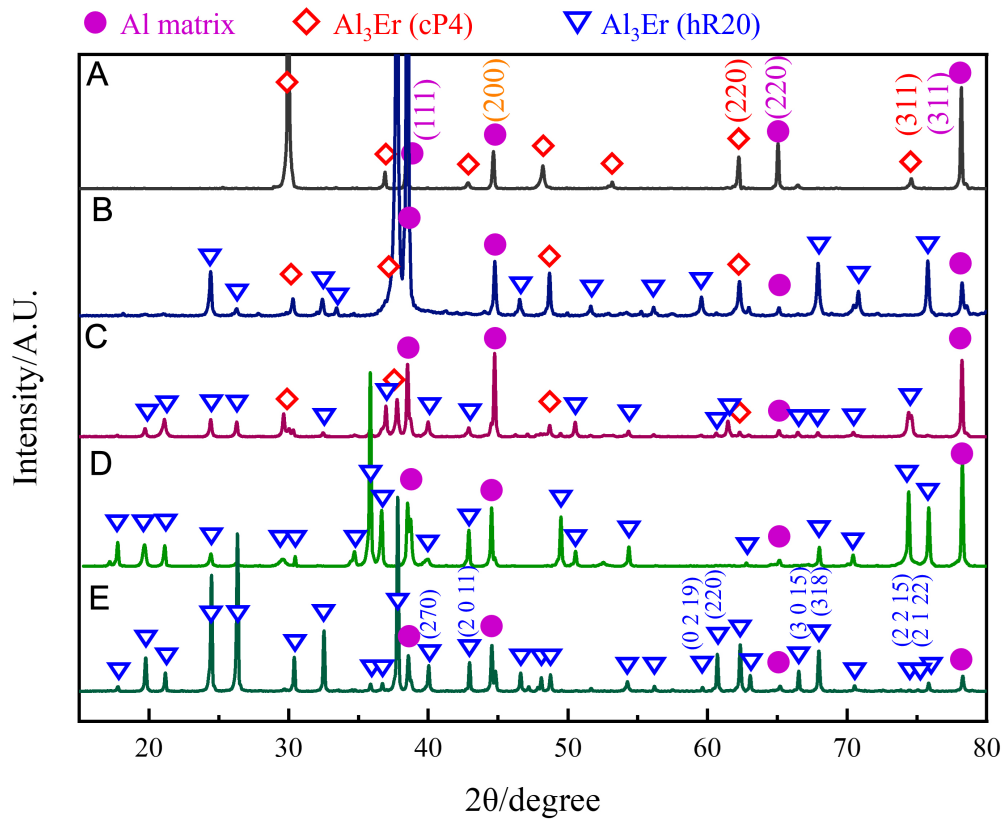


Figure 3. XRD patterns of Al-30Er alloy samples cooled at different rates: (A) 60 °C/h; (B) 50 °C/h; (C) 40 °C/h; (D) 30 °C/h; and (E) 5 °C/h. XRD: X-ray diffraction.

The high-angle diffraction peaks ($2\theta > 60^\circ$) of the samples cooled at 60 °C/h and 5 °C/h were selected for rescanning at a slower speed and finer step. The resulting diffraction patterns are shown in Figures 4 and 5. Figure 4 shows the (220), (311), (222) and (320) diffraction peaks of the cP4-structured Al_3Er phase, and the (220), (311) and (222) diffraction peaks of the Al matrix. It can be seen that the diffraction peaks of the cP4-structured Al_3Er phase, indexed as (220), (311) and (222), are located close to those of the Al phase. This is attributed to the similarity of the cP4 and fcc structures with low lattice mismatch. Figure 5 shows the (027), (2 0 11), (0 2 19), (220), (3 0 15), (318), (2 2 15) and (2 1 22) diffraction peaks of the hR20-structured Al_3Er phase, and the (111), (200), (220) and (311) diffraction peaks of the Al matrix. As the hR20 structure belongs to the rhombohedral crystal system with low symmetry, whereas cP4 belongs to the cubic crystal system with high symmetry, there is no correlation between the diffraction peak positions.

The lattice constants were obtained by fitting the experimental diffraction peaks using the Pseudo-Voigt and Pearson VII functions^[24] to extract accurate peak positions, which were then used in the Bragg equation.

The lattice constants of Al and cP4-structured Al_3Er were calculated to be 0.4047 ± 0.0002 nm and 0.4212 ± 0.0003 nm, and the lattice mismatch between the two phases was $4.08 \pm 0.09\%$. The lattice constants of the hR20-structured Al_3Er were calculated to be $a = 0.6036 \pm 0.0006$ nm and $c = 3.5705 \pm 0.0004$ nm. The experimental results obtained in this work and in other experiments^[13–15,20] are compared in Table 1. As shown in Table 1, the measured values are generally consistent with the reported values, with differences comparable to the typical spread among independent experimental studies.

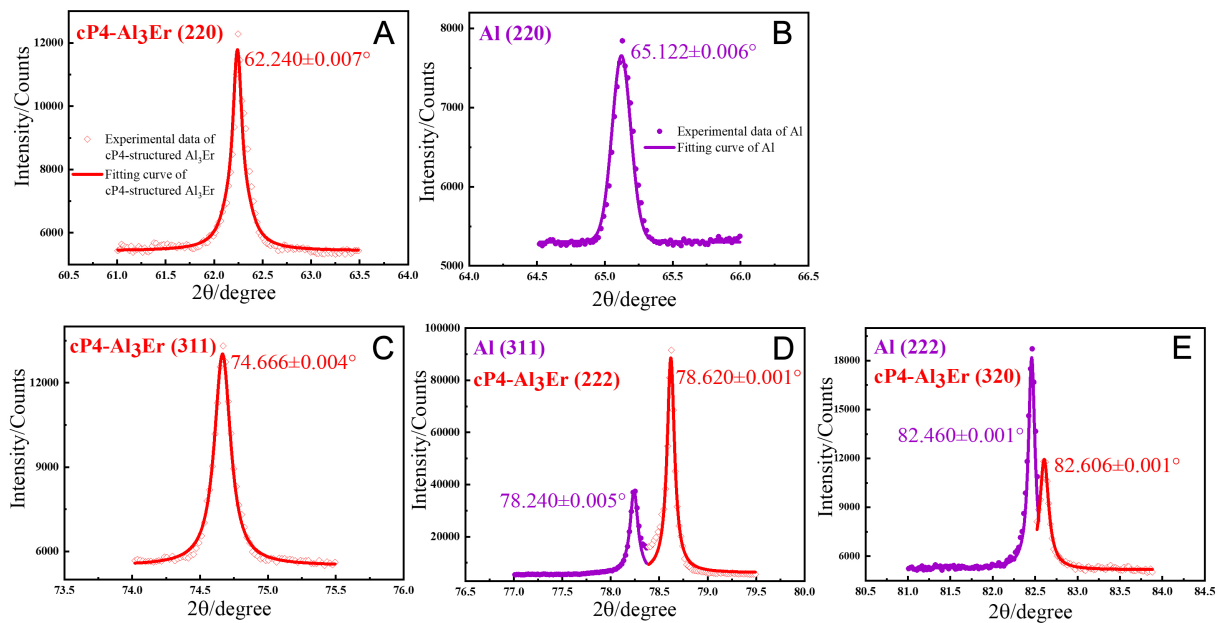


Figure 4. High-resolution diffraction peaks of the cP4-structured Al_3Er and the Al matrix after slow scanning: (A) Al_3Er (220); (B) Al (220); (C) Al_3Er (311); (D) Al (311) and Al_3Er (222); (E) Al (222) and Al_3Er (320).

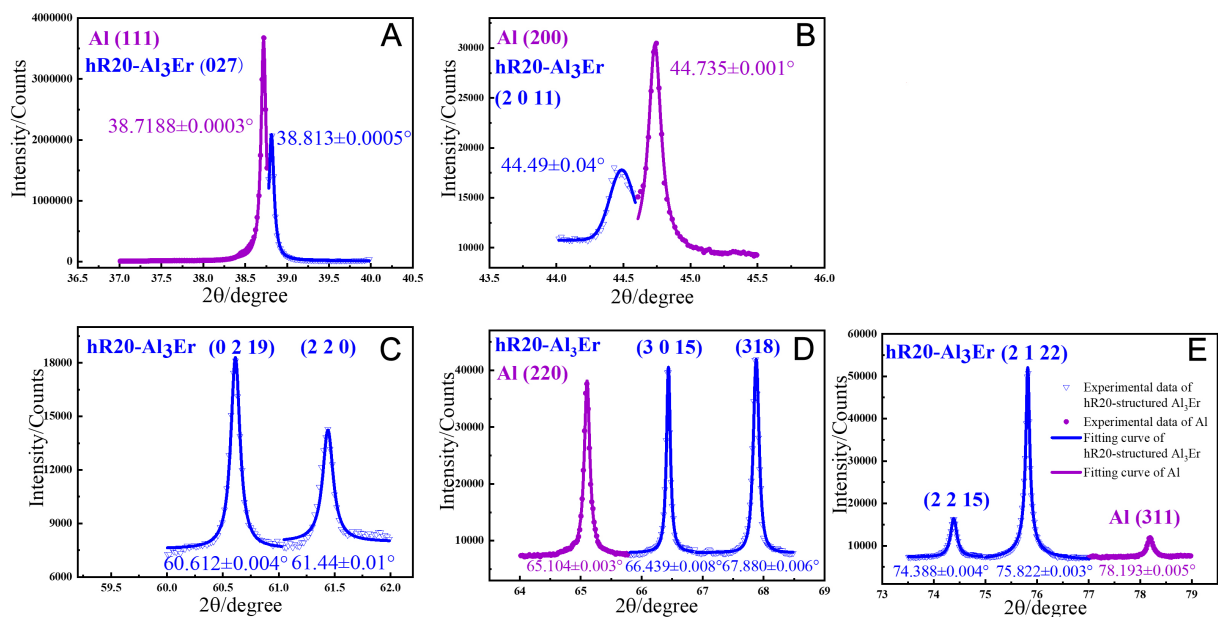


Figure 5. High-resolution diffraction peaks of the hR20-structured Al_3Er and the Al matrix after slow scanning: (A) Al (111) and Al_3Er (027); (B) Al (200) and Al_3Er (2 0 11); (C) Al_3Er (0 2 19), Al_3Er (220); (D) Al (220) and Al_3Er (3 0 15), Al_3Er (318); (E) Al (311) and Al_3Er (2 2 15), Al_3Er (2 1 22).

Morphology of Al_3Er formed at different cooling rates

Primary Al_3Er phase

The microstructural morphology of the Al_3Er phase formed at different cooling rates was characterized using SEM and EBSD, and typical SEM micrographs of the primary Al_3Er phase are shown in Figure 6. Energy-dispersive X-ray spectroscopy (EDS) point analyses of the primary Al_3Er particles from the samples confirmed an Er content of approximately 25 at.% for both the cP4- and hR20-structured particles, consistent

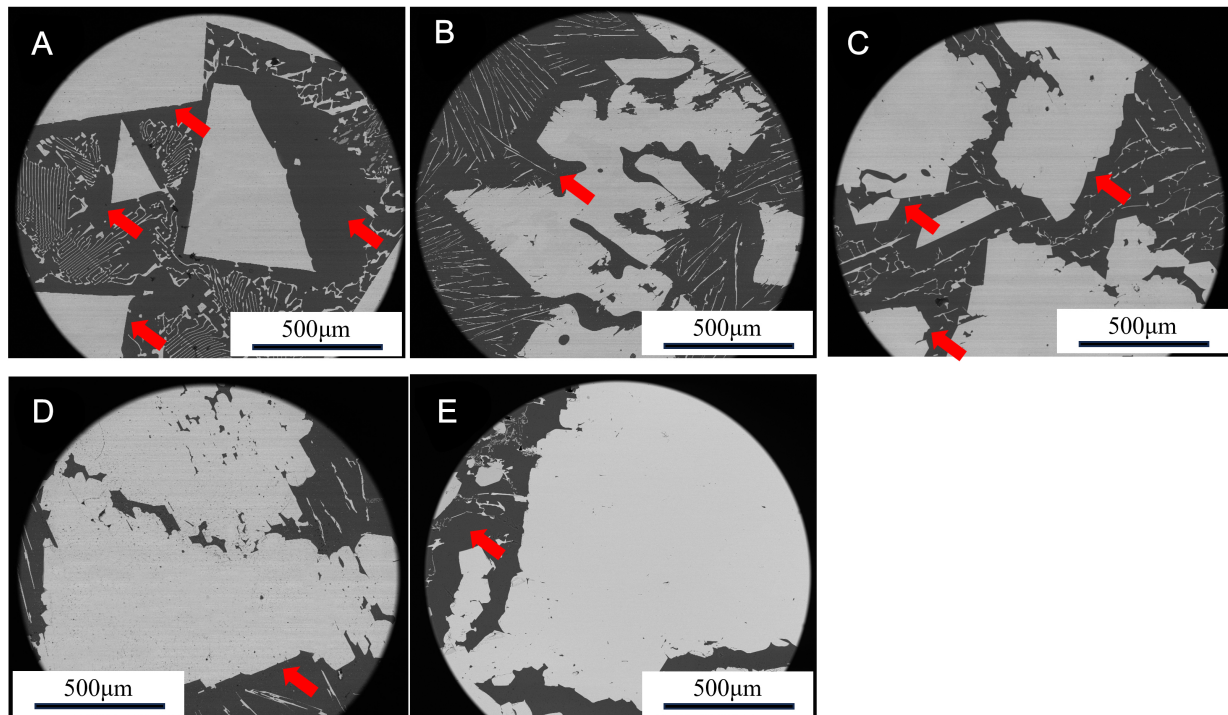


Figure 6. SEM micrographs of primary Al_3Er phase formed at different cooling rates. (A) 60 °C/h; (B) 50 °C/h; (C) 40 °C/h; (D) 30 °C/h; and (E) 5 °C/h. SEM: Scanning electron microscopy.

Table 1. Lattice constants and lattice mismatch values between the Al matrix and cP4 and hR20 structured Al_3Er obtained by different experiments

Al	Lattice constant (nm)		Mismatch	Reference
	cP4- Al_3Er	hR20- Al_3Er		
a	a	a	c	
0.4047 ± 0.0002	0.4212 ± 0.0003	0.6036 ± 0.0006	3.5705 ± 0.0004	$4.08 \pm 0.09\%$ This work
0.40495	0.4207			3.89% [13]
	0.4231			4.48% [14]
	0.4215			4.09% [15]
		0.6025	3.5675	[20]

The letters a and c are lattice constants.

with the stoichiometric composition of Al_3Er . No measurable compositional difference between the two types of particles was detected, supporting the conclusion that they are iso-compositional polymorphs. The primary Al_3Er phase appears as light gray blocky particles regardless of the cooling rate, and each primary phase particle is surrounded by a dark gray halo, as indicated by the red arrows. The outline of the halo conforms to the external outline of the primary Al_3Er particles. Surrounding the halo are the alternating light and dark regions, corresponding to the eutectic structure. Combined with the XRD results, the light gray particles are identified as the primary Al_3Er phase, while the dark gray regions correspond to the Al phase, confirming that the halo surrounding the primary Al_3Er phase is the Al phase formed during solidification.

With decreasing cooling rate, the average size of the primary Al_3Er particles increases progressively. At a cooling rate of 60 °C/h, the average size of the primary Al_3Er particles is approximately 500 μm . When the cooling rate decreases to 50 °C/h, 40 °C/h, 30 °C/h and 5 °C/h, the particle size increases to 800 μm , 1 mm,

1.5 mm and 2 mm, respectively. During solidification of the hypereutectic Al-30Er alloy from the liquid state, a lower cooling rate extends the residence time in the liquid/solid two-phase region, which allows the primary phase particles to have sufficient time for nucleation and growth, resulting in larger primary particles at room temperature.

In addition, the morphology of the primary Al_3Er particle boundaries also varies with the cooling rate. As seen in Figure 6A, at a cooling rate of 60 °C/h, the Al_3Er grain boundaries are predominantly straight. At cooling rates of 50 °C/h and 40 °C/h [Figure 6B and C], the grain boundaries exhibit a mixed character, with some grain boundaries appearing straight and others wavy. At cooling rates of 30 °C/h and 5 °C/h [Figure 6D and E], the grain boundaries are wavy. Correlating these observations with the XRD results in Figure 3, the primary cP4-structured Al_3Er particles show straight boundaries, whereas the primary hR20-structured Al_3Er particles exhibit wavy boundaries. The difference in micromorphology is likely related to their different crystal structures, which have different degrees of symmetry.

To investigate the grain orientation and grain size distribution of the primary Al_3Er grains, EBSD analysis was conducted on the primary phase in four samples cooled at 60, 50, 40 and 5 °C/h, as shown in Figure 7. In the phase distribution maps [Figure 7A2, B2, C2, D2], the red regions are the cP4-structured Al_3Er phase and the blue regions are the hR20-structured Al_3Er phase. As seen in Figure 7A, at a cooling rate of 60 °C/h, the primary phase exhibits a cP4 structure. In Figure 7B and C, at 50 °C/h and 40 °C/h, both cP4 and hR20 structures coexist. At the lowest cooling rate of 5 °C/h [Figure 7D], only the hR20 structure exists. These results are in good agreement with the XRD results in Figure 3. In the inverse pole figure (IPF) maps [Figure 7A3, B3, C3, D3], each primary cP4-structured Al_3Er particle exhibits a single crystallographic orientation, regardless of the cooling rate, indicating that it is a single crystal. In contrast, each primary hR20-structured Al_3Er particle displays at least two distinct orientations, signifying that it is polycrystalline. The average internal grain size of the primary cP4-structured Al_3Er particles at 60 °C/h is 211 μm , while that of the primary hR20-structured Al_3Er particles at 5 °C/h is 92 μm .

As the EBSD provides crystallographic information from a relatively small near-surface region of the sample, a bulk-sensitive micro-XRD analysis was conducted on the sample containing the primary cP4-structured Al_3Er phase cooled at 60 °C/h, as shown in Figure 8. The X-ray beam was focused on the position marked by the red cross in Figure 2. The sharp bright diffraction spot in Figure 8A indicates that the measured volume of the primary cP4-structured Al_3Er phase corresponds to a single crystal. The diffraction pattern corresponding to the diffraction spot in Figure 8A is shown in Figure 8B, which exhibits only one strong (200) diffraction reflection, indicating that the (200) planes of the primary Al_3Er particle are parallel to the sample surface.

Eutectic structure

The eutectic structures in samples cooled at different rates are presented in Figure 9. At a cooling rate of 60 °C/h, the eutectic structure appears either as irregular flocculent or lamellar, as shown in Figure 9A. At lower cooling rates, the eutectic structure uniformly exhibits a lamellar morphology, as shown in Figure 9B–E. At higher cooling rates, the increased undercooling results in a flocculent eutectic morphology, whereas lower cooling rates promote near-equilibrium solidification and the development of a lamellar morphology.

As the cooling rate decreases, the interlamellar spacing of the eutectic structure increases from approximately 20 μm to 80 μm , consistent with the reduced undercooling at slower cooling rates. In Figure 9C and D, lamellar faults (highlighted by red circles) can be observed within the regular lamellar eutectic structure. These lamellar faults arise from the rotation of the eutectic Al and Al_3Er phases to achieve favorable low-energy crystallographic orientations.

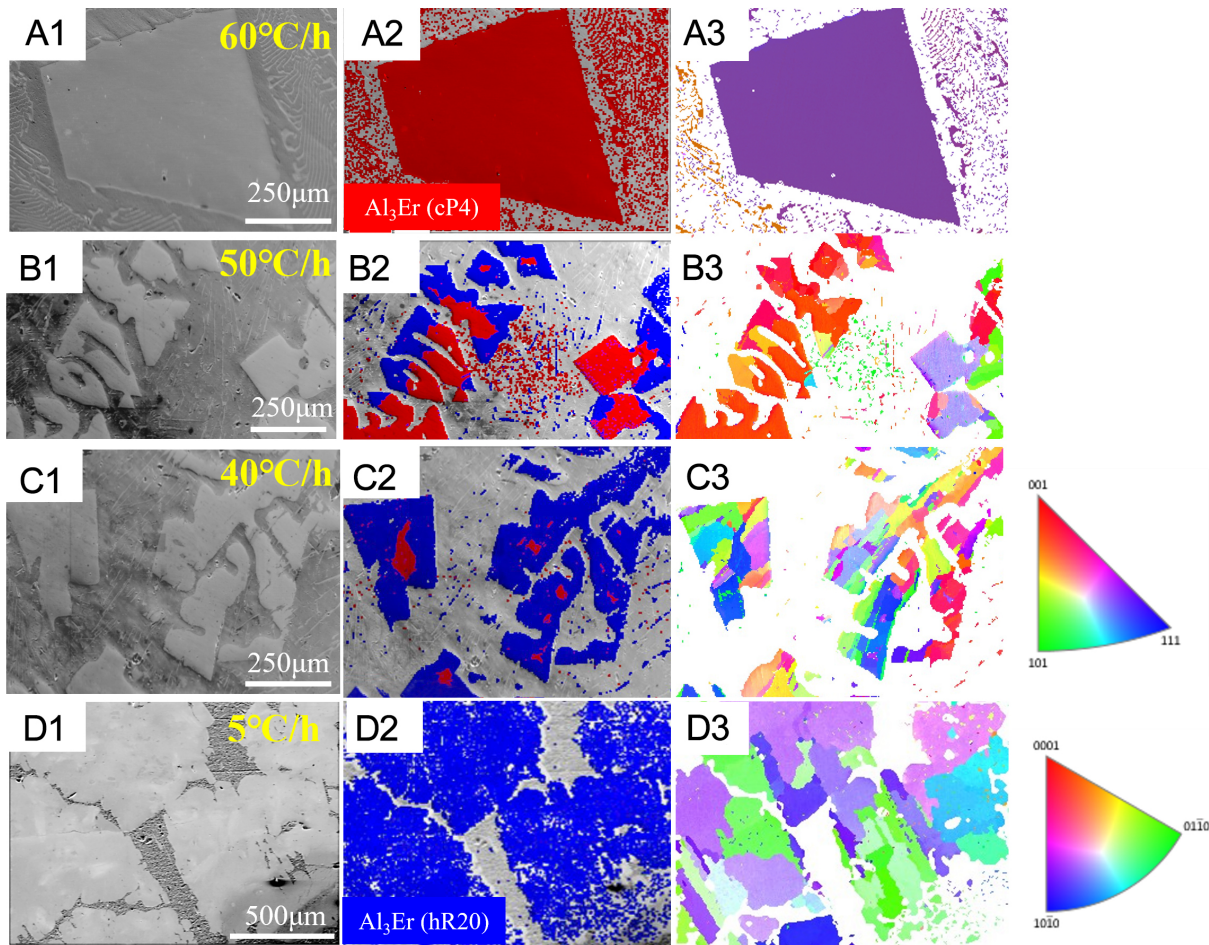


Figure 7. EBSD orientation maps of Al_3Er in samples cooled at different rates: (A) 60 °C/h; (B) 50 °C/h; (C) 40 °C/h; (D) 5 °C/h; (1) microstructure diagram; (2) phase distribution diagram; (3) IPF maps. EBSD: Electron backscatter diffraction; IPF: inverse pole figure.

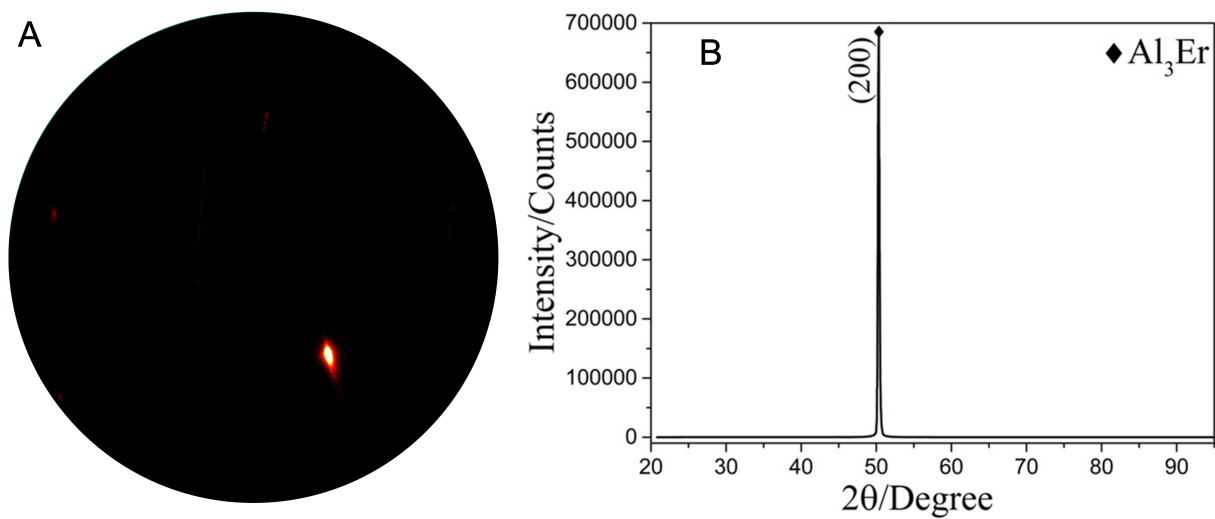


Figure 8. Micro-XRD diffraction results of primary cP4-structured Al_3Er phase in the sample cooled at 60 °C/h: (A) diffraction spots from the red cross in Figure 2; (B) diffraction pattern from the red cross in Figure 2. XRD: X-ray diffraction.

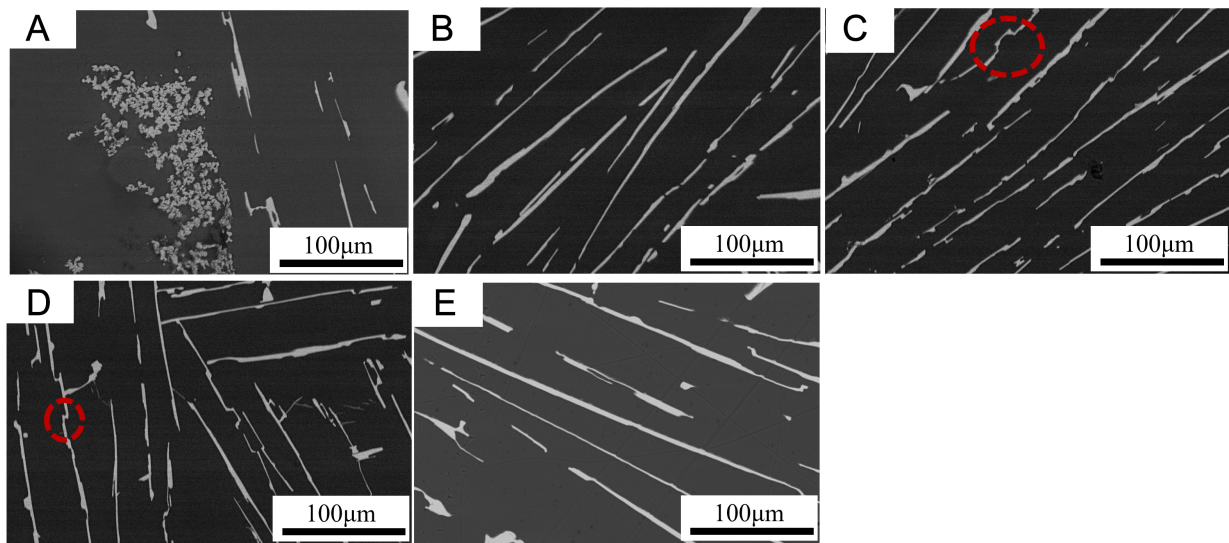


Figure 9. SEM micrographs of eutectic structures cooled at different rates. (A) 60 °C/h; (B) 50 °C/h; (C) 40 °C/h; (D) 30 °C/h; and (E) 5 °C/h. SEM: Scanning electron microscopy.

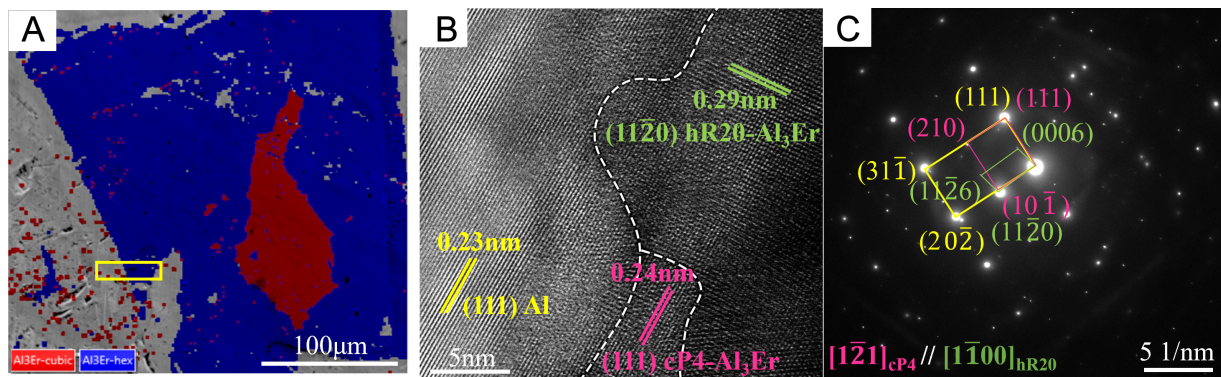


Figure 10. TEM characterization of the Al/Al₃Er interface in the alloy cooled at 40 °C/h. (A) Backscattered electron image showing the location of TEM specimen preparation; (B) HRTEM image of the Al/Al₃Er interface and (C) corresponding SAED pattern. TEM: Transmission electron microscopy; HRTEM: high-resolution transmission electron microscopy; SAED: selected-area electron diffraction.

Orientation relationship between the cP4- and hR20-structured Al₃Er phases

At cooling rates of 50 °C/h and 40 °C/h, two different structures of the Al₃Er phase coexist, indicating an ongoing polymorphic transformation and the potential existence of an orientation relationship between these two phases. A site-specific lift-out specimen containing both Al₃Er phase structures was extracted from the sample cooled at 40 °C/h [Figure 7C] using FIB and subsequently examined by TEM. The specimen was lifted out from a specific location identified in the EBSD phase distribution map, as marked by the yellow box in Figure 10A. Figure 10B shows a HRTEM image in which the phase interfaces are clear, as indicated by the white dashed lines. Figure 10C presents the selected-area electron diffraction (SAED) pattern, confirming the presence of three sets of diffraction spots from the three different phases, as indexed in the figure. The orientation relationship between the cP4-structured Al₃Er phase and the hR20-structured Al₃Er phase is $[1\bar{2}1]_{cP4} // [1\bar{1}00]_{hR20}$, $(111)_{cP4} // (0006)_{hR20}$. Given the lower symmetry of the rhombohedral hR20 structure compared with the cubic cP4 structure, multiple crystallographically equivalent orientation variants may exist.

DISCUSSION

The structural transformation and thermodynamically stable structure of the Al₃Er phase

As shown in Figures 3 and 7, the structure of the Al₃Er phase varies with cooling rate, gradually transforming from the cP4 structure to the hR20 structure as the cooling rate decreases. Since slower cooling rates produce crystal structures at room temperature that more closely approach the equilibrium state^[25–27], this experimental result demonstrates that the hR20-structured Al₃Er phase is more stable than the cP4-structured Al₃Er phase. This result does not support the conclusion presented in the Al–Er phase diagrams^[4,5].

Furthermore, the observations in Figure 7 show that the average grain size of the polycrystalline hR20-structured Al₃Er phase obtained at 5 °C/h is 92 μm, which is unexpectedly much smaller than that of the single-crystalline cP4-structured Al₃Er phase obtained at 60 °C/h (211 μm). This finding contradicts the classical solidification principle that a reduction in cooling rate increases grain size^[26]. To clarify whether the hR20-structured Al₃Er phase precipitates directly from the liquid or forms through a polymorphic transformation from the cP4-structured Al₃Er phase, classical nucleation theory calculations were performed^[26,28].

The driving force for nucleation of a phase from the melt can be calculated using^[29]:

$$\Delta G_V \approx \frac{L_m \cdot \Delta T}{T_m} \quad (1)$$

Where L_m is the latent heat of fusion per unit volume. ΔT is the undercooling and T_m is the melting point of Al₃Er, which is 1,603 K^[4,5].

The critical nucleation energy and the steady-state nucleation rate are calculated using^[28]:

$$\Delta G^* = \frac{16\pi\gamma_{SL}^3}{3(\Delta G_V)^2} \quad (2)$$

$$J = N_v Z \beta^* \exp\left(-\frac{\Delta G^*}{k_B T}\right) \exp\left(-\frac{Q_d}{RT}\right) \quad (3)$$

where γ_{SL} is the solid-liquid interfacial energy. N_v is the number of nucleation sites per unit volume. The Zeldovich factor^[28] is given by $Z = \frac{V_\alpha (\Delta G_V)^2}{8\pi\sqrt{\gamma_{SL}^3 k_B T}}$, where V_α is the average atomic volume of Al₃Er^[3]. $k_B = 1.38 \times 10^{-23}$ J/K is Boltzmann's constant. $R = 8.314$ J/(mol · K) is the molar gas constant. The rate of atomic attachment to a growing embryo^[28] is $\beta^* = \frac{16\pi\gamma_{SL}^2 D_L}{(\Delta G_V)^2 a^4}$. D_L is the diffusion coefficient in the liquid. a is the characteristic atomic spacing. Q_d is the activation energy for atomic transfer across the liquid-solid interface^[30].

For three-dimensional equiaxed solidification, the average grain diameter d is related to the steady-state nucleation rate J and the grain growth velocity G by the following relationship^[26,28]:

$$d \propto \left(\frac{G}{J}\right)^{1/4} \quad (4)$$

Under similar undercooling conditions, G is determined by the crystal structure^[26]. Since both cP4 and hR20 are Al₃Er phases, the atomic attachment kinetics at the solid-liquid interface are not expected to differ substantially; it is reasonable to assume $G_{cP4} \approx G_{hR20}$. Consequently, the difference in grain size resulting from the direct solidification of the two structures is predominantly governed by the nucleation rate J .

From Equations (1)–(3), the volumetric driving force ΔG_v increases monotonically with undercooling ΔT , the critical nucleation energy ΔG^* decreases monotonically with increasing ΔG_v , and the nucleation rate J therefore increases monotonically with ΔT . This directional relationship is independent of any specific parameter values. Combined with Equation (4), a lower nucleation rate necessarily leads to larger grains. However, the experimentally measured grain size of the Al_3Er phase (hR20-structured) cooled at 5 °C/h is unexpectedly smaller than that of the Al_3Er phase (cP4-structured) cooled at 60 °C/h, which directly contradicts the theoretical prediction. The irreconcilable discrepancy rules out the possibility that the hR20-structured Al_3Er phase precipitates directly from the liquid.

Therefore, the plausible transformation pathway is that the primary cP4-structured Al_3Er phase precipitates first from the liquid and grows into a single crystal. Then, during slow cooling, the hR20-structured Al_3Er phase nucleates heterogeneously at the boundaries of the cP4-structured Al_3Er phase through a polymorphic transformation and grows, finally forming a polycrystalline hR20 microstructure.

This conclusion is in perfect agreement with the EBSD results in Figure 7. The cP4- and hR20-structured Al_3Er phases are iso-compositional polymorphs, and the first-principles calculations indicate that the difference in their formation energies is very small^[22,23], implying a limited chemical driving force for the phase transformation. Therefore, rapid cooling suppresses diffusion and inhibits the transformation, whereas slow cooling extends the residence time of the sample in the high-temperature regime, providing sufficient kinetic conditions for atomic rearrangement. Furthermore, the hR20-structured Al_3Er phase nucleates at the boundaries of the cP4-structured Al_3Er particles, i.e., at the cP4-structured Al_3Er /Al phase interface, which substantially reduces the critical nucleation energy required to initiate the transformation^[26,28].

The hR20-structured Al_3Er phase particles obtained at a slow cooling rate are polycrystalline, with each grain having nucleated independently at the phase boundary of the cP4-structured Al_3Er grain and subsequently grown. The relatively high nucleation density leads to a fine final grain size for the hR20-structured Al_3Er grains. By comparison, the cP4-structured Al_3Er phase particles formed at a fast cooling rate are single crystals, having grown from a single nucleus in the melt. Therefore, the smaller grain size of the polycrystalline hR20-structured Al_3Er phase particles is a consequence of the polymorphic transformation.

The cooling-rate-dependent structural evolution from cP4 to hR20 directly influences the intrinsic mechanical properties of the Al_3Er phase. Beyond this intrinsic difference, the structural transformation has important implications for the strengthening performance of Er-containing aluminum alloys. In these alloys, the strengthening effect of cP4-structured Al_3Er precipitates relies on their coherency with the α -Al matrix. The lattice mismatch between cP4- Al_3Er and the α -Al matrix is only $4.08 \pm 0.09\%$. This relatively small lattice mismatch facilitates the formation of coherent or semi-coherent interfaces that generate the elastic strain fields responsible for effective dislocation pinning. Although the present study investigates primary Al_3Er particles formed during solidification, the structural evolution identified here is also relevant to nanoscale Al_3Er strengthening precipitates, for which interface coherency with the α -Al matrix is critical to precipitation strengthening. In contrast, the hR20-structured Al_3Er precipitates possess rhombohedral symmetry, which is less compatible with the cubic α -Al lattice than the cP4 structure. Therefore, hR20-structured precipitates in the Al matrix would be expected to exhibit reduced coherency with the Al matrix, potentially decreasing their contribution to precipitation strengthening. Furthermore, reduced interface coherency may increase the interfacial energy, thereby decreasing the resistance to precipitate coarsening and potentially reducing creep resistance at elevated temperatures. Consequently, an uncontrolled cP4 to hR20 transformation during solidification or high-temperature service could adversely affect both the room-temperature strength and the high-temperature stability of Er-containing aluminum alloys. Although direct measurements of strengthening in Er-containing aluminum alloys are beyond the scope of the present work,

the structural evolution identified here has important implications for alloy performance. The critical cooling rate of 36 °C/h determined in this work therefore provides practical guidance for avoiding such structural evolution during alloy processing.

The critical cooling rate of the Al₃Er phase

Based on the above results, the polymorphic transformation from the cP4 structure to the hR20 structure is a thermally activated diffusion process. That is, a critical cooling rate exists at which the hR20-structured Al₃Er phase fully precipitates. The variation of the transformed volume fraction f with time t can be described by the Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation^[28,31]:

$$f = 1 - \exp \left[-K t_{\text{eff}}^n \right] \quad (5)$$

where f is the volume fraction of the transformed hR20-structured Al₃Er phase, K is the temperature-dependent rate constant, n is the Avrami exponent (which depends on the transformation mechanism), and $t_{\text{eff}} = \frac{\Delta T}{v}$ is the effective residence time, where v is the cooling rate and ΔT is the temperature interval over which the transformation occurs.

f_{hR20} was determined from the area fraction of the hR20-structured phase in the EBSD phase distribution maps in Figure 7. At a cooling rate of 50 °C/h, $f_{\text{hR20}} = 0.4$, while at 40 °C/h, $f_{\text{hR20}} = 0.9$. According to the Al-Er binary phase diagram, the liquidus temperature of the Al-30Er hypereutectic alloy is approximately 860 °C, i.e., 1,133 K^[4,5]. The primary Al₃Er phase precipitates below the liquidus and persists throughout the subsequent cooling process; therefore, $\Delta T = 1,133 \text{ K} - 773 \text{ K} = 360 \text{ K}$.

By fitting the two sets of data using the JMAK equation, the linear regression result shows that $n \approx 6.8$ and $K \approx 8.3 \times 10^{-7}$. With only two data points, n and K are fully determined but the goodness of fit cannot be assessed. Nevertheless, the derived values provide a reasonable estimate for subsequent calculations. Taking $f_{\text{crit}} = 0.99$ as the criterion for completion of the transformation, the corresponding constant $C = -\ln(0.01) \approx 4.605$. The critical time is then calculated as:

$$t_{\text{crit}} = \left(\frac{C}{K} \right)^{1/n} = \left(\frac{4.605}{8.3 \times 10^{-7}} \right)^{1/6.8} \approx 10 \text{ h} \quad (6)$$

The corresponding critical cooling rate is:

$$V_{\text{crit}} = \frac{\Delta T}{t_{\text{crit}}} = \frac{360 \text{ K}}{10.0 \text{ h}} = 36 \text{ °C/h} \quad (7)$$

This value is in excellent agreement with the experimental observations. A slow cooling rate of 30 °C/h or 5 °C/h, being below the critical rate, allows the transformation to proceed completely and yields sole hR20-structured Al₃Er phase. A fast cooling rate of 60 °C/h, which exceeds the critical rate, suppresses the transformation and yields the cP4-structured Al₃Er phase. Intermediate cooling rates of 50 °C/h and 40 °C/h result in a two-phase coexistence microstructure. This finding provides strong evidence that the polymorphic transformation requires a sufficiently long high-temperature residence time and is governed by a well-defined critical cooling rate. Slow cooling rates offer sufficient time for the transformation to nucleate and grow, whereas fast cooling rates lead to its arrest.

It should be pointed out that the JMAK analysis is based on only two available data points; the fitted n and k values are therefore used primarily as engineering parameters for the extrapolated estimation of the critical

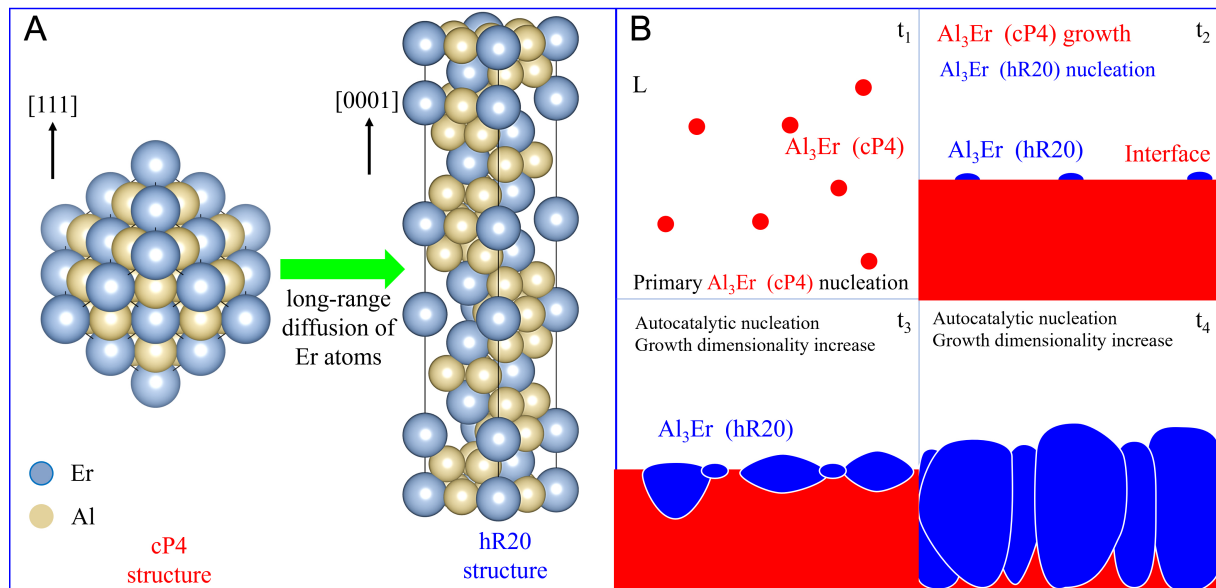


Figure 11. Schematic diagram of the cP4 to hR20 polymorphic transformation mechanism. (A) Distribution of closely packed plane atoms in the two-structured Al₃Er phases; (B) Initial state (t_1) and the three transformation stages (t_2 – t_4) of microstructure evolution during polymorphic transformation. t_1 : Initial formation of cP4-structured Al₃Er phase; t_2 : Isolated nucleation of the hR20-structured Al₃Er phase on phase boundaries of cP4-structured Al₃Er phase; t_3 : Autocatalytic nucleation and growth dimensionality transition of hR20-structured Al₃Er phase; t_4 : Formation of polycrystalline hR20-structured Al₃Er.

cooling rate. The unusually high Avrami exponent of 6.8 reveals the pronounced self-acceleration character of the transformation. Regarding the determination of the critical cooling rate, its reliability does not rely on the precise value of n but is directly constrained by the two measured data points. No matter what kinetic model is adopted, the fact that the transformation from $f_{\text{hR20}} = 0.4$ to $f_{\text{hR20}} = 0.9$ is completed within a difference of only about 1.8 h in effective residence time itself demonstrates an extremely high cooling rate sensitivity of the transformation. The critical cooling rate thus estimated is highly self-consistent with the experimental observations, further confirming the reasonableness of the estimate.

The diffusional nature of the cP4 to hR20 polymorphic transformation in Al₃Er

Classical polymorphic transformation from a cubic-based structure to a rhombohedral-based structure, such as the fcc to hcp transition in Co-alloy^[32] and Fe₄₀Mn₄₀Co₁₀Cr₁₀^[33], and the 3C to 6H transformation in SiC^[28,34], typically occurs through the glide of Shockley partial dislocations and is classified as a diffusionless displacive phase transformation. However, in this study, the cP4 to hR20 transition exhibits remarkable diffusion-controlled characteristics. The kinetic analysis presented in Section 4.2 reveals a well-defined critical cooling rate of 36 °C/h for the transformation, along with a high Avrami exponent of nearly 6.8, which is characteristic of diffusion-controlled transformations with site-saturated nucleation and three-dimensional growth. These observations indicate that the transformation is not a fault-mediated displacive process but rather a diffusion-displacive coupled phase transformation.

In the cP4 structure, Er atoms are uniformly distributed on every {111} close-packed plane with an Er:Al ratio of 1:3. In contrast, the hR20 structure exhibits a heterogeneous layered arrangement of Er atoms, wherein certain layers are enriched in Er while others consist entirely of Al, resulting in a periodic compositional modulation, as seen in Figure 11A. Therefore, the transformation from the cP4 to the hR20 structure requires not only a change in the stacking sequence but also long-range diffusion of Er atoms between different stacking layers to achieve the site-occupancy reconstruction. This diffusional requirement governs the thermally activated transformation; i.e., within the effective temperature range, the interlayer diffusion of Er atoms governs the transformation rate. As the temperature decreases, the competition of the decreasing diffusion coefficient and the increasing driving force leads to the characteristic C-curve and the

critical cooling rate.

SAED pattern in Figure 10 determined the orientation relationship is $[1\bar{2}1]_{\text{cP4}} // [1\bar{1}00]_{\text{hR20}}$, $(111)_{\text{cP4}} // (0006)_{\text{hR20}}$. This parallel relationship of close-packed planes is geometrically consistent with the glide of Shockley partial dislocations on $\{111\}$ planes, indicating the polymorphic transformation involves periodic glide of $\frac{1}{6}\langle 112 \rangle$ partial dislocation on the (111) planes. The parallel close-packed planes serve as the crystallographic prerequisite for the displacive component in the diffusion-displacive coupling mechanism that the glide of partial dislocations alters the stacking sequence, while the interlayer diffusion of Er atoms achieves site-occupancy reconstruction, with both processes proceeding cooperatively on the same close-packed plane. Therefore, this orientation relationship provides crystallographic evidence for the involvement of a displacive component in the transformation.

Furthermore, the Avrami exponent of 6.8 is considerably higher than the typical range of 1–4 in classical JMAK theory. This result indicates that during the polymorphic transformation, the synergistic effects of autocatalytic nucleation, the self-assembly of diffusion channels and the transition in growth dimensionality come into play^[35–37].

In the early stages of the cP4 to hR20 transformation, the hR20 phase nucleates primarily at preferred sites on phase boundaries of the cP4-structured phase, and the nucleation rate is relatively low. Once a nucleus forms, a semi-coherent interface develops between the hR20 and cP4 phases, as seen in Figure 10, and this interface is characterized by substantial lattice distortion and a high vacancy concentration, which provides a high diffusion rate channel^[38,39]. When these interfacial regions become interconnected along the cP4-structured Al₃Er/Al phase interfaces, they act as highly efficient catalytic sites for subsequent nucleation. As the volume fraction of the hR20-structured phase increases, the total interfacial area expands rapidly, and the number of effective nucleation sites grows exponentially, causing the nucleation rate to rise dramatically^[35,36,40]. This positive feedback loop continues until neighboring hR20 particles impinge upon one another, after which the nucleation rate begins to decrease.

The EBSD phase distribution maps in Figure 7 show that in partially transformed samples, the hR20-structured phase is distributed preferentially along the cP4 grain boundaries, forming a continuous or semi-continuous network that surrounds the untransformed cP4 cores, which is a characteristic morphological feature of autocatalytic nucleation. Moreover, the grain size of the polycrystalline hR20-structured phase formed at the lower cooling rate is much smaller than that of the cP4-structured phase formed at the higher cooling rate, indicating a substantially higher nucleation density. A statistical analysis was performed using the EBSD IPF maps in Figure 7. Within the same field of view, the number density of hR20-structured phase in the 40 °C/h sample is about 3.4 times that in the 50 °C/h sample. As the transformation fraction increases from 0.4 to 0.9, the number density increases by a factor of 3.4, exceeding the increase expected from linear growth. Such an increase is characteristic of an autocatalytic nucleation mechanism, in which new nuclei are generated rapidly at the transformation interface. Furthermore, the coefficient of variation of the hR20-structured phase grain size distribution in the 40 °C/h sample is 0.68, larger than the value of 0.41 measured for the 50 °C/h sample. This indicates the coexistence of fine grains and coarse grains, consistent with the broad grain size distribution expected for autocatalytic nucleation. These results provide direct evidence for the autocatalytic nucleation mechanism.

If the nucleation rate increases exponentially with time, i.e., $\dot{N}(t) = \dot{N}_0 \exp(\lambda t)$, the effective Avrami exponent can be expressed as $n_{\text{eff}} = a + bm + \lambda t^*$, where λ is the autocatalytic coefficient and t^* is the time at which the transformation is half complete^[41]. When the autocatalytic effect is pronounced, n_{eff} can substantially exceed 4. It has been reported that when nucleation occurs on grain boundaries and there is an “infection” effect between nucleation sites, the Avrami exponent can reach values as high as 5 to 7^[40].

In addition, the dynamic transition of growth dimensionality further contributes to the elevated Avrami exponent. The formation of the hR20-structured phase can be divided into two stages^[37]. During the initial stage, hR20-structured phase elongates one-dimensionally along phase boundaries of the primary cP4-structured Al_3Er particles, with growth controlled by grain-boundary diffusion, corresponding to a growth dimensionality of 1. When the phase boundaries are completely covered by the hR20-structured phase, the growth mode shifts to two- or three-dimensional thickening into the grain interiors, corresponding to growth dimensionalities of 2 and 3. The schematic diagram of the polymorphic transformation process is shown in Figure 11B. The EBSD micrographs in Figure 7 reveal that the hR20-structured phase grains are elongated at grain boundaries, which is a typical characteristic of one-dimensional growth, while being equiaxed or spherical within the grains, which is a typical characteristic of two- or three-dimensional growth. These results are consistent with the model of the growth-dimensionality transition. This change in growth dimensionality means that the dimensionality parameter evolves during the transformation. The effective Avrami exponent becomes a weighted average of contributions from different stages. Higher-dimensionality growth occurs in the middle and later stages, when the volume fraction increases most rapidly. As a result, its contribution to the overall Avrami exponent is amplified, producing the markedly high apparent Avrami exponent (n) obtained by fitting^[37].

The diffusion-displacive coupled cP4 to hR20 polymorphic transformation manifests itself in the fact that the layer difference in Er atom occupancy necessitates long-range diffusion during the phase transformation, which in turn dictates the existence of a critical cooling rate of 36 °C/h. The autocatalytic nucleation, the self-assembly of diffusion channels and the transition in growth dimensionality during the transformation lead to an anomalously high Avrami exponent of 6.8, which has a clear mathematical foundation and is experimentally supported in this work.

In summary, the cP4 to hR20 polymorphic transformation can be divided into three stages. At the first stage, the primary cP4-structured Al_3Er phase has precipitated from the liquid as a single crystal, and Er atoms at grain boundaries begin interlayer migration through diffusion, with preferential sites becoming potential nucleation sites. The nucleation rate is extremely low during this stage, corresponding to t_2 schematic diagram in Figure 11B. At the second stage, which is the autocatalytic acceleration period, once the first hR20-structure Al_3Er nucleus forms at the grain boundary, its interface becomes a catalytic site, and the nucleation rate increases exponentially. The number density increases significantly. The grains extend one-dimensionally along the grain boundary, as shown in t_3 of Figure 11. At the third stage, after the grain boundaries are covered, growth shifts to thickening toward the grain interior, ultimately forming a polycrystalline hR20-structured Al_3Er microstructure. The essence of this model lies in the coupling of diffusion and displacement. Diffusion controls the transformation rate, which determines the critical cooling rate, while displacement assists in stacking rearrangement.

CONCLUSIONS

(1) After the solidification process, the cP4-structured Al_3Er phase grains are formed under a fast cooling rate of 60 °C/h, the hR20-structured Al_3Er phase grains are formed under a slow cooling rate of 30 °C/h or below, and the two structures coexist under an intermediate cooling rate of 50 or 40 °C/h. The thermodynamically stable structure of the Al_3Er phase is the hR20 structure.

(2) The lattice constant of the cP4-structured Al_3Er phase is 0.4212 ± 0.0003 nm, with a lattice mismatch of $4.08 \pm 0.09\%$ relative to the Al matrix. The lattice constants of the hR20-structured Al_3Er phase are $a = 0.6036 \pm 0.0006$ nm, $c = 3.5705 \pm 0.0004$ nm. The orientation relationship between the cP4-structured and hR20-structured Al_3Er phases is: $[1\bar{2}1]_{\text{cP4}} // [1\bar{1}00]_{\text{hR20}}$, $(111)_{\text{cP4}} // (0006)_{\text{hR20}}$.

(3) At a cooling rate of 60 °C/h, the primary cP4-structured Al_3Er phase is single-crystalline with an average grain size of 211 μm , exhibiting straight boundaries and the eutectic structure consists of both flocculent and lamellar features. In contrast, at a cooling rate of 5 °C/h, the primary hR20-structured Al_3Er phase is polycrystalline with an average grain size of 92 μm , displaying wavy boundaries, and its eutectic structure is lamellar.

(4) Classical nucleation calculations indicate that the hR20-structured Al_3Er cannot precipitate directly from the liquid. The feasible transformation pathway is that during slow cooling, the cP4-structured Al_3Er phase precipitates first and the hR20-structured Al_3Er phase subsequently nucleates heterogeneously at the grain boundaries.

(5) JMAK kinetic analysis shows that the critical cooling rate for the polymorphic transformation is 36 °C/h. The polymorphic transformation is a diffusion-displacive coupled phase transformation, in which the interlayer difference in Er atom occupancy necessitates long-range diffusion of Er atoms. The high Avrami exponent of 6.8 originates from autocatalytic nucleation, a transition in growth dimensionality, and the self-assembly of diffusion channels.

DECLARATIONS

Authors' contributions

Made substantial contributions to conception and design of the study and performed data analysis and interpretation: Wang, J.; Gao, K.; Xiong, X.; Ning, J.
performed data acquisition and provided administrative, technical, and material support: Wu, X.; Wen, S.; Wei, W.; Huang, H.; Nie, Z.; Zhou, D.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

AI and AI-assisted tools statement

Not applicable.

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Conflicts of interest

Zhou, D. is affiliated with Yin Bang Clad Material Co. Ltd., while the other authors declared that they have no conflicts of interest.

Ethical approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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