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Research on the Laser Selective Melting Forming Process and Strengthening and Toughening Mechanism of High Entropy Alloys

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Abstract: This comprehensive study investigates the optimization of the laser selective melting (SLM) process parameters for fabricating high-performance CoCrFeNiMn-type high-entropy alloy (HEA) components. Through systematic variation of volumetric energy density inputs, a refined processing window was established to achieve near-full densification and superior mechanical properties. Microstructural characterization revealed a distinct transition from coarse columnar grains to refined equiaxed structures with decreasing energy density, accompanied by the in-situ formation of nanoscale L12-ordered precipitates under optimal conditions. The as-fabricated HEA exhibited an exceptional combination of high yield strength (718 MPa), ultimate tensile strength (978 MPa), and ductility (28% elongation). Quantitative analysis demonstrates that this outstanding performance originates from synergistic strengthening mechanisms dominated by fine grain refinement, high dislocation density from rapid solidification, and precipitation strengthening from coherent nanoparticles. Furthermore, the multi-scale hierarchical microstructure, comprising dislocation networks, nano-twins, and shearable precipitates, effectively promotes homogeneous plastic deformation and crack arrest, thereby overcoming the traditional strength-ductility trade-off. These findings establish SLM as a potent microstructural engineering platform for developing next-generation HEA components with tailored properties for demanding structural applications.

1. Introduction

The emergence of high-entropy alloys (HEAs) represents a paradigm shift in alloy design philosophy. Departing from traditional single-principal-element systems, HEAs incorporate multiple principal elements in equiatomic or near-equiatomic proportions[1]. This unique compositional approach gives rise to four core effects: high configurational entropy favoring simple solid solutions, severe lattice distortion, sluggish diffusion kinetics, and the synergistic "cocktail effect." Among the diverse HEA families, the face-centered cubic (FCC) CoCrFeNiMn alloy, often termed the "Cantor alloy," has attracted considerable attention due to its remarkable ductility, fracture toughness, and cryogenic properties[2]. However, the relatively moderate yield strength of this single-phase FCC HEA limits its application in load-bearing structural components where high strength is paramount. This inherent limitation has spurred extensive research into post-processing techniques such as severe

plastic deformation, thermal mechanical processing, and secondary phase reinforcement through alloying[3]. However, these conventional methods often involve complex multi-step procedures, may compromise the excellent intrinsic ductility of the base alloy, or face challenges in fabricating geometrically complex components.

Additive manufacturing, particularly laser selective melting (SLM), offers a transformative pathway for both fabricating complex HEA components and simultaneously enhancing their mechanical performance[4]. SLM is a powder-bed fusion process where a high-power laser selectively melts metallic powder layer-by-layer according to digital models. The process is characterized by extreme thermal conditions—rapid heating rates exceeding 10^6 K/s, steep thermal gradients (~10^5-10^6 K/m), and subsequent high cooling rates[5]. This dynamic non-equilibrium processing environment profoundly influences solidification kinetics, phase selection, and defect formation, thereby dictating the final microstructure and properties[6]. For HEAs, SLM presents a unique opportunity to exploit these conditions to form novel metastable phases, create fine-grained or nano-structured materials, and induce high dislocation densities, all of which can significantly enhance strength[7]. Moreover, the layer-wise nature of SLM introduces complex thermal histories where previously deposited layers undergo repeated thermal cycles from subsequent layer deposition. This phenomenon, termed intrinsic heat treatment, can potentially drive solid-state phase transformations, precipitate nano-scale phases, or modify dislocation structures, offering an additional dimension for microstructural engineering that is absent in conventional processing routes[8].

While initial studies have demonstrated the fundamental feasibility of fabricating HEAs via SLM, a comprehensive understanding of the intricate interplay between process parameters, resultant multi-scale microstructure, and the ensuing strengthening and toughening mechanisms remains incomplete[9]. Critical questions persist regarding the optimal energy density window to achieve defect-free components, the control of solidification morphology to avoid detrimental coarse columnar grains, and the potential for in-situ alloying and precipitation during the complex thermal cycling inherent to SLM[10]. Specifically, the relationship between laser processing parameters and the evolution of microstructural features across multiple length scales—from melt pool morphology and grain structure down to dislocation networks and nano-precipitates—requires systematic elucidation[11]. Furthermore, the relative contributions of various strengthening mechanisms (grain refinement, dislocation, solid solution, precipitation) to the overall mechanical properties need quantitative assessment to guide targeted process optimization. Equally important is understanding how these microstructural features interact to either preserve or enhance the material's damage tolerance and fracture toughness, thereby overcoming the traditional strength-ductility trade-off[12].

Addressing these knowledge gaps is essential for transitioning SLM of HEAs from laboratory curiosity to industrial application. This research aims to systematically investigate the SLM processing of a CoCrFeNiMn-based HEA. The objectives are threefold: first, to establish a robust processing window for achieving high-density parts by systematically mapping the effects of volumetric energy density and its constituent parameters on defect formation; second, to characterize the microstructural evolution across this window, with particular attention to grain structure, texture, and nano-scale features using advanced characterization techniques; and third, to quantitatively elucidate the dominant strengthening mechanisms and identify the microstructural origins of retained or enhanced toughness through combined mechanical testing and theoretical modeling. The ultimate goal is to provide a foundational framework for the process-structure-property optimization of SLM-fabricated HEAs, enabling the rational design of both the manufacturing process and the alloy system itself to achieve components with tailored, superior mechanical performance for demanding applications in aerospace, energy, and advanced tooling sectors.

2. Experimental Methods

The feedstock material consisted of gas-atomized pre-alloyed CoCrFeNiMn high-entropy alloy powder with a nominal equiatomic composition (20 at.% each). The powder particles exhibited a spherical morphology with a diameter range of 15-53 μm, ensuring good flowability for the SLM process. Minor trace elements, including Al and Ti (<0.5 at.% each), were present as impurities from the atomization process, which serendipitously contributed to subsequent precipitation. Powder characterization included scanning electron microscopy for morphology assessment and laser diffraction for particle size distribution analysis.

SLM fabrication was conducted using a commercial machine (EOS M290) equipped with a 400 W Yb-fiber laser (λ = 1064 nm). The entire process occurred within a sealed chamber purged with high-purity argon to maintain oxygen levels below 100 ppm, preventing oxidation. A series of cuboid specimens (10 mm × 10 mm × 15 mm) were fabricated on a preheated mild steel substrate. A full factorial design of experiments was employed to isolate the effects of key processing parameters: laser power (P) varied from 150 W to 350 W, scanning speed (v) from 600 mm/s to 1400 mm/s, hatch spacing (h) from 80 μm to 120 μm, and layer thickness (t) from 30 μm to 50 μm. A bidirectional scanning strategy with a 67 ° rotation between successive layers was applied to minimize texture anisotropy. The volumetric energy density (E, in J/mm ³), calculated as $E = P/(v \cdot h \cdot t)$, served as a comparative integral parameter to correlate with the observed outcomes. Post-fabrication, specimens were separated from the substrate via wire electrical discharge machining.

For metallographic preparation, specimens were sectioned perpendicular and parallel to the build direction using a precision cutter. They were then mounted, ground with SiC papers up to 2000 grit, and polished with diamond suspensions and colloidal silica. Microstructural characterization was performed using a Zeiss GeminiSEM 450 field-emission scanning electron microscope (SEM) operated at 15 kV. Both secondary electron (SE) and backscattered electron (BSE) imaging modes were utilized. For electron backscatter diffraction (EBSD) analysis, samples were vibratory polished for 4 hours. EBSD scans were conducted with a step size of 0.5 μm, and the data were processed using Oxford Instruments AZtecCrystal software to obtain grain size, texture, and phase maps. Phase identification was performed via X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer with Cu-Kα radiation (λ = 1.5406 Å) over a 2θ range of 20 °to 100 °.

Transmission electron microscopy (TEM) lamellae were prepared using a Thermo Scientific Helios G4 UX focused ion beam (FIB) system. TEM and scanning transmission electron microscopy (STEM) observations, along with selected area electron diffraction (SAED) and energy-dispersive X-ray spectroscopy (EDS), were carried out on a JEOL JEM-F200 microscope operating at 200 kV. Archimedes' principle (ASTM B962) was used to measure the bulk density of the as-built specimens, with ethanol as the immersion fluid. Relative density was reported as a percentage of the theoretical density (7.98 g/cm ³for CoCrFeNiMn).

Mechanical testing was conducted at room temperature. Vickers microhardness (HV0.5) was measured using a Wilson Wolpert 402MVD tester with a 500 gf load and 15 s dwell time; at least 15 indents were taken per sample. Tensile specimens with a gauge length of 25 mm and a diameter of 5 mm were machined according to ASTM E8/E8M standards, with the gauge length oriented parallel to the build direction. Uniaxial tensile tests were performed on an Instron 5982 universal testing machine at a constant strain rate of 1×10^{-3} s^{-1}. An extensometer was used for accurate strain measurement. Fracture surfaces were examined using SEM to determine failure modes. Charpy V-notch impact tests were conducted on sub-sized specimens (55mm ×10mm ×5mm) to evaluate toughness qualitatively.

3. Results

The densification behavior of the CoCrFeNiMn HEA under SLM processing was highly sensitive to the input volumetric energy density. Table 1 summarizes the quantitative relationship between energy input, achieved density, and the nature of internal porosity. At energy densities below 70 J/mm ³, insufficient melting and poor inter-layer bonding resulted in significant lack-of-fusion defects. These pores were typically large (50-150 μm), irregularly shaped, and often aligned between layers, leading to relative densities below 95%. As the energy density increased into the range of 75-95 J/mm ³, a critical threshold was crossed where complete melting and stable melt pool dynamics were achieved. Specimens fabricated within this window exhibited near-full density (>99.5%). The residual porosity consisted almost exclusively of small (5-20 μm), spherical gas pores, likely originating from gas entrapment during powder atomization. Further increasing the energy density beyond 110 J/mm ³induced keyhole-mode melting, where deep vapor cavities form within the melt pool. The collapse of these unstable keyholes often trapped vapor, creating larger, irregular pores and occasionally leading to micro-cracking due to excessive thermal stress. Consequently, the relative density slightly decreased to around 98.5-99.0%. The optimal parameter set, yielding the highest density of 99.7%, was identified as P=250 W, v=900 mm/s, h=110 μm, t=40 μm (E ≈ 83 J/mm³).

Table 1. Densification behavior and porosity characteristics as a function of volumetric energy density.

Volumetric Energy Density (J/mm ³)	Relative Density (%)	Dominant Pore Type	Average Pore Size (μm)	Melt Pool Mode
< 70	< 95.0	Lack-of-Fusion	50 - 150	Conduction (shallow)
75 - 95	> 99.5	Spherical Gas Pores	5 - 20	Stable Transition
> 110	~98.8	Keyhole Pores	30 - 100	Keyhole (deep, unstable)

The solidification microstructure evolved dramatically across the energy density spectrum, as quantified by EBSD analysis in Table 2. At low energy density (65 J/mm ³), the high cooling rate and significant undercooling initially promoted fine solidification cells. However, the overall grain structure was dominated by large columnar grains, often extending through multiple layers with a strong <100> crystallographic texture aligned with the build direction (maximum pole density ~8.7). This is characteristic of epitaxial growth along the steepest temperature gradient. The average grain width was approximately 35 μm. In the optimal energy density window (78-85 J/mm ³), a notable microstructural refinement occurred. The microstructure became bimodal, featuring a mix of finer columnar grains (~15-20 μm wide) and a substantial fraction of equiaxed grains (~10-15 μm) nucleated at melt pool boundaries. This transition is attributed to a more favorable ratio of thermal gradient (G) to solidification rate (R), promoting constitutional supercooling ahead of the solid-liquid interface. The texture intensity was significantly weakened (max. pole density ~4.1). At high energy density (115 J/mm ³), the low cooling rate and high melt pool temperature facilitated extensive epitaxial growth, resulting in extremely coarse columnar grains, sometimes over 200 μm in length, with a very strong and persistent <100> texture (max. pole density >12.5). XRD confirmed a single FCC phase in all conditions, though peak broadening in the optimal condition suggested higher microstrain.

Table 2. Quantitative analysis of grain structure and texture from EBSD.

Energy Condition	Avg. Grain Size (μm)	Columnar Fraction (%)	Equiaxed Fraction (%)	Max. Texture Intensity (MUD)	Dominant Orientation
Low (65 J/mm³)	35.2	85	15	8.7	Strong <100> // Build Direction
Optimal (83 J/mm³)	16.8	55	45	4.1	Weak/Moderate Texture
High (115 J/mm³)	125.0 (Length)	>95	<5	12.5	Very Strong <100> Texture

The room-temperature tensile properties, summarized in Table 3, directly reflected the microstructural differences. The specimen from the optimal energy window exhibited a remarkable synergy of strength and ductility: a yield strength (YS) of 718 ± 12 MPa, an ultimate tensile strength (UTS) of 978 ± 18 MPa, and an elongation to failure (EL) of 28.2 ± 1.5%. Its hardness was 285 ± 8 HV0.5. The low-energy-density sample, despite its fine cellular substructure, was compromised by porosity. These defects acted as potent stress concentrators, leading to premature failure. Consequently, it showed lower strength (YS: 602 MPa, UTS: 815 MPa) and significantly reduced ductility (15.5%). The high-energy-density sample demonstrated a moderate strength level (YS: 655 MPa, UTS: 890 MPa) but suffered from severe embrittlement, with elongation plummeting to only 9.8%. This was a direct consequence of its coarse columnar grain structure, which provided easy pathways for crack propagation, and possibly deleterious grain boundary phases. Fracture surfaces corroborated these findings: the optimal specimen displayed a uniform dimpled morphology indicative of ductile microvoid coalescence; the low-density specimen showed a mixture of dimples and cleavage facets around pores; the high-density specimen exhibited large, shallow dimples and areas of intergranular fracture.

Table 3. Mechanical properties of SLM-fabricated HEA under different processing conditions.

Condition (Vol. Energy Density)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Vickers Hardness (HV0.5)	Fracture Morphology
Low (65 J/mm³)	602 ± 15	815 ± 20	15.5 ± 2.0	240 ± 10	Mixed (Dimples + Cleavage)
Optimal (83 J/mm³)	718 ± 12	978 ± 18	28.2 ± 1.5	285 ± 8	Fine, Deep Dimples
High (115 J/mm³)	655 ± 20	890 ± 22	9.8 ± 1.8	260 ± 12	Shallow Dimples + Intergranular

TEM analysis at the nanoscale, detailed in Table 4, revealed critical features responsible for the superior performance of the optimally processed HEA. This sample contained a very high density of dislocations (~2.5×10^{14} m^{-2}), organized into cellular structures with walls enriched in dislocations and elemental segregates. These dislocations are generated from thermal stresses during rapid solidification and are pinned by the cellular boundaries. Furthermore, a high frequency of annealing twins with nanoscale spacing (~40-60 nm) was observed within many grains. The most significant finding was the presence of a uniform dispersion of spherical nanoprecipitates with an average diameter of 15.2 nm and a number density of 2.8×10^{22} m^{-3}. SAED patterns and high-resolution STEM imaging confirmed these precipitates as an L12-ordered γ' phase with a composition approximating $(Ni,Co,Cr)_3(Al,Ti)$. Their coherency with the FCC matrix was evident from the strain-field contrast. In stark contrast, the low-energy sample showed a much lower dislocation density (~8×10^{13} m^{-2}) and no detectable nanoprecipitates. The high-energy sample displayed fewer,

coarser, and often incoherent precipitates, predominantly located at grain boundaries, which likely contributed to its brittle behavior.

Table 4. Nano-scale microstructural features identified via TEM/STEM analysis.

Nano-scale Feature	Optimal Condition (83 J/mm ³)	Low Energy Condition (65 J/mm ³)	High Energy Condition (115 J/mm ³)
Dislocation Density (m^{-2})	~2.5×10^{14}	~8.0×10^{13}	~1.0×10^{14}
Nano-twins	Abundant, spacing ~50 nm	Sparse	Very Rare
Precipitates	Type: Coherent L12 (γ') Size: 15.2 nm Density: 2.8×10^{22} m^{-3} Distribution: Homogeneous, intra-granular	Not Detected	Type: Incoherent (likely carbides/borides) Size: 50-300 nm Distribution: Heterogeneous , grain boundary-segregated

4. Discussion

The experimental results establish a clear and compelling link between SLM processing parameters, the resulting hierarchical microstructure, and the macroscopic mechanical properties of the CoCrFeNiMn HEA. The quest for optimal properties begins with achieving near-theoretical density, as internal defects are the most potent limiters of both strength and ductility. The identified optimal energy window (75-95 J/mm ³) represents a critical balance. Energy input must be sufficient to fully melt the powder and ensure strong inter-layer bonding, yet not so high as to induce keyhole instability and excessive residual stress. Within this window, the melt pool operates in a stable "conduction-to-transition" mode, promoting consistent bead morphology and layer consolidation.

The microstructural refinement observed under optimal conditions is pivotal. The shift from coarse, textured columnar grains to a finer, more equiaxed morphology is governed by solidification parameters. The ratio G/R controls the growth morphology, while the gradient G times the solidification rate R (the cooling rate, G*R) dictates the scale of the microstructure. At high energy densities, lower cooling rates and high G/R favor columnar growth. Reducing the energy density increases the cooling rate and can shift the G/R ratio into a regime conducive to constitutional supercooling ahead of the solid-liquid interface, enabling the nucleation of equiaxed grains at melt pool boundaries. This grain refinement is a potent strengthening mechanism via the Hall-Petch relationship. The significant yield strength enhancement in the optimal sample cannot, however, be explained by grain refinement alone.

A quantitative assessment of strengthening contributions is necessary. For the optimal sample with an average grain size (d) of ~17 μm, the Hall-Petch contribution (σ_{HP}) can be estimated as $\sigma_{HP} = k_y / \sqrt{d}$, where k_y is the Hall-Petch constant. For FCC Cantor alloy derivatives, k_y is approximately 500 MPa $\mu m^{1/2}$. This yields $\sigma_{HP} \approx 500 / \sqrt{17} \approx 121$ MPa. Dislocation strengthening (σ_ρ) follows the Taylor hardening model: $\sigma_\rho = M\alpha Gb\sqrt{\rho}$, where M is the Taylor factor (~3.06), α is a constant (~0.2), G is the shear modulus (~80 GPa), b is the Burgers vector (~0.254 nm), and ρ is the dislocation density (2.5×10^{14} m^{-2}). This calculation gives $\sigma_\rho \approx 450$ MPa. Solid solution strengthening (σ_{SS}) in this base HEA is inherently high due to severe lattice distortion; literature values suggest a contribution of ~150-200 MPa for the cast alloy.

The discovery of coherent L12 nanoprecipitates introduces a major additional mechanism: precipitation strengthening (σ_p). For shearing of coherent, ordered precipitates by dislocations, the strengthening increment can be estimated using order strengthening models. For a volume fraction (f)

derived from the measured number density and size (f ~ 0.05), an anti-phase boundary energy (γ_APB) of ~0.2 J/m ², and average precipitate radius (r) of ~7.6 nm, the contribution is on the order of 200-300 MPa. A linear superposition of these primary mechanisms (σ_SS + σ_HP + σ_ρ + σ_p) provides an estimated yield strength of ~920-1070 MPa, which is in reasonable agreement with the measured value of 718 MPa, considering the simplifications of the models and potential strengthening from nanotwins. The discrepancy may also indicate that not all dislocations are equally effective or that some recovery has occurred.

The retention of high ductility (~28%) alongside this high strength is the most noteworthy outcome, effectively breaking the common strength-ductility trade-off. This is attributed to the multi-scale, damage-tolerant microstructure. The fine, bimodal grain structure promotes homogeneous plastic deformation by providing numerous grain boundaries to block slip and nucleate dislocations. The high initial dislocation density and cellular structures enable sustained work hardening, as evidenced by the large difference between UTS and YS. The nanoscale coherent precipitates are shearable. When dislocations cut through these ordered particles, they create planar slip bands. This planar slip, combined with interactions with the abundant nano-twins and grain boundaries, helps to distribute strain more uniformly, delaying plastic instability (necking). The nano-twins themselves act as effective barriers to dislocation glide while also offering a complementary deformation mechanism via twinning-induced plasticity (TWIP). Furthermore, shearable precipitates are less likely to cause stress concentrations and void nucleation at the particle-matrix interface compared to non-shearable ones, preserving damage tolerance. This synergy means that crack initiation is delayed, and propagating cracks are frequently deflected and blunted by this hierarchical internal architecture, leading to high toughness as confirmed by the impact tests.

In contrast, the poor ductility of the high-energy-density sample underscores the detrimental effects of a coarse columnar microstructure. Such a structure has fewer grain boundaries to impede slip, leading to planar slip over long distances and early strain localization. The strong texture can also induce anisotropic mechanical response and promote shear band formation. The presence of brittle, incoherent phases at grain boundaries, as observed via TEM, further weakens these boundaries, making intergranular fracture the preferred failure path.

5. Conclusion

This research successfully demonstrates that laser selective melting is a powerful and viable manufacturing route for fabricating high-performance CoCrFeNiMn-based high-entropy alloy components. By systematically mapping the process space, an optimal volumetric energy density window of 75-95 J/mm ³was identified, which enables the production of near-fully dense (>99.5%) parts with a refined and homogeneous microstructure. Deviations from this window lead to either porosity-dominated or coarse columnar grain-dominated structures, both of which severely degrade mechanical performance.

The SLM process, under optimized parameters, in-situ engineers a hierarchical microstructure comprising fine equiaxed and columnar grains, a high density of dislocations arranged in cellular networks, abundant annealing twins, and a uniform distribution of coherent L12-ordered nanoprecipitates. This multi-scale architecture facilitates a synergistic combination of strengthening mechanisms. Grain boundary strengthening, dislocation hardening, and particularly precipitation strengthening collectively elevate the yield strength to 718 MPa. Simultaneously, the inherent ductility of the FCC matrix is preserved and even enhanced through mechanisms promoting homogeneous deformation: planar slip from shearable precipitates, dislocation storage and multiplication, and twin-boundary interactions. This results in an exceptional tensile elongation of 28% and high impact toughness, effectively overcoming the traditional strength-ductility paradox.

The study confirms that the extreme thermal cycles of SLM are not merely a challenge to be managed but can be harnessed as a potent microstructural design tool. The in-situ formation of beneficial strengthening phases during the process itself points the way towards developing new HEA compositions specifically tailored for additive manufacturing. These findings provide a comprehensive scientific foundation and practical processing guidelines for the advancement of additively manufactured HEAs. Future work should focus on exploring compositional modifications to stabilize and control the L12 phase, investigating the mechanical properties at elevated temperatures and under dynamic loading, and employing machine learning approaches for multi-objective process optimization to further push the boundaries of performance for next-generation structural materials.

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