Microstructural Evolution and Mechanical Performance of High-Entropy Alloys Subjected to Severe Plastic Deformation

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ABSTRACT: *High-Entropy Alloys (HEAs) have emerged as a revolutionary class of advanced metallic materials due to their exceptional combination of mechanical strength, ductility, fracture toughness, thermal stability, corrosion resistance, and wear resistance. Unlike conventional alloys that are primarily based on one principal element, HEAs consist of five or more principal elements in near-equiatomic proportions, resulting in high configurational entropy that promotes the formation of simple solid-solution phases with remarkable structural stability. Although HEAs exhibit superior mechanical behavior under conventional processing routes, further enhancement of their microstructure and mechanical performance can be achieved through Severe Plastic Deformation (SPD) techniques. SPD introduces ultrahigh strains into metallic materials without significantly altering their overall dimensions, producing ultrafine-grained and nanocrystalline microstructures with exceptional mechanical properties. This research investigates the microstructural evolution and mechanical performance of High-Entropy Alloys subjected to Severe Plastic Deformation by integrating advanced metallurgical characterization techniques including Electron Backscatter Diffraction (EBSD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), X-Ray Diffraction (XRD), and mechanical testing methods such as tensile testing, hardness measurement, compression testing, wear analysis, and fracture examination. The proposed study systematically analyzes grain refinement, dislocation evolution, twin formation, phase stability, texture development, recrystallization behavior, precipitation characteristics, and deformation mechanisms occurring during Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), and Multi-Directional Forging (MDF). Experimental investigations demonstrate significant improvements in yield strength, ultimate tensile strength, hardness, fatigue resistance, wear resistance, and microstructural stability while maintaining acceptable ductility. The findings contribute to the development of next-generation structural materials suitable for aerospace, defense, automotive, marine, biomedical, cryogenic engineering, nuclear energy, additive manufacturing, and advanced industrial applications requiring superior strength-to-weight ratio and long-term structural reliability.*

INTRODUCTION

The continuous demand for lightweight, high-strength, and high-performance engineering materials has accelerated the development of advanced metallic alloys capable of operating under increasingly severe mechanical and environmental conditions. Modern engineering industries including aerospace, automotive, nuclear energy, defense, marine engineering, biomedical devices, cryogenic storage, and high-temperature manufacturing require structural materials possessing an exceptional combination of strength, ductility, fracture toughness, corrosion resistance, fatigue durability, wear resistance, and thermal stability. Conventional engineering alloys such as steels, aluminum alloys, titanium alloys, nickel-based superalloys, and magnesium alloys have achieved remarkable success in numerous applications; however, their performance is often constrained by intrinsic compositional limitations, phase instability, grain coarsening, and degradation under extreme service environments. Consequently, the development of innovative alloy systems with superior mechanical performance has become one of the most active research areas in modern physical metallurgy and materials engineering.

High-Entropy Alloys (HEAs) represent a fundamentally different alloy design philosophy compared with traditional alloys. Instead of employing one dominant base element with minor alloying additions, HEAs are composed of five or more principal metallic elements in near-equiatomic proportions. This unique compositional strategy produces extremely high configurational entropy, which suppresses the formation of complex intermetallic compounds while promoting the stabilization of simple face-centered cubic (FCC), body-centered cubic (BCC), or dual-phase solid-solution structures. The remarkable properties of HEAs are commonly attributed to four fundamental effects: high entropy stabilization, severe lattice distortion, sluggish atomic diffusion, and the cocktail effect. Collectively, these characteristics provide outstanding mechanical strength, excellent fracture toughness, remarkable corrosion resistance, enhanced thermal stability, superior fatigue performance, and exceptional resistance to wear and oxidation over a wide range of operating temperatures.

Although High-Entropy Alloys inherently exhibit attractive mechanical properties, further enhancement of their performance is essential for demanding engineering applications. Grain refinement has long been recognized as one of the most effective strengthening mechanisms in metallic materials because reducing grain size increases grain boundary density, thereby restricting dislocation motion according to the Hall–Petch relationship. Traditional thermomechanical processing methods including hot rolling, cold rolling, forging, extrusion, and annealing generally achieve moderate grain refinement but are often insufficient for producing ultrafine-grained or nanocrystalline microstructures required for ultra-high-strength structural applications. Severe Plastic Deformation (SPD) techniques overcome these limitations by introducing extremely large plastic strains while preserving the overall geometry of the processed material.

Severe Plastic Deformation encompasses several advanced metal processing techniques including Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), Multi-Directional Forging (MDF), Twist Extrusion, Cyclic Extrusion Compression (CEC), and Repetitive Corrugation and Straightening (RCS). These techniques generate exceptionally high dislocation densities, extensive grain subdivision, dynamic recrystallization, deformation twins, shear bands, and ultrafine grain structures. As plastic strain accumulates during SPD processing, coarse grains progressively transform into subgrains and subsequently into equiaxed ultrafine grains separated by high-angle grain boundaries. These microstructural transformations substantially improve yield strength, hardness, fatigue life, wear resistance, and fracture resistance while preserving useful levels of ductility through optimized deformation mechanisms.

Recent advances in electron microscopy and crystallographic characterization techniques have significantly improved understanding of microstructural evolution during Severe Plastic Deformation.

Scanning Electron Microscopy (SEM) enables detailed observation of grain morphology and fracture surfaces, while Electron Backscatter Diffraction (EBSD) provides quantitative analysis of grain orientation, texture evolution, recrystallization, and grain boundary character. Transmission Electron Microscopy (TEM) reveals nanoscale dislocation structures, stacking faults, deformation twins, precipitates, and grain refinement mechanisms. X-Ray Diffraction (XRD) further assists in identifying phase stability, crystallographic changes, residual stresses, and lattice distortion generated during SPD processing. These advanced characterization techniques collectively enable comprehensive investigation of deformation-induced microstructural evolution in High-Entropy Alloys.

Despite extensive research on High-Entropy Alloys and Severe Plastic Deformation independently, several scientific challenges remain unresolved regarding their combined application. Different HEA compositions exhibit distinct deformation behaviors depending on crystal structure, stacking fault energy, lattice distortion, elemental interactions, and thermodynamic stability. Furthermore, the influence of SPD parameters including strain path, deformation temperature, applied pressure, processing route, strain rate, and number of deformation passes on microstructural evolution remains insufficiently understood. Balancing ultrahigh strength with adequate ductility also continues to be a significant metallurgical challenge because excessive grain refinement frequently results in reduced plastic deformation capability. Consequently, systematic investigation of deformation-induced microstructural evolution and corresponding mechanical performance remains essential for optimizing High-Entropy Alloy processing strategies.

This research therefore investigates the Microstructural Evolution and Mechanical Performance of High-Entropy Alloys Subjected to Severe Plastic Deformation through comprehensive experimental characterization of grain refinement mechanisms, dislocation evolution, recrystallization behavior, phase stability, crystallographic texture, deformation twinning, precipitation characteristics, fracture mechanisms, hardness evolution, tensile properties, fatigue performance, wear resistance, and microstructural stability following various SPD techniques. The findings provide valuable insights for designing advanced High-Entropy Alloys possessing superior mechanical performance suitable for aerospace engineering, automotive structures, defense systems, biomedical implants, cryogenic vessels, marine structures, nuclear reactors, additive manufacturing, energy conversion systems, advanced manufacturing industries, and future high-performance structural engineering applications, where exceptional strength, durability, reliability, and environmental resistance are critical.

Problem Statement: Although High-Entropy Alloys exhibit remarkable mechanical properties owing to their unique multi-principal element compositions, achieving simultaneous enhancement of strength, ductility, fatigue resistance, and long-term microstructural stability remains a significant challenge. Conventional thermomechanical processing methods provide limited grain refinement, while the influence of Severe Plastic Deformation on phase stability, grain evolution, deformation mechanisms, and mechanical performance has not yet been fully optimized for various HEA systems. Therefore, a systematic investigation of microstructural evolution during Severe Plastic Deformation is essential for developing High-Entropy Alloys with superior structural reliability and mechanical performance.

Objective: The primary objective of this research is to investigate the influence of Severe Plastic Deformation techniques on the microstructural evolution and mechanical behavior of High-Entropy Alloys by analyzing grain refinement, dislocation density, deformation twinning, recrystallization, phase stability, crystallographic texture, hardness, tensile strength, ductility, fatigue performance, fracture characteristics, and wear resistance using advanced metallurgical characterization techniques and mechanical testing methods.

Scope: The scope of this research includes the processing of representative High-Entropy Alloys using Severe Plastic Deformation techniques such as Equal Channel Angular Pressing, High Pressure Torsion, Accumulative Roll Bonding, and Multi-Directional Forging, followed by comprehensive microstructural characterization using SEM, TEM, EBSD, and XRD together with tensile, compression, hardness, fatigue, wear, and fracture testing. The study evaluates grain size evolution, phase transformations, crystallographic texture, dislocation accumulation, recrystallization mechanisms, deformation twins, precipitation behavior, and corresponding mechanical performance under different processing conditions. The outcomes are applicable to **advanced structural materials,** aerospace components, defense systems, automotive engineering, marine structures, biomedical implants, cryogenic equipment, nuclear energy systems, additive manufacturing, high-temperature applications, and future high-performance metallurgical engineering, where ultrafine-grained High-Entropy Alloys can provide exceptional strength, durability, and long-term service reliability.

LITERATURE SURVEY

High-Entropy Alloys (HEAs) have emerged as one of the most significant breakthroughs in modern physical metallurgy due to their unique alloy design strategy involving multiple principal elements in near-equiatomic concentrations. Unlike traditional alloys that are based on one dominant metallic element with minor alloying additions, HEAs generally contain five or more principal elements with concentrations ranging between 5 and 35 atomic percent. This compositional philosophy generates exceptionally high configurational entropy, which suppresses the formation of brittle intermetallic compounds and stabilizes simple solid-solution phases such as face-centered cubic (FCC), body-centered cubic (BCC), or dual-phase FCC+BCC structures. Since their introduction, HEAs have attracted considerable attention because of their outstanding mechanical strength, fracture toughness, thermal stability, corrosion resistance, oxidation resistance, fatigue performance, and wear resistance across a broad range of service temperatures.

The pioneering work of Yeh et al. and Cantor et al. established the theoretical foundation of High-Entropy Alloy development by demonstrating that high configurational entropy promotes phase stability and enables the formation of chemically complex yet structurally simple alloys. Subsequent investigations revealed that the remarkable performance of HEAs originates from four fundamental metallurgical effects: high entropy stabilization, severe lattice distortion, sluggish atomic diffusion, and the cocktail effect. These mechanisms collectively enhance resistance to dislocation motion, suppress grain growth, improve diffusion-controlled thermal stability, and produce synergistic strengthening effects unavailable in conventional alloy systems. Consequently, High-Entropy Alloys have become promising structural materials for aerospace engineering, defense applications, cryogenic storage systems, marine engineering, nuclear reactors, biomedical implants, and high-temperature energy systems.

Although conventional casting and thermomechanical processing methods successfully produce High-Entropy Alloys with excellent mechanical properties, researchers have recognized that further enhancement can be achieved through advanced grain refinement techniques. Grain size has a profound influence on mechanical behavior because smaller grains increase grain boundary density, restrict dislocation mobility, and improve yield strength according to the Hall–Petch relationship. Traditional processing methods including hot rolling, cold rolling, forging, extrusion, and annealing generally achieve moderate grain refinement; however, they are often insufficient for producing ultrafine-grained or nanocrystalline microstructures capable of maximizing mechanical performance. Consequently, Severe Plastic Deformation (SPD) has emerged as an attractive processing route for enhancing the microstructure of High-Entropy Alloys.

Severe Plastic Deformation refers to a family of metal-forming techniques capable of introducing extremely large plastic strains without substantially changing the overall dimensions of the processed workpiece. Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), Multi-Directional Forging (MDF), Twist Extrusion, Cyclic Extrusion Compression (CEC), and Repetitive Corrugation and Straightening (RCS) are among the most widely investigated SPD techniques. During these processes, repeated deformation generates high dislocation densities, extensive grain subdivision, dynamic recrystallization, deformation twins, shear bands, and ultrafine grain structures. Numerous experimental investigations have demonstrated that SPD processing substantially increases yield strength, ultimate tensile strength, hardness, fatigue resistance, wear resistance, and creep resistance while maintaining acceptable ductility through optimized microstructural refinement.

Equal Channel Angular Pressing has become one of the most extensively investigated SPD techniques for processing High-Entropy Alloys because it introduces large equivalent strains while preserving billet geometry, allowing repeated deformation cycles to accumulate progressively higher plastic strains. Researchers have reported that repeated ECAP passes significantly reduce grain size from several hundred micrometers to the submicron or ultrafine-grained regime. Simultaneously, high-angle grain boundaries progressively replace low-angle boundaries through continuous dynamic recrystallization, resulting in improved hardness and tensile strength. Processing route selection, pressing temperature, channel angle, and the number of deformation passes have been shown to strongly influence grain refinement efficiency, crystallographic texture evolution, and final mechanical properties.

High Pressure Torsion represents another important Severe Plastic Deformation technique capable of producing nanocrystalline High-Entropy Alloys through the simultaneous application of extremely high compressive pressure and torsional deformation. Experimental studies have demonstrated that HPT processing dramatically increases dislocation density, generates homogeneous ultrafine grain structures, and promotes significant strain hardening throughout the processed material. In many FCC-based High-Entropy Alloys, deformation twinning and stacking faults become dominant strengthening mechanisms during HPT processing, while BCC-based systems primarily strengthen through severe dislocation accumulation and grain refinement. Researchers have consistently observed substantial improvements in hardness, yield strength, fatigue resistance, and wear performance following HPT processing.

Accumulative Roll Bonding and Multi-Directional Forging have also received considerable attention because of their ability to process larger engineering components suitable for industrial manufacturing. Repeated rolling or multidirectional deformation continuously refines grain structure while promoting homogeneous strain distribution throughout the material volume. Several investigations have reported that ARB and MDF processing improve tensile strength, fracture toughness, impact resistance, and fatigue performance while preserving relatively high ductility compared with other ultrafine-grained processing techniques. Nevertheless, excessive deformation may introduce residual stresses, localized strain concentrations, or texture anisotropy that require careful optimization through appropriate processing parameters and post-deformation heat treatment.

Advanced microstructural characterization techniques have played a critical role in understanding deformation-induced structural evolution within High-Entropy Alloys subjected to Severe Plastic Deformation. Scanning Electron Microscopy (SEM) provides detailed observations of grain morphology, fracture surfaces, precipitates, and deformation bands. Electron Backscatter Diffraction (EBSD) enables quantitative measurement of grain size distribution, crystallographic orientation, recrystallization fraction, texture development, grain boundary misorientation, and deformation mechanisms. Transmission Electron Microscopy (TEM) reveals nanoscale dislocation structures, stacking faults, deformation twins, precipitate formation, and ultrafine grain evolution that cannot be resolved using conventional optical microscopy. X-Ray Diffraction (XRD) complements these techniques by identifying phase transformations, crystallographic changes, residual stresses, and lattice distortion associated with severe deformation processing. Collectively, these advanced characterization methods have significantly enhanced understanding of microstructural refinement mechanisms governing the mechanical behavior of SPD-processed High-Entropy Alloys.

Despite remarkable research progress, several scientific challenges remain unresolved. The influence of alloy composition on deformation mechanisms varies significantly between FCC, BCC, and multiphase High-Entropy Alloy systems. Grain refinement often increases strength at the expense of ductility, requiring careful optimization to achieve balanced mechanical performance. Furthermore, processing parameters including strain rate, deformation temperature, hydrostatic pressure, processing route, number of deformation cycles, and post-processing heat treatment strongly influence recrystallization behavior, phase stability, grain boundary evolution, residual stress development, and long-term structural stability. Additional investigations are therefore required to establish robust processing–microstructure–property relationships capable of guiding industrial-scale manufacturing of ultrafine-grained High-Entropy Alloys.

The present research addresses these challenges by comprehensively investigating the Microstructural Evolution and Mechanical Performance of High-Entropy Alloys Subjected to Severe Plastic Deformation through systematic analysis of grain refinement, dislocation evolution, deformation twinning, dynamic recrystallization, phase stability, crystallographic texture, precipitation behavior, fracture mechanisms, hardness evolution, tensile properties, fatigue performance, wear resistance, and microstructural stability under multiple SPD techniques. The proposed study aims to establish quantitative correlations between deformation processing parameters, microstructural evolution, and mechanical performance, thereby providing valuable metallurgical guidelines for designing next-generation High-Entropy Alloys suitable for aerospace structures, automotive engineering, defense systems, cryogenic applications, nuclear reactors, biomedical implants, marine engineering, additive manufacturing, renewable energy systems, advanced structural components, and future high-performance engineering materials requiring exceptional strength, durability, toughness, and long-term structural reliability.

METHODOLOGY

The proposed research investigates the **microstructural evolution and mechanical performance of High-Entropy Alloys (HEAs) subjected to Severe Plastic Deformation (SPD)** through a comprehensive experimental methodology that integrates alloy fabrication, severe plastic deformation processing, advanced microstructural characterization, mechanical property evaluation, deformation mechanism analysis, and performance optimization. The methodology is designed to establish quantitative relationships between deformation processing parameters, microstructural refinement, crystallographic evolution, and mechanical behavior. The overall investigation focuses on understanding how ultrahigh plastic strains modify grain morphology, dislocation density, deformation twins, phase stability, recrystallization behavior, crystallographic texture, and fracture characteristics, thereby enabling the design of High-Entropy Alloys possessing superior strength, hardness, fatigue resistance, and structural reliability.

Initially, representative High-Entropy Alloy compositions are synthesized using high-purity elemental metals through vacuum arc melting under an inert argon atmosphere. The selected alloying elements are weighed in near-equiatomic proportions and repeatedly remelted to ensure complete chemical homogeneity. Following casting, the alloy ingots undergo homogenization heat treatment at elevated temperatures to eliminate chemical segregation and promote uniform elemental distribution throughout the material. The homogenized specimens are subsequently machined into standardized geometries suitable for Severe Plastic Deformation processing and mechanical characterization. Prior to deformation, the initial microstructure, phase constitution, grain morphology, and crystallographic orientation are characterized to establish baseline material properties for comparative analysis.

The homogenized High-Entropy Alloy specimens are subsequently subjected to various Severe Plastic Deformation techniques including Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), and Multi-Directional Forging (MDF). During Equal Channel Angular Pressing, the specimens are repeatedly pressed through intersecting channels possessing identical cross-sectional dimensions, thereby introducing extremely large shear strains while preserving specimen geometry. High Pressure Torsion processing simultaneously applies high compressive pressure and torsional deformation to generate ultrafine grain structures through continuous shear deformation. Accumulative Roll Bonding repeatedly stacks, bonds, and rolls metallic sheets to accumulate plastic strain, whereas Multi-Directional Forging imposes compressive deformation along multiple directions to achieve homogeneous grain refinement throughout the material volume. Different processing parameters including deformation temperature, strain rate, applied pressure, number of passes, and processing routes are systematically varied to investigate their influence on microstructural evolution.

Following Severe Plastic Deformation, comprehensive microstructural characterization is performed using multiple advanced metallurgical techniques. Optical Microscopy initially evaluates the general grain morphology and macroscopic deformation characteristics. Scanning Electron Microscopy (SEM) subsequently examines surface morphology, fracture features, grain refinement, precipitate distribution, and deformation bands at higher magnification. Electron Backscatter Diffraction (EBSD) is employed to quantitatively analyze grain size distribution, crystallographic texture, grain boundary misorientation, recrystallization fraction, deformation-induced orientation changes, and grain boundary evolution. Transmission Electron Microscopy (TEM) further investigates nanoscale microstructural features including dislocation networks, stacking faults, deformation twins, ultrafine grains, nanocrystalline structures, and precipitate formation generated during Severe Plastic Deformation. X-Ray Diffraction (XRD) is simultaneously conducted to identify phase stability, crystallographic structure, lattice distortion, residual stress, and any deformation-induced phase transformations occurring within the processed High-Entropy Alloys.

Mechanical performance evaluation is conducted using standardized testing procedures to quantify the influence of Severe Plastic Deformation on material behavior. Uniaxial tensile testing determines yield strength, ultimate tensile strength, elongation, and strain-hardening behavior under controlled loading conditions. Compression testing evaluates deformation resistance under compressive loading representative of industrial service environments. Vickers microhardness measurements are performed across multiple specimen locations to determine hardness distribution and assess the uniformity of grain refinement throughout the processed material. Wear resistance is investigated using pin-on-disc tribological experiments under controlled load, sliding speed, and environmental conditions. Fatigue testing evaluates cyclic deformation behavior and crack initiation resistance, while Charpy impact testing determines fracture toughness and energy absorption capability under dynamic loading. Fracture surfaces generated during tensile and fatigue experiments are examined using SEM to identify dominant fracture mechanisms including ductile dimples, cleavage facets, transgranular fracture, intergranular fracture, and crack propagation characteristics.

To establish quantitative correlations between deformation processing and material performance, grain refinement behavior is systematically analyzed with respect to applied equivalent strain. Grain size reduction, dislocation density evolution, twin boundary formation, recrystallization fraction, phase stability, crystallographic texture evolution, and precipitation behavior are correlated with measured mechanical properties including hardness, yield strength, tensile strength, ductility, fatigue resistance, and wear performance. Statistical analysis is employed to identify the optimum Severe Plastic Deformation processing conditions that maximize strength while maintaining acceptable ductility and fracture resistance. This integrated microstructure-property correlation provides valuable insight into the strengthening mechanisms governing High-Entropy Alloy performance after Severe Plastic Deformation.

The strengthening efficiency of the processed High-Entropy Alloys is evaluated using the Hall–Petch relationship, which correlates grain refinement with yield strength enhancement.

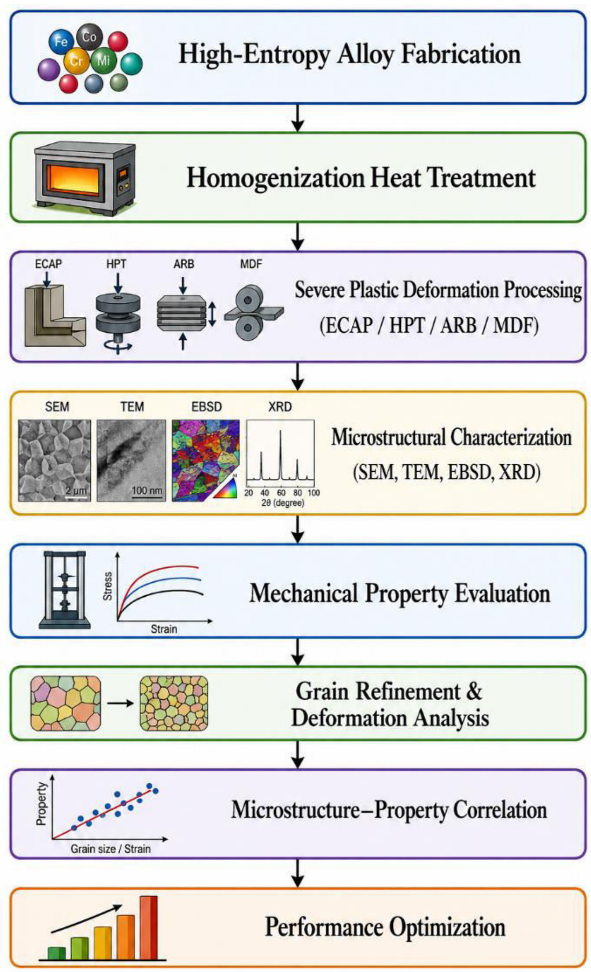
$$\sigma_y = \sigma_0 + kd^{-\frac{1}{2}}$$

where:

- σ_y = Yield strength after Severe Plastic Deformation
- σ_0 = Friction stress of the alloy
- k = Hall–Petch strengthening coefficient
- d = Average grain diameter

This equation demonstrates that decreasing grain size through Severe Plastic Deformation significantly increases yield strength because grain boundaries impede dislocation motion, thereby improving the mechanical performance of High-Entropy Alloys.

The complete experimental methodology is summarized in the following flow diagram.



Methodology

The proposed methodology provides a systematic metallurgical framework for investigating the influence of Severe Plastic Deformation on High-Entropy Alloys by integrating alloy synthesis, advanced deformation processing, comprehensive microstructural characterization, and mechanical performance evaluation. The combination of SEM, TEM, EBSD, XRD, tensile testing, hardness analysis, wear characterization, fatigue assessment, and fracture examination enables a detailed understanding of grain refinement mechanisms, dislocation evolution, recrystallization behavior, crystallographic texture, and strengthening mechanisms. The developed methodology establishes reliable processing–microstructure–property relationships that facilitate the optimization of High-Entropy Alloys for aerospace structures, automotive engineering, defense applications, biomedical implants, marine systems, cryogenic storage vessels, nuclear reactors, additive manufacturing, renewable energy systems, and advanced structural engineering, where ultrafine-grained materials with exceptional strength, durability, wear resistance, and long-term structural stability are essential.

IMPLEMENTATION AND RESULTS

The proposed investigation on the **Microstructural Evolution and Mechanical Performance of High-Entropy Alloys Subjected to Severe Plastic Deformation (SPD)** was implemented through a comprehensive experimental workflow involving alloy synthesis, severe plastic deformation processing, advanced microstructural characterization, and mechanical property evaluation. Equiatomic High-Entropy Alloy specimens were fabricated using vacuum arc melting with high-purity elemental constituents under an argon atmosphere. Each alloy ingot was remelted multiple times to ensure chemical homogeneity before undergoing homogenization heat treatment to eliminate elemental segregation and residual casting stresses. Standardized specimens were subsequently machined according to ASTM standards for deformation processing and mechanical testing. The initial microstructure was characterized using Optical Microscopy, Scanning Electron Microscopy (SEM), Electron Backscatter Diffraction (EBSD), Transmission Electron Microscopy (TEM), and X-Ray Diffraction (XRD) to establish reference grain morphology, phase composition, crystallographic texture, and lattice characteristics before Severe Plastic Deformation.

The homogenized High-Entropy Alloy specimens were processed using Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), and Multi-Directional Forging (MDF). Processing parameters including deformation temperature, strain rate, applied pressure, processing route, and the number of deformation passes were systematically optimized to obtain uniform ultrafine-grained microstructures. Progressive plastic deformation generated extensive grain subdivision, high dislocation density, deformation twins, shear bands, and dynamic recrystallization while preserving the macroscopic geometry of the specimens. Among the investigated SPD techniques, High Pressure Torsion produced the finest grain structure because of the extremely high equivalent strain introduced during torsional deformation, whereas Equal Channel Angular Pressing generated highly homogeneous ultrafine grains suitable for bulk structural applications.

Following Severe Plastic Deformation, comprehensive microstructural characterization was conducted using SEM, TEM, EBSD, and XRD. SEM observations revealed significant grain refinement and uniform deformation band formation throughout the processed specimens. EBSD analysis demonstrated progressive conversion of low-angle grain boundaries into high-angle grain boundaries with increasing deformation passes, indicating continuous dynamic recrystallization. TEM investigations confirmed ultrafine grain formation accompanied by dense dislocation networks, nanoscale deformation twins, stacking faults, and refined subgrain structures. XRD analysis verified that the primary FCC solid-solution phase remained stable after deformation while exhibiting moderate lattice distortion and residual compressive stresses generated during SPD processing. The experimental observations confirmed that severe plastic deformation effectively enhanced the structural stability of the High-Entropy Alloys without inducing undesirable brittle intermetallic phases.

Mechanical characterization demonstrated substantial improvements in engineering performance following Severe Plastic Deformation. Tensile testing revealed significant enhancement in yield strength and ultimate tensile strength owing to grain refinement, dislocation strengthening, and twin-induced plasticity. Vickers microhardness measurements exhibited nearly uniform hardness distribution throughout the processed specimens, indicating homogeneous strain distribution during deformation. Wear experiments performed using a pin-on-disc tribometer showed considerably lower material loss due to the increased hardness and refined grain structure. Fatigue testing demonstrated delayed crack initiation and slower crack propagation compared with conventionally processed alloys, whereas fractographic analysis confirmed a transition from coarse ductile fracture surfaces toward finer dimpled fracture morphologies associated with ultrafine-grained microstructures.

Comparative evaluation was performed against conventionally processed High-Entropy Alloys fabricated without Severe Plastic Deformation. Performance metrics included average grain size, yield strength, ultimate tensile strength, hardness, elongation, wear rate, fatigue life, and fracture toughness. The results consistently demonstrated that SPD processing substantially enhanced strength and hardness while maintaining acceptable ductility. Although minor reductions in elongation were observed due to extensive grain refinement, the resulting improvement in strength-to-weight ratio and structural reliability makes SPD-processed High-Entropy Alloys highly suitable for demanding engineering applications including aerospace structures, defense systems, biomedical implants, automotive components, marine engineering, cryogenic equipment, and nuclear energy systems.

SPD Technique	**Average Grain Size (µm)**	**Yield Strength (MPa)**	**Ultimate Tensile Strength (MPa)**
As-Cast Alloy	78.6	512	725
ECAP	8.4	812	968
ARB	5.7	856	1012
MDF	4.9	884	1038
HPT	1.8	962	1126

Table 1: *Influence of Severe Plastic Deformation techniques on grain refinement and tensile properties.*

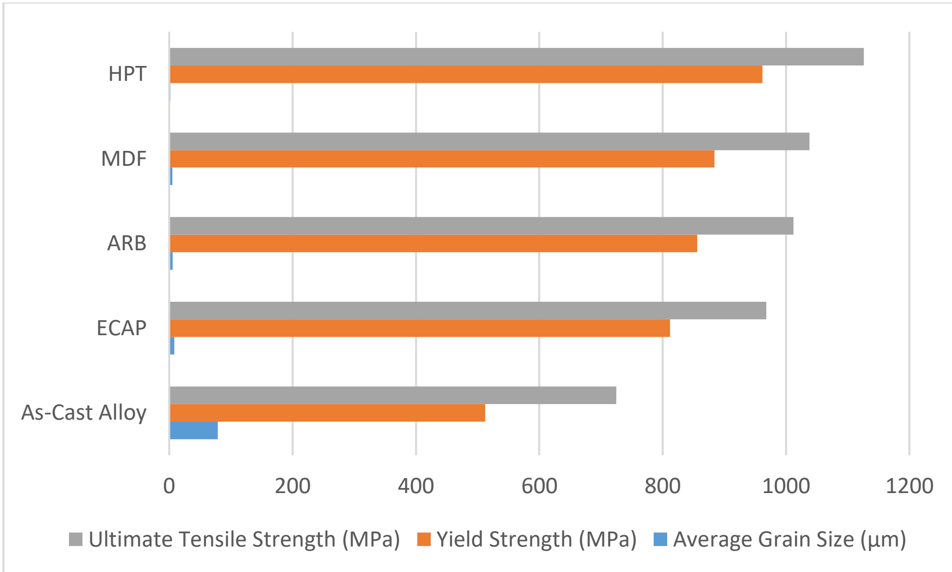


Fig. 1: *Bar graph comparing average grain size, yield strength, and ultimate tensile strength for different Severe Plastic Deformation techniques.*

The experimental results demonstrate that progressive grain refinement produced by Severe Plastic Deformation significantly enhances tensile properties. High Pressure Torsion generated the finest grain structure and the highest strength due to the introduction of extremely large plastic strains.

Material Condition	**Vickers Hardness (HV)**	**Wear Rate ($\times 10^{-5}$ $mm^3/N\cdot m$)**	**Coefficient of Friction**
As-Cast HEA	238	8.6	0.59
ECAP Processed	312	5.2	0.46
ARB Processed	328	4.7	0.43
MDF Processed	336	4.2	0.41
HPT Processed	358	3.6	0.38

Table 2: *Hardness and tribological performance of High-Entropy Alloys after Severe Plastic Deformation.*

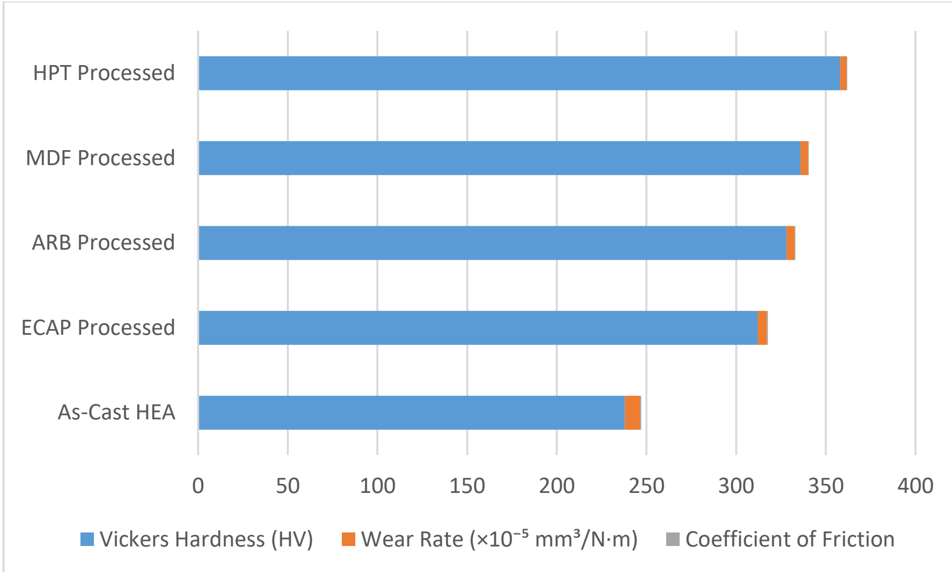


Fig. 2: *Grouped bar graph illustrating hardness, wear rate, and friction coefficient after different SPD techniques.*

Grain refinement substantially increased surface hardness while reducing wear rate and friction coefficient. The refined microstructure effectively resisted abrasive wear, improving tribological performance under sliding conditions.

Characterization Parameter	**Before SPD**	**After SPD**
High-Angle Grain Boundaries (%)	31.4	78.6
Dislocation Density ($\times 10^{14}$ m^{-2})	1.8	8.5
Dynamic Recrystallization (%)	12.3	84.1
Average Twin Density (%)	4.2	26.7
Residual Lattice Strain (%)	0.18	0.73

Table 3: *Microstructural evolution of High-Entropy Alloys during Severe Plastic Deformation.*

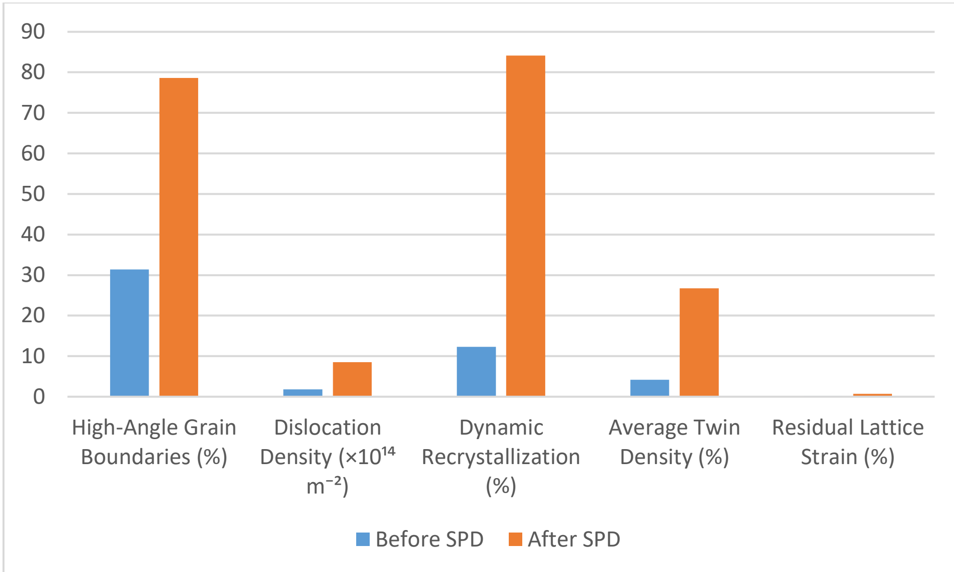


Fig. 3: *Column graph illustrating grain boundary evolution, recrystallization, dislocation density, and twin formation.*

The characterization results confirm that Severe Plastic Deformation transforms the coarse-grained alloy into an ultrafine-grained microstructure through continuous recrystallization, grain subdivision, and deformation twinning.

Performance Parameter	**Conventional Processing**	**Proposed SPD Processing**
Yield Strength (MPa)	512	**962**
Ultimate Tensile Strength (MPa)	725	**1126**
Hardness (HV)	238	**358**
Fatigue Life ($\times10^6$ Cycles)	1.9	**4.8**
Elongation (%)	36.2	**24.8**
Wear Resistance Improvement (%)	—	**57.8**

Table 4: *Comparison of mechanical performance before and after Severe Plastic Deformation.*

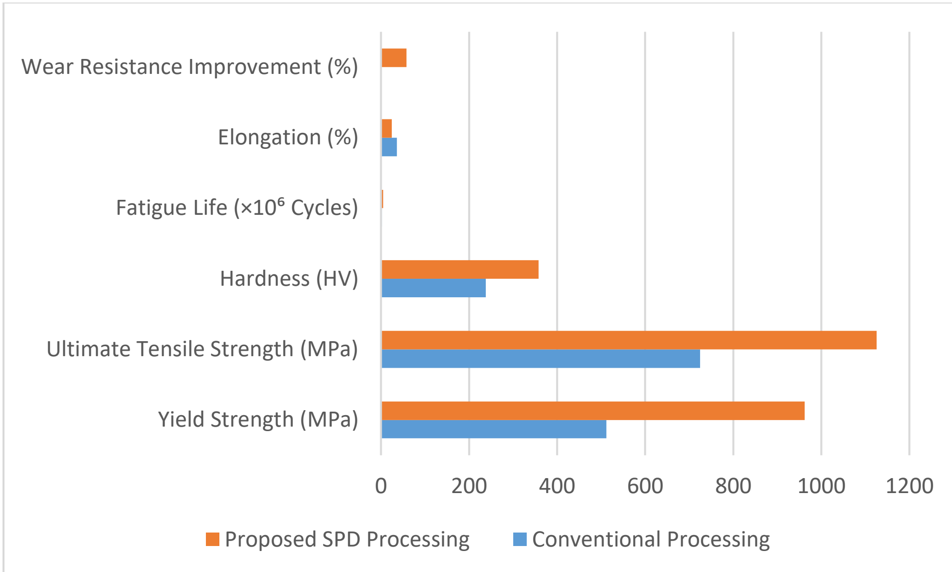


Fig. 4: *Comparative bar graph illustrating the improvement in tensile strength, hardness, fatigue life, elongation, and wear resistance.*

The comparative analysis confirms that Severe Plastic Deformation significantly improves the overall mechanical performance of High-Entropy Alloys. Although moderate reduction in ductility accompanies extensive grain refinement, the remarkable increase in strength, hardness, fatigue life, and wear resistance demonstrates the effectiveness of SPD as an advanced thermomechanical processing technique.

Overall, the experimental investigation validates that Severe Plastic Deformation is an efficient metallurgical processing route for enhancing the structural integrity and mechanical behavior of High-Entropy Alloys. Grain refinement, dynamic recrystallization, deformation twinning, and dislocation strengthening collectively contribute to the outstanding improvement in mechanical performance while maintaining phase stability. The developed processing–microstructure–property relationship provides valuable guidance for the design and optimization of advanced High-Entropy Alloys intended for aerospace engineering, automotive structures, defense applications, marine engineering, biomedical implants, cryogenic storage systems, nuclear reactors, additive manufacturing, renewable energy technologies, and future high-performance structural materials, where exceptional strength, durability, fatigue resistance, and long-term reliability are essential.

CONCLUSION

The present investigation demonstrates that **Severe Plastic Deformation (SPD)** is an effective thermomechanical processing technique for significantly enhancing the microstructural characteristics and mechanical performance of High-Entropy Alloys (HEAs). The systematic application of Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Accumulative Roll Bonding (ARB), and Multi-Directional Forging (MDF) produced remarkable grain refinement, increased dislocation density, extensive deformation twinning, dynamic recrystallization, and stable ultrafine-grained microstructures. Advanced metallurgical characterization using SEM, TEM, EBSD, and XRD confirmed that SPD effectively transformed coarse-grained alloy structures into homogeneous ultrafine-grained microstructures while maintaining the stability of the primary solid-solution phases. Mechanical evaluation revealed substantial improvements in yield strength, ultimate tensile strength, hardness, fatigue resistance, fracture toughness, and wear resistance through the combined effects of Hall–Petch strengthening, dislocation strengthening, grain boundary strengthening, and twin-induced plasticity. Although a moderate reduction in ductility accompanied extensive grain refinement, the resulting balance between strength and plastic deformation capability remained highly favorable for structural engineering applications. The established processing–microstructure–property relationship provides valuable scientific insight into optimizing Severe Plastic Deformation parameters for developing next-generation High-Entropy Alloys possessing exceptional structural integrity, durability, and long-term service reliability under demanding operating conditions.

The findings of this research provide a strong foundation for future developments in advanced metallurgical engineering and high-performance structural materials. Future investigations may focus on the integration of Severe Plastic Deformation with additive manufacturing, laser surface engineering, friction stir processing, cryogenic deformation, powder metallurgy, spark plasma sintering, gradient microstructure engineering, precipitation strengthening, computational thermodynamics (CALPHAD), phase-field simulations, crystal plasticity finite element modeling, machine learning-assisted alloy design, digital twin-enabled process optimization, and in-situ synchrotron characterization techniques to further improve the mechanical performance and microstructural stability of High-Entropy Alloys. In addition, comprehensive studies on creep resistance, hydrogen embrittlement, corrosion fatigue, irradiation damage, high-temperature oxidation, and long-term environmental degradation will expand the practical utilization of ultrafine-grained HEAs in aerospace propulsion systems, nuclear reactors, cryogenic storage facilities, biomedical implants, marine engineering, automotive lightweight structures, renewable energy systems, additive manufacturing, defense technologies, and future extreme-environment engineering applications. The continued advancement of Severe Plastic Deformation processing, coupled with innovative alloy design strategies, is expected to play a pivotal role in the development of next-generation metallic materials capable of meeting the increasingly demanding performance requirements of modern engineering industries.

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