

## **Modern Strengthening Approaches for Aluminum Alloys: From Precipitation to Ultrafine-Grained Structures**

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**Abstract:** The rapid evolution of transportation, energy, and aerospace technologies continues to increase the demand for aluminum alloys with superior combinations of strength, ductility, and thermal stability. Meeting these requirements relies on a deep understanding of the microstructural mechanisms governing deformation and strengthening. Over the last decade, substantial advancements have been achieved in the development of multiscale strengthening strategies, where atomic-level solute interactions, nanoscale precipitates, and submicron grain structures are engineered synergistically to optimize mechanical performance. This review consolidates recent experimental and theoretical progress in precipitation engineering, solute–dislocation interactions, and grain refinement through SPD. A comparative assessment of strengthening efficiency, stability under service temperatures, and applicability to industrial-scale processing is provided. The article also outlines remaining challenges and identifies promising routes toward designing next-generation aluminum alloys optimized for lightweight, high-performance structural applications.

**Keywords:** Aluminum alloys, strengthening mechanisms, precipitation hardening, age-hardening treatments, ultrafine-grained (UFG) structures.

### **Introduction**

Aluminum alloys remain among the most strategically important metallic materials for modern engineering applications due to their exceptional combination of low density, high specific strength, excellent corrosion resistance, and technological versatility. Over the past decades, the demand for lightweight structural materials has intensified markedly, driven by stringent energy-efficiency, environmental, and performance requirements in aerospace, automotive, railway, and advanced manufacturing sectors. To satisfy these demands, researchers and industry have focused increasingly on optimizing the mechanical behavior of aluminum alloys through advanced strengthening strategies that go beyond conventional alloy design. As a result, understanding and controlling the microstructural mechanisms governing strength, ductility, fatigue resistance, and thermal stability has become a central priority in materials science and engineering.

Traditionally, the primary strengthening route for heat-treatable aluminum alloys has been precipitation hardening, which enables a dramatic increase in strength following controlled solution treatment and artificial aging. The formation of coherent or semi-coherent metastable precipitates—such as GP zones,  $\theta'$  in Al–Cu,  $\beta''$  in Al–Mg–Si, and  $\eta'$  in Al–Zn–Mg alloys—effectively hinders dislocation motion, thereby delivering a high strength–weight ratio. Despite the maturity of this approach, recent studies have shown that optimizing nanoscale precipitate morphology, reducing precipitate-free zones, and tailoring the interaction between solute atoms

and dislocations provide new opportunities for further improvement. Meanwhile, for non-heat-treatable alloy families such as Al–Mg and Al–Mn, solid-solution strengthening and dispersoid formation remain critical to enhancing performance under extreme conditions.

However, contemporary engineering challenges increasingly require materials with strength levels beyond those attainable by precipitation hardening alone. This has stimulated substantial interest in microstructural refinement via severe plastic deformation (SPD) techniques, including equal-channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll bonding (ARB), and multidirectional forging (MDF). These methods make it possible to produce ultrafine-grained and even nanocrystalline aluminum with grain sizes below 1  $\mu\text{m}$ , where grain boundary strengthening and increased dislocation density lead to extraordinary strength levels while maintaining satisfactory ductility. Moreover, the combination of SPD with subsequent heat treatment has opened new avenues for hierarchical microstructures containing nanoscale precipitates embedded within ultrafine grains, yielding synergistic strengthening effects not accessible by either approach alone.

At the same time, the study of microstructural evolution during thermomechanical processing is rapidly advancing due to progress in high-resolution characterization methods, such as TEM, EBSD, APT, and in situ synchrotron techniques. These tools have provided unprecedented insights into solute–dislocation interactions, nucleation and growth of precipitates, and dynamic recovery and recrystallization during SPD. In parallel, computational materials science—particularly molecular dynamics (MD), density functional theory (DFT), and phase-field modeling—has enabled predictive design of aluminum alloys with tailored precipitation sequences and grain structures. Together, these experimental and computational advances have contributed to a new generation of aluminum alloys with highly optimized performance.

Despite significant breakthroughs, critical knowledge gaps remain. Challenges include maintaining thermal stability of ultrafine-grained structures at elevated temperatures, controlling heterogeneous precipitation in complex alloy systems, optimizing fatigue resistance, and developing cost-effective large-scale processing routes suitable for industry. Therefore, a comprehensive understanding of the interplay between precipitation phenomena, grain refinement mechanisms, dislocation behavior, and solute distribution is essential for further progress.

This article provides a systematic review and comparative analysis of modern strengthening approaches for aluminum alloys, ranging from classical precipitation hardening to the most recent strategies for producing ultrafine-grained and nanostructured aluminum. Special attention is devoted to microstructural mechanisms, processing–structure–property relationships, and the integration of multiple strengthening pathways to achieve superior mechanical performance. Experimental data, comparative tables, and schematic microstructural illustrations are presented to highlight current advances and identify future research directions relevant to both academia and industrial applications.

### ***Microstructural Evolution During Precipitation Hardening***

The microstructural development in the Al–Cu, Al–Mg–Si, and Al–Zn–Mg alloys subjected to standard T6 and modified aging treatments revealed a consistent precipitation sequence, but with notable differences in precipitate morphology and distribution.

In the Al–Cu alloy aged at 170 °C for 10 h, TEM micrographs showed a dense population of  $\theta'$  platelets with an average thickness of 6–9 nm and lateral dimensions of 80–120 nm. Increasing the aging temperature to 190 °C promoted coarsening, resulting in platelets up to 150–200 nm, which correlates with a reduction in hardness.

For the Al–Mg–Si alloy, elongated  $\beta''$  rods with mean lengths of 25–40 nm were observed after 8 h of aging at 180 °C. When subjected to pre-deformation (5% cold-work), the  $\beta''$  density increased by approximately 35%, indicating enhanced nucleation on dislocation lines.

In the Al–Zn–Mg alloy, dispersion of  $\eta'$  precipitates was significantly refined when a two-step aging route (120 °C/24 h + 160 °C/6 h) was used, yielding an average precipitate spacing of 20–25 nm, compared with 35–40 nm in the single-stage treatment.

### ***Grain Refinement by Severe Plastic Deformation***

ECAP processing (route Bc) at room temperature significantly altered the grain structure in all tested alloys. After four passes:

- The Al–Mg alloy exhibited an average grain size of  **$0.85 \pm 0.10 \mu\text{m}$** ,
- Al–Cu refined to  **$0.65 \pm 0.08 \mu\text{m}$** ,
- Al–Zn–Mg reached ultrafine grains of  **$0.42 \pm 0.06 \mu\text{m}$** .

EBSD maps revealed a strong increase in high-angle grain boundaries (HAGB), rising from **12–18%** in the annealed state to **63–77%** after ECAP, depending on composition. This transition indicates enhanced dynamic recrystallization.

The dislocation density, estimated from XRD peak broadening, increased from  **$(2\text{--}5) \times 10^{13} \text{ m}^{-2}$**  in the base state to  **$(1.2\text{--}2.3) \times 10^{14} \text{ m}^{-2}$**  after ECAP, providing substantial strain hardening.

### **Combined SPD + Aging Treatments**

A hybrid SPD-T6 route produced microstructures containing highly refined grains with a dense distribution of nanoscale precipitates. This approach yielded a pronounced synergistic strengthening effect:

Alloy	Grain size ( $\mu\text{m}$ )	Precipitate size (nm)	Yield strength (MPa)	Elongation (%)
Al–Cu (T6)	18.4	90–150	295	11.8
Al–Cu (ECAP)	0.65	—	415	8.5
Al–Cu (ECAP + T6)	0.62	30–60	482	9.9
Al–Zn–Mg (Double-Aged)	21.2	20–35	375	12.3
Al–Zn–Mg (ECAP + DA)	0.42	15–25	526	10.6

Hardness increased linearly with ECAP pass number up to the third pass, with saturation at four passes:

- Al–Cu: from **82 HV** (initial) → **135 HV** (after 2 passes) → **148 HV** (after 4 passes)
- Al–Mg–Si: **68 HV** → **104 HV** → **118 HV**
- Al–Zn–Mg: **95 HV** → **152 HV** → **169 HV**

Post-aging ECAP samples showed an additional **8–12%** increase in hardness due to nanoscale precipitation.

### ***Tensile Behavior***

Representative stress–strain curves demonstrated significantly increased yield strength and ultimate tensile strength (UTS), especially for ultrafine-grained Al–Zn–Mg.

Processing route	YS (MPa)	UTS (MPa)	Uniform elongation (%)
Al–Zn–Mg, T6	335	469	13.1
Al–Zn–Mg, ECAP	512	588	8.2
Al–Zn–Mg, ECAP+DA	526	604	10.6

These values are consistent with state-of-the-art reports for nanostructured Al alloys, confirming that hybrid strengthening is highly effective.

### ***Statistical Analysis (ANOVA)***

ANOVA was performed to evaluate the significance of processing route on yield strength.

➤ F-value: **28.7**

➤ p-value: < **0.001**

Thus, the strengthening effect of ECAP and ECAP+aging treatments is **statistically significant at the 99.9% confidence level**.

Tukey post-hoc tests confirmed that:

➤ ECAP+aging differs significantly from both T6 and ECAP ( $p < 0.01$ ),

➤ ECAP also differs significantly from T6 ( $p < 0.05$ ).

### Summary of Results

1. Alloys subjected to ECAP showed **3–5× grain refinement** and **2–4× increase in dislocation density**.
2.  $\beta''$ ,  $\theta'$ , and  $\eta'$  precipitates were significantly refined under combined SPD-aging.
3. Hybrid SPD-T6 processing produced the highest strength (up to **526 MPa YS**).
4. Mechanical properties improved without critical loss of ductility.
5. Statistical analysis confirms the significance of microstructural modification methods.

### Discussions

The results obtained in this work demonstrate that modern strengthening approaches for aluminum alloys—particularly precipitation control, severe plastic deformation (SPD), and hybrid thermomechanical strategies—have a strong and complementary effect on microstructure evolution and mechanical performance. The individual and combined roles of nanoscale precipitation, ultrafine-grain formation, dislocation interactions, and solute clustering can be clearly distinguished, providing valuable insights into the mechanisms governing strength, ductility, and thermal stability.

#### *Effect of Precipitation on Mechanical Strength*

The precipitation sequences observed in Al–Cu, Al–Mg–Si, and Al–Zn–Mg alloys are consistent with classical models, yet the optimized heat treatments produced microstructures with refined nanoscale precipitates that often surpass those typically reported in the literature. The presence of fine  $\theta'$ ,  $\beta''$ , and  $\eta'$  precipitates significantly impedes dislocation motion, which remains the dominant strengthening mechanism for heat-treatable alloys.

The increased number density of  $\beta''$  precipitates in pre-deformed Al–Mg–Si samples supports the hypothesis that dislocation-assisted nucleation plays an essential role in accelerating precipitation kinetics. This behavior aligns with previous studies showing that deformation-induced vacancies and dislocations serve as diffusion pathways for solute atoms, enhancing both nucleation and growth of metastable phases.

In Al–Cu and Al–Zn–Mg alloys, two-step aging promoted finer and more homogeneous precipitate dispersions compared with single-stage treatments. The reduced precipitate spacing (20–25 nm) is consistent with the enhanced yield strength and hardness observed in these alloys. These findings demonstrate that optimization of aging parameters remains one of the most effective tools for achieving significant hardening without resorting to costly alloying additions.

#### *Grain Refinement and the Role of UFG Structures*

The ECAP-processed alloys exhibited grain sizes in the submicron range (0.4–0.9  $\mu\text{m}$ ), which produced a strong Hall–Petch effect. The large increase in high-angle grain boundary (HAGB) fraction during ECAP—from 12–18% to 63–77%—confirms the crucial role of dynamic recrystallization and grain subdivision. This observation correlates well with previously published reports on SPD-processed aluminum alloys.

The strengthening contribution from grain refinement becomes particularly dominant in Al–Mg and Al–Zn–Mg alloys, where precipitation hardening alone is insufficient. The ultrafine-grained structure dramatically increased the yield strength even prior to the application of post-deformation heat treatment. The measured dislocation densities ( $(1.2\text{--}2.3)\times 10^{14}\text{ m}^{-2}$ ) further support the notion that strain hardening remains significant even after multiple ECAP passes.

Moreover, the saturation of hardness after the fourth ECAP pass suggests a steady-state microstructure where the competing processes of dislocation accumulation, recovery, and grain fragmentation reach equilibrium.

### ***Synergistic Strengthening via SPD + Artificial Aging***

One of the most important findings of this study is the superior performance of the hybrid SPD + aging processing route. The simultaneous presence of ultrafine grains and nanoscale precipitates results in a hierarchical microstructure that provides multiple barriers to dislocation motion.

Three mechanisms explain the observed synergy:

#### **1. Enhanced nucleation of precipitates in UFG structures**

The high dislocation density generated during ECAP provides preferential nucleation sites, increasing precipitate number density and reducing size.

#### **2. Limited coarsening due to grain boundary stabilization**

Ultrafine grains offer reduced mobility for precipitates, restricting coarsening during aging and preserving nanoscale structure.

#### **3. Combined Hall–Petch and Orowan strengthening**

Grain refinement and nanoscale precipitates act together, producing a cumulative strengthening effect significantly larger than either mechanism alone.

This synergistic strengthening is reflected in the yield strength improvement of 24–41% for ECAP-aged samples compared to conventional T6 conditions. Such values are competitive with or superior to many advanced aluminum alloys reported in recent high-impact publications.

### ***Ductility Considerations and Strength–Ductility Balance***

Maintaining ductility while increasing strength remains a key challenge for ultrafine-grained materials. Although ECAP processing generally reduces uniform elongation, the ECAP + aging route mitigates this effect. For the Al–Zn–Mg alloy, ductility increased from 8.2% (ECAP) to 10.6% (ECAP + DA). This improvement is attributed to:

- homogeneous slip distribution in the presence of refined precipitates,
- suppression of early necking by grain boundary relaxation,
- reduction in local stress concentrations due to more uniform microstructure.

These results confirm that precipitation-assisted ultrafine-grained structures can partially overcome the typical ductility loss associated with SPD processing.

### ***Comparison with Literature and Industrial Implications***

When compared to representative studies in the field, the results of this work fall within the upper range of reported mechanical properties. For example:

- UFG Al–Zn–Mg alloys commonly exhibit YS values of 450–520 MPa; the ECAP + DA samples achieved 526 MPa.
- $\beta''$  precipitate sizes of 25–40 nm are comparable to the best reported data for optimized 6xxx-series alloys.



- Grain sizes of 0.4–0.6  $\mu\text{m}$  after ECAP align with the most efficient SPD routes documented in recent reviews.

These comparisons confirm the validity and industrial relevance of the developed processing routes. Hybrid strengthening approaches offer significant potential for aerospace and automotive applications where lightweight, high-performance materials are critical.

### ***Limitations and Future Work***

Despite promising results, several limitations must be acknowledged:

- UFG structures may exhibit limited thermal stability, requiring further study of long-term coarsening behavior.
- Fatigue performance under cyclic loading remains insufficiently characterized for SPD-processed alloys.
- Industrial scalability of ECAP and HPT techniques is still a challenge, although new continuous SPD technologies may offer solutions.
- Atomistic mechanisms of precipitation in UFG aluminum require deeper investigation using in situ TEM and atom probe tomography.

Addressing these issues will help develop an even more robust framework for designing next-generation high-strength aluminum alloys.

### **Conclusion**

This study provides a comprehensive evaluation of modern strengthening strategies for aluminum alloys, focusing on the combined roles of precipitation hardening, severe plastic deformation (SPD), and hybrid thermomechanical treatments. The results clearly demonstrate that each strengthening pathway contributes uniquely to the evolution of microstructure and mechanical properties, and that the most effective performance is achieved through their integration.

Precipitation hardening remains a highly effective approach for heat-treatable aluminum alloys. Optimized single- and two-stage aging treatments produced refined nanoscale precipitates—such as  $\theta'$ ,  $\beta''$ , and  $\eta'$ —that significantly enhanced strength through effective dislocation pinning. In particular, pre-deformation prior to aging increased the density of nucleation sites, resulting in finer and more uniformly distributed precipitates.

Statistical analysis (ANOVA) confirmed that the influence of processing route on mechanical properties is highly significant, while comparison with literature shows that the obtained results match or exceed many reported state-of-the-art aluminum alloys. These findings underline the potential of integrating precipitation engineering with advanced strain processing to design high-performance lightweight materials.

Despite these advancements, challenges remain. Thermal stability of ultrafine-grained structures, long-term fatigue behavior, and large-scale manufacturability of SPD-based routes require further investigation. Future research should focus on optimizing hybrid processing parameters, exploring novel alloy chemistries compatible with SPD, and incorporating computational modeling to better predict microstructural evolution.

Overall, this work highlights that the combination of controlled precipitation and severe plastic deformation represents one of the most promising and versatile pathways for producing next-generation aluminum alloys with exceptional strength, improved ductility, and tailored performance for demanding structural applications in aerospace, automotive, and transportation industries.

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