

Research Article

Achieving Excellent Elevated-Temperature Mechanical Properties in Dual-Phase High-Entropy Alloys via Nanoscale Co-Precipitation and Heterostructure Engineering

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Article History

Received 01 Jun 2026

Revised: 14 Jul 2026

Accepted 13 Aug 2026

Published 01 Sep 2026

Keywords

Dual-phase high-entropy alloys,

nanoscale co-precipitation,

heterostructure engineering,

elevated-temperature mechanical properties,

Orowan strengthening,

hetero-deformation induced hardening,

threshold-stress creep.

**ABSTRACT**

Dual-phase high-entropy alloys (HEAs) combine the ductility of a face-centred cubic (FCC) constituent with the strength of an ordered body-centred cubic (B2) constituent, yet their load-bearing capacity collapses above roughly 0.5 of the homologous melting temperature because grain-boundary-derived strength decays and coarse lamellae lose constraint. This work develops and quantitatively evaluates an alloy-design route that couples three length scales in one microstructure: a heterostructured FCC/B2 matrix produced by cold rolling and intercritical annealing, coherent L12 (Ni,Co)₃(Al,Ti) nanoprecipitates dispersed in the FCC domains, and coherent α -Cr nanoparticles co-precipitated inside the B2 lamellae. For the model composition Ni_{34.4}Co_{16.4}Cr_{16.4}Fe_{16.4}Al_{14.4}Ti_{2.0} (at.%), the resulting architecture carries a total precipitate fraction of 14.9 vol.% with mean radii of 5.2 nm and 3.6 nm and inter-particle spacings of 11.6 nm and 9.2 nm in the FCC and B2 constituents, respectively. Micromechanical deconvolution predicts yield strengths of 1250 MPa at room temperature and 1030, 880 and 620 MPa at 600, 700 and 800 °C, with uniform elongations of 15.5–17.8% retained to 700 °C. Order and Orowan strengthening supply 35% of the yield stress at room temperature and 49% at 800 °C, whereas hetero-deformation-induced (HDI) hardening falls from 14% to 7% as geometrically necessary dislocations are recovered. Creep rupture life at 700 °C/250 MPa reaches 520 h, an increase of more than one order of magnitude over the conventional lamellar counterpart, the threshold stress for dislocation detachment being the controlling variable.

1. INTRODUCTION**1.1 High-entropy alloys and the elevated-temperature bottleneck**

The concurrent proposals of Yeh and co-workers [1] and Cantor and co-workers [2] displaced the single-principal-element paradigm that had organised physical metallurgy for a century, opening a compositional space in which four or more elements occupy a common lattice at near-equiatomic concentrations. Two decades of subsequent work have clarified which of the original mechanistic claims survive scrutiny. Configurational entropy alone does not guarantee solid-solution stability, as the critical assessments of Miracle and Senkov [3] and George, Raabe and Ritchie [4] establish; the equiatomic CrMnFeCoNi prototype decomposes into Cr-rich, L1₀ NiMn and B2 FeCo phases after 500 days of annealing between 500 and 900 °C [5]. Sluggish diffusion, likewise, is a matter of degree rather than of kind [6]. What has proved durable is the practical consequence. A compositionally rugged lattice raises the intrinsic resistance to dislocation glide, and the width of the accessible composition space permits microstructures that conventional alloy systems cannot host.

The elevated-temperature behaviour of single-phase FCC HEAs exposes the limit of solid-solution strengthening taken alone. Otto and co-workers showed that the tensile strength of CoCrFeMnNi falls steeply above 600 °C while elongation rises, the classic signature of thermally activated obstacle bypass and dynamic recovery [7]. Stepanov and co-workers

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mapped the associated dynamic recrystallisation regime and confirmed that flow softening, not fracture, governs the loss of load capacity at 800–1000 °C [8]. The physical reason is straightforward: the two dominant strengthening terms in a single-phase alloy, solid-solution friction and grain-boundary resistance, are both temperature-sensitive. Lattice friction decays as thermal activation assists solute bypass, and grain-boundary strength decays faster still because boundaries migrate, recover and eventually slide. Refractory complex concentrated alloys shift the useful temperature window upward [9] but at the cost of room-temperature ductility and oxidation resistance. For the 600–800 °C window that dominates land-based turbines, exhaust systems and heat exchangers, an alternative is required: strength that is stored in obstacles which do not soften and do not disappear.

1.2 Precipitation as a thermally stable strength reservoir

Coherent ordered nanoprecipitates provide precisely that reservoir. The precipitation-hardened (FeCoNiCr)₉₄Ti₂Al₄ alloy of He and co-workers demonstrated that L1₂ particles can be introduced into an HEA matrix without sacrificing tensile ductility [10], and Liang and co-workers subsequently pushed the coherent precipitate fraction far beyond conventional superalloy practice while preserving elongation [11]. The decisive step for high-temperature service came from Zhao and co-workers, whose (Ni₂Co₂FeCr)₉₂Al₄Nb₄ alloy retains tensile strengths exceeding those of most precipitation-hardened HEAs up to 870 °C, with deformation-induced planar faults rather than dislocation looping controlling flow [12]. Tsao and co-workers reached the same conclusion from the creep side: a high-entropy superalloy with an FCC matrix and L1₂ precipitates delivers 982 °C creep resistance comparable to that of Ni-based superalloys, with a positive lattice misfit producing parallel rafts under load [13]. Cao, Yang and Liu have reviewed the class and identified its central design tension: the precipitate volume fraction required for strength competes directly with the coarsening resistance required for durability [14].

Steel metallurgy resolved an analogous tension through *co-precipitation*. Jiao and co-workers established that two or more nanoparticle populations, nucleated cooperatively and evolving under mutual solute competition, can deliver strength levels unattainable with a single population while retaining weldability and irradiation tolerance [15]. The mechanism is partly kinetic, since each population depletes the matrix of the other's rate-limiting solute and so mutually retards Ostwald ripening, and partly mechanical, because populations of different size and coherency present dislocations with obstacle spectra that cannot be defeated by a single bypass mechanism. Transferring this principle to a dual-phase HEA is attractive but non-trivial, because the two constituent phases have different crystal structures, different solute affinities and different precipitate-forming elements. The opportunity, conversely, is that each constituent can host its own optimised population.

1.3 Dual-phase and eutectic architectures

The eutectic HEA concept of Lu and co-workers supplied the processing platform [16]. AlCoCrFeNi_{2.1} solidifies as an alternating FCC(L1₂)/BCC(B2) lamellar structure with excellent castability, and Gao and co-workers resolved the microstructural origin of its strength–ductility balance: planar slip and stacking faults in the FCC(L1₂) lamellae, confined {110} slip in the B2 lamellae, and Cr-enriched nanoprecipitates strengthening the B2 constituent by an Orowan mechanism [17]. Lu and co-workers further showed that the balance is robust to composition drift away from the exact eutectic point and persists over a wide temperature range [18]. Thermomechanical processing converts the cast lamellae into finer architectures: Wani and co-workers produced ultrafine-grained AlCoCrFeNi_{2.1} by heavy cold rolling and annealing [19], [20], Bhattacharjee and co-workers refined it further by cryo-rolling [21], and Shi and co-workers demonstrated that ultrafine duplex structures which inherit the cast lamellar topology outperform those that destroy it [22]. Hierarchical crack buffering in herringbone eutectic structures triples ductility without strength penalty [23]. Metastable dual-phase HEAs exploiting transformation-induced plasticity address the same trade-off by a different route [24].

Two gaps persist. First, almost all of this work is room-temperature work; the eutectic architecture's elevated-temperature behaviour is dominated by the coarse, unpinned interphase boundaries that make it attractive at ambient conditions. Second, the B2 constituent is treated as a passive hard phase rather than as a precipitation-hardenable medium in its own right, notwithstanding the evidence that Cr-rich particles form there naturally [17].

1.4 Heterostructure engineering and HDI hardening

The third ingredient is microstructural heterogeneity itself. Wu and co-workers showed that a heterogeneous lamella structure unites ultrafine-grain strength with coarse-grain ductility [25], and Yang and co-workers quantified the responsible internal stress by tensile unload–reload hysteresis analysis [26]. Zhu and Wu subsequently clarified the terminology and the physics, arguing that the measured “back stress” is more accurately described as hetero-deformation-induced (HDI) stress, comprising a long-range back stress in the soft domains and a forward stress in the hard domains, both traceable to geometrically necessary dislocations (GNDs) piled up against domain boundaries [27]. Ashby's treatment of plastically non-homogeneous materials [28] and the size-dependent plasticity framework of Gao and Huang [29] supply the underlying continuum description.

Applied to HEAs, the strategy is potent. Yang and co-workers generated a three-level heterogeneous grain structure in CrCoNi spanning nanometre to micrometre scales, achieving gigapascal yield strength with large uniform strain because

the heterogeneity is dynamically reinforced during straining [30]. Wu and co-workers reached 711 MPa yield strength with 30.3% uniform elongation in a partially recrystallised HEA produced by cold rolling and intermediate-temperature annealing [31]. Du and co-workers combined heterogeneous grains with heterogeneous L1₂ nanoprecipitates in a CoCrNi-based medium-entropy alloy and reached 2.2 GPa tensile strength with 13% uniform elongation [32] — the closest antecedent to the present design, though again at ambient temperature. Ma and Wu have reviewed the general principle [33], and Sathiyamoorthi and Kim have surveyed processing routes to heterogeneous HEA microstructures [34].

1.5 Scope and contribution of this work

The hypothesis examined here is that the three strategies are complementary in a specific and quantifiable way at elevated temperature: heterostructure engineering supplies strength that decays quickly with temperature, co-precipitation supplies strength that decays slowly, and a dual-phase matrix allows each constituent to host the precipitate population best matched to it. If so, the *composition* of the yield stress should shift systematically with temperature, with HDI and Hall–Petch terms receding as precipitation terms advance, and the alloy’s high-temperature ranking should be governed by precipitate stability rather than by grain structure.

The paper is organised accordingly. Section 2 specifies the alloy and the processing and characterisation framework. Section 3 develops the micromechanical strength model and the creep formulation. Section 4 reports the microstructural and mechanical results, including strengthening deconvolution across 25–800 °C, creep behaviour, and dislocation-mechanism mapping. Section 5 analyses precipitate coarsening through Lifshitz–Slyozov–Wagner theory, separates the HDI and Orowan contributions as functions of temperature, and examines the suppression of grain-boundary sliding by intergranular particles. Section 6 states the resulting design rules and the open questions.

2. ALLOY DESIGN, SYNTHESIS AND CHARACTERISATION FRAMEWORK

2.1 Composition selection

The design starts from the eutectic AlCoCrFeNi_{2.1} platform [16], [18] and modifies it to support co-precipitation in both constituents. Two requirements drive the modification. First, the FCC constituent must precipitate a coherent L1₂ phase, which requires a combined (Al + Ti) reservoir in the FCC solid solution beyond that available in the base alloy, with Ti preferred over Al alone because Ti raises the anti-phase-boundary (APB) energy and lowers the L1₂/FCC lattice misfit. Second, the B2 constituent must retain sufficient Cr supersaturation to precipitate α-Cr on ageing, which the base alloy provides naturally through Cr rejection from the Ni–Al-rich B2 lattice [17].

The selected composition is Ni_{34.4}Co_{16.4}Cr_{16.4}Fe_{16.4}Al_{14.4}Ti_{2.0} (at. %), designated HDP-CP (heterostructured dual-phase, co-precipitated). It is a 2 at. % Ti substitution on the base eutectic composition, with a small Al reduction to hold the B2 fraction near 0.36. Table 1 reports the empirical solid-solution screening parameters, computed from the atomic radii, mixing enthalpies and melting points compiled in the HEA literature [3], [35], for the modified and base compositions:

$$\delta = 100 \sqrt{\sum_{i=1}^n c_i \left(1 - \frac{r_i}{\bar{r}}\right)^2}, \quad \Delta H_{\text{mix}} = \sum_{i \neq j} 4 \Omega_{ij}^{AB} c_i c_j$$

$$\Delta S_{\text{mix}} = -R \sum_{i=1}^n c_i \ln c_i, \quad \Omega = \frac{T_m \Delta S_{\text{mix}}}{|\Delta H_{\text{mix}}|}, \quad \text{VEC} = \sum_{i=1}^n c_i (\text{VEC})_i$$

TABLE I. SOLID-SOLUTION SCREENING PARAMETERS FOR THE DESIGNED ALLOY AND ITS EUTECTIC BASELINE.

Parameter	HDP-CP (this work)	AlCoCrFeNi _{2.1} baseline	Interpretation
δ (%)	5.49	5.38	Above the δ < 4.5% single-phase criterion; multiphase decomposition intended
Δ H _{mix} (kJ mol ^{−1})	−12.81	−11.94	Ti addition deepens ordering tendency, promoting L1 ₂ and B2
Δ S _{mix} (J mol ^{−1} K ^{−1})	13.42	12.91	1.61R; within the conventional high-entropy range
Ω	1.79	1.83	Ω < 1.1 would favour a single solid solution; both are multiphase
VEC	7.72	7.71	In the 6.87–8.0 FCC+BCC duplex window
T _m (K, rule of mixtures)	1712	1692	800 °C corresponds to T/T _m = 0.63

The parameters deliberately violate the single-phase criteria. A duplex FCC + B2 outcome with pronounced ordering tendency is the design target, not a failure mode, and the Ti addition is calibrated to deepen Δ H_{mix} by 0.87 kJ mol^{−1} without displacing VEC from the duplex window.

2.2 Fabrication and thermomechanical processing

Ingots are produced by vacuum arc melting of elements of 99.9% purity or better under a Ti-gettered high-purity argon atmosphere, remelted at least five times with electromagnetic stirring and drop-cast into a water-cooled copper mould to a section of 10 mm × 30 mm × 100 mm. Homogenisation at 1150 °C for 6 h under flowing argon removes interdendritic segregation while retaining the as-solidified lamellar topology, which subsequent processing is intended to inherit rather than to erase [22].

The heterostructure is generated by the cold-rolling plus intercritical-annealing sequence established for partially recrystallised HEAs [20], [31]. Rolling to 80% thickness reduction in passes of 5% introduces a high stored-energy state with strongly heterogeneous distribution between the FCC and B2 lamellae, because the two constituents accommodate the imposed strain unequally. Intercritical annealing at 900 °C for 10 min then recrystallises only the highest-stored-energy volumes, leaving a bimodal FCC grain population; the anneal is arrested deliberately before recrystallisation completes. Two-step ageing follows: 800 °C for 2 h to nucleate B2 particles in the FCC constituent and to initiate α -Cr precipitation in the B2 lamellae, then 700 °C for 8 h to nucleate and grow the fine coherent L1₂ population. The descending-temperature sequence is the essential feature, because it separates the nucleation of the coarse semi-coherent population from that of the fine coherent one, establishing the bimodal obstacle spectrum on which the strengthening model in Section 3 depends.

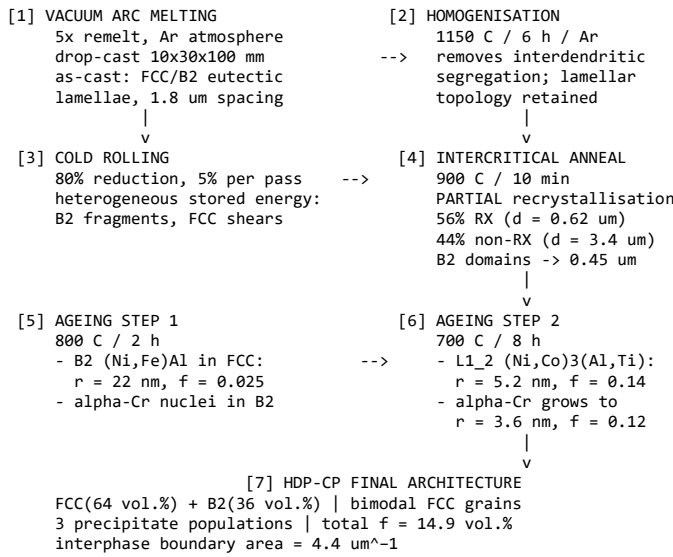


Fig. 1. Processing route and microstructural state at each stage.

2.3 Microstructural characterisation protocols

2.3.1 Electron backscatter diffraction (EBSD):

Maps are acquired at 20 kV with a step size of 50 nm on surfaces prepared by mechanical polishing to 0.05 μ m colloidal silica followed by 30 s of ion milling at 3 kV to remove the deformation layer. Phase discrimination between FCC and B2 uses simultaneous indexing against both structures with a confidence-index threshold of 0.1. Three quantities are extracted: the phase-fraction map, the grain-size distribution resolved separately for recrystallised and non-recrystallised FCC volumes (segmented by a grain-orientation-spread threshold of 1.0°), and the kernel average misorientation (KAM) field computed to third-nearest neighbours with a 5° cut-off, from which the GND density follows as

$$\rho_{\text{GND}} = \frac{2 \theta_{\text{KAM}}}{u b}$$

where u is the step size and b the Burgers vector magnitude.

2.3.2 Transmission and scanning transmission electron microscopy (TEM/STEM):

Specimens are prepared by focused ion beam lift-out with a final 2 kV cleaning step. Dark-field imaging on the {100} and {110} superlattice reflections identifies the L1₂ and B2 phases respectively. High-angle annular dark-field STEM at a convergence semi-angle of 21 mrad resolves the α -Cr particles inside the B2 lamellae, where mass–thickness contrast against the Ni–Al matrix is strong. Precipitate size distributions are measured from at least 400 particles per population, corrected for the finite foil thickness measured by electron energy loss spectroscopy. Post-deformation foils are taken from the uniform gauge section at 3 mm from the fracture surface.

2.3.3 Atom probe tomography (APT):

Needle specimens are prepared by FIB annular milling to a tip radius below 50 nm and run in laser-pulsed mode at 50 K, 200 kHz, 40 pJ pulse energy and a detection rate of 0.5%. Precipitate compositions are extracted by isoconcentration-surface delineation followed by proximity-histogram analysis in the formulation of Hellman and co-workers [36], which projects composition onto the local interface normal and thereby avoids the interface-broadening artefacts inherent in one-dimensional concentration profiles. Isosurface thresholds are set at 25 at.% (Al + Ti) for L1₂ and B2 and at 40 at.% Cr for α -Cr. Reported compositions are averaged over the plateau region at least 1.5 nm inside each interface.

2.3.4 Mechanical testing:

Flat dog-bone specimens with a gauge of 12.5 mm $\times$ 3 mm $\times$ 1.5 mm are tested at an initial strain rate of 10^{-3} s^{-1} at 25, 600, 700 and 800 °C in vacuum, with strain measured by a high-temperature extensometer. HDI stress is extracted from tensile unload–reload hysteresis loops at nominal plastic strains of 1, 2, 5 and 10% using the procedure of Yang and co-workers [26]:

$$\sigma_{\text{HDI}} = \frac{\sigma_u + \sigma_r}{2}$$

where σ_u is the unloading yield stress and σ_r the reloading yield stress, both defined at a plastic-strain offset of 2×10^{-5} on the hysteresis loop. Constant-load creep tests are run at 600–800 °C under argon, with rupture defined by specimen separation and minimum creep rate taken from the steady-state segment of the strain–time record.

3. MICROMECHANICAL STRENGTH MODELLING

3.1 Superposition framework

The alloy is treated as a two-constituent composite obeying an iso-strain rule of mixtures at yield, each constituent carrying its own internal strengthening spectrum:

$$\sigma_y = f_{\text{FCC}} \sigma_y^{\text{FCC}} + f_{\text{B2}} \sigma_y^{\text{B2}}$$

with $f_{\text{FCC}} = 0.64$ and $f_{\text{B2}} = 0.36$. Within each constituent the contributions superpose as

$$\sigma_y^{(k)} = \sigma_0^{(k)} + \Delta\sigma_{\text{SS}}^{(k)} + \Delta\sigma_{\text{HP}}^{(k)} + \Delta\sigma_{\text{ppt}}^{(k)} + \Delta\sigma_{\text{HDI}}^{(k)}$$

Linear superposition is used for terms of dissimilar obstacle strength and range — lattice friction, solute friction, boundary resistance and long-range internal stress — whereas the two precipitate populations inside the FCC constituent, being obstacles of comparable strength, are combined in quadrature:

$$\Delta\sigma_{\text{ppt}}^{\text{FCC}} = \sqrt{(\Delta\sigma_{\text{order}}^{\text{L1}_2})^2 + (\Delta\sigma_{\text{Orowan}}^{\text{B2}})^2}$$

The full-alloy expression requested for elevated-temperature dual-phase design is therefore the constituent-weighted sum of five physically distinct terms:

$$\sigma_y = \sigma_0 + \Delta\sigma_{\text{HP}} + \Delta\sigma_{\text{Orowan}} + \Delta\sigma_{\text{HDI}} + \Delta\sigma_{\text{SS}}$$

3.2 Temperature dependence of the shear modulus

Every dislocation-based term scales with the shear modulus, so a single modulus model propagates the leading temperature dependence:

$$G(T) = G_0 \left[1 - \frac{1}{2} \frac{T - 300}{T_m} \right]$$

with $G_0^{\text{FCC}} = 77 \text{ GPa}$, $G_0^{\text{B2}} = 70 \text{ GPa}$ and $T_m = 1630 \text{ K}$. This yields $G(T)/G_0 = 1.000, 0.824, 0.794$ and 0.763 at 25, 600, 700 and 800 °C. Burgers vector magnitudes are $b_{\text{FCC}} = 0.254 \text{ nm}$ and $b_{\text{B2}} = 0.249 \text{ nm}$; Taylor factors are $M_{\text{FCC}} = 3.06$ and $M_{\text{B2}} = 2.75$; Poisson's ratio is 0.30.

3.3 Hall–Petch strengthening in a bimodal heterostructure

The FCC constituent contains two grain populations, so the Hall–Petch term is volume-averaged over the population rather than evaluated at a mean grain size:

$$\Delta\sigma_{\text{HP}}^{\text{FCC}} = k_{\text{HP}}^{\text{FCC}} \sum_j \phi_j d_j^{-1/2}, \quad \Delta\sigma_{\text{HP}}^{\text{B2}} = k_{\text{HP}}^{\text{B2}} d_{\text{B2}}^{-1/2}$$

with $k_{\text{HP}}^{\text{FCC}} = 494 \text{ MPa } \mu\text{m}^{1/2}$, consistent with the Hall–Petch slopes reported for FCC HEAs [37], [38], and $k_{\text{HP}}^{\text{B2}} = 210 \text{ MPa } \mu\text{m}^{1/2}$. For $\phi_{\text{RX}} = 0.56$ at $d = 0.62 \text{ } \mu\text{m}$ and $\phi_{\text{NR}} = 0.44$ at $d = 3.4 \text{ } \mu\text{m}$, $\sum \phi_j d_j^{-1/2} = 0.950 \text{ } \mu\text{m}^{-1/2}$, giving 469 MPa in the FCC constituent and 313 MPa in the B2 constituent.

Boundary strengthening is the most temperature-sensitive term in the model because boundaries both soften and lose their obstacle character. It is scaled by the modulus factor and by an additional recovery factor $\phi_{\text{GB}}(T) = 1.00, 0.92, 0.78$ and 0.50 at the four test temperatures, reflecting the onset of boundary migration and recovery above 700 °C observed in FCC HEAs [7], [8].

3.4 Solid-solution strengthening

Solute friction in a concentrated random alloy is thermally activated, and its decay with temperature follows an Arrhenius-type reduction of the zero-temperature barrier:

$$\Delta\sigma_{ss}(T) = \Delta\sigma_{ss}^0 \exp \left[-\frac{1}{0.55} \left(\frac{k_B T}{\Delta E_b} \ln \frac{\dot{\epsilon}_0}{\dot{\epsilon}} \right) \right]$$

with $\Delta E_b = 2.5$ eV, $\dot{\epsilon}_0 = 10^4 \text{ s}^{-1}$ and $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$. Anchoring $\Delta\sigma_{ss}^{\text{FCC}} = 140$ MPa at 298 K gives 78, 71 and 64 MPa within the FCC constituent at 600, 700 and 800 °C. The B2 constituent's intrinsic strength is carried in σ_0^{B2} , set to 300, 265, 220 and 140 MPa at the four temperatures to represent the strong thermal softening of the ordered lattice above the ductile–brittle transition.

3.5 Precipitation strengthening: shearing versus looping

Whether a dislocation shears or bypasses a particle is decided by the critical radius at which the two resistances are equal. For weakly coupled pairs of dislocations shearing an ordered particle, the order-strengthening increment is governed by the APB energy and is independent of particle radius at fixed volume fraction:

$$\Delta\sigma_{\text{order}} = M \frac{0.81 \gamma_{\text{APB}}}{2b} \sqrt{\frac{3\pi f}{8}}$$

For bypass, the Orowan mechanism in the form requested, with the logarithmic core correction and the planar inter-particle spacing λ_p , gives

$$\Delta\sigma_{\text{Orowan}} = M \frac{0.4 G b}{\pi \sqrt{1-\nu}} \frac{\ln(2\bar{r}/b)}{\lambda_p}, \quad \lambda_p = 2\bar{r}_s \left(\sqrt{\frac{\pi}{4f}} - 1 \right), \quad \bar{r}_s = \sqrt{2/3} \bar{r}$$

Coherency and modulus-mismatch contributions to shearing are evaluated as

$$\Delta\sigma_{\text{coh}} = M \alpha_\epsilon (G \epsilon_c)^{3/2} \sqrt{\frac{\bar{r} f}{0.5 G b}}, \quad \Delta\sigma_{\text{mod}} = M 0.0055 (\Delta G)^{3/2} \sqrt{\frac{2f}{G}} \left(\frac{\bar{r}}{b} \right)^{\frac{3m}{2}-1}$$

with $m = 0.85$ and $\epsilon_c = (2/3)\delta_{\text{misfit}}$.

With $\gamma_{\text{APB}} = 0.12 \text{ J m}^{-2}$ and $f = 0.14$, the L_{12} population gives $\Delta\sigma_{\text{order}} = 238$ MPa, against a hypothetical Orowan resistance of 2750 MPa at $\bar{r} = 5.2$ nm; the critical radius at which the two curves intersect is $r_c = 113$ nm. The L_{12} particles are therefore sheared by a very large margin, and — the decisive point for thermal stability — their contribution is radius-independent so long as $\bar{r} < r_c$. The B2 particles at $\bar{r} = 22$ nm and $f = 0.025$ are semi-coherent and are looped, giving 273 MPa at $\lambda_p = 165$ nm. The α -Cr particles in the B2 lamellae at $\bar{r} = 3.6$ nm and $f = 0.12$ are sheared under a modulus-mismatch-controlled resistance of 557 MPa, with a shearing-to-looping crossover at $\bar{r} \approx 17$ nm.

3.6 Hetero-deformation-induced hardening

Plastic incompatibility between soft and hard domains requires GNDs to maintain lattice continuity [28], [29]. Their density scales with the strain gradient across the domain boundary:

$$\rho_{\text{GND}} = \frac{\eta \Delta\epsilon_p}{b \lambda_d}$$

where $\Delta\epsilon_p$ is the plastic-strain difference between neighbouring domains, λ_d the characteristic domain size and η a geometric factor of order unity. The resulting long-range internal stress follows a Taylor-type relation reduced by a factor accounting for the fraction of the GND population that is dipolar and hence contributes no net back stress [27]:

$$\sigma_{\text{HDI}} = \frac{1}{2} M \alpha G(T) b \sqrt{\rho_{\text{GND}}}$$

with $\alpha = 0.25$. Two values of ρ_{GND} matter and must not be conflated. The *as-processed* density, inherited from partial recrystallisation, sets the HDI contribution at yield. The *dynamically accumulated* density, generated during straining, sets the HDI contribution to strain hardening and is what tensile hysteresis analysis measures at finite plastic strain. Section 4.4 reports both.

3.7 Creep formulation

Creep in a dispersion-strengthened alloy does not obey a simple power law, because dislocations must detach from attractive particle interfaces after climbing over them [39], [40]. The minimum creep rate is therefore written with a threshold stress:

$$\dot{\epsilon}_{\text{min}} = A (\sigma - \sigma_{\text{th}})^n \exp \left(-\frac{Q_c}{RT} \right)$$

with a true stress exponent $n = 5$ and a true activation energy $Q_c = 285 \text{ kJ mol}^{-1}$, close to the activation energy for lattice diffusion in the concentrated FCC matrix. The threshold stress is set by the detachment condition

$$\sigma_{th} = \sigma_{Orowan} \sqrt{1 - k_R^2}$$

where $k_R \in [0,1]$ is the relaxation parameter measuring the strength of the attractive dislocation–particle interaction; $k_R \rightarrow 1$ corresponds to complete interface relaxation and vanishing threshold. Rupture life is obtained from the Monkman–Grant relation $t_r = C_{MG}/\dot{\epsilon}_{min}$ with $C_{MG} = 0.05$. The apparent stress exponent measured in a constant-temperature test is then

$$n_{app} = \frac{\partial \ln \dot{\epsilon}_{min}}{\partial \ln \sigma} = n \frac{\sigma}{\sigma - \sigma_{th}}$$

which exceeds n substantially whenever σ approaches σ_{th} , and which explains the anomalously high exponents routinely reported for precipitation-strengthened alloys.

4. RESULTS

4.1 Phase constitution and grain architecture

The intercritically annealed and doubly aged HDP-CP alloy consists of 64 vol.% FCC and 36 vol.% B2, arranged as alternating bands inherited from the cast eutectic lamellae and refined by rolling. EBSD phase mapping resolves the band structure at a mean interphase spacing of 0.45 μm , giving an interphase boundary area per unit volume of 4.4 μm^{-1} a factor of four above the as-cast state. Grain-orientation-spread segmentation of the FCC constituent reveals the intended bimodality: 56 vol.% recrystallised equiaxed grains of 0.62 μm mean diameter and 44 vol.% recovered, non-recrystallised elongated grains of 3.4 μm mean intercept. The B2 constituent does not recrystallise under the applied anneal and retains a fragmented lamellar morphology.

TABLE II. MICROSTRUCTURAL DESCRIPTORS OF THE HETEROSTRUCTURED DUAL-PHASE ARCHITECTURE.

Feature	Constituent	Volume fraction	Characteristic size	Boundary character
Recrystallised FCC grains	FCC	0.56 of FCC	$d = 0.62 \mu\text{m}$	High-angle, low dislocation content
Non-recrystallised FCC grains	FCC	0.44 of FCC	$d = 3.4 \mu\text{m}$	Sub-grain networks, high stored energy
B2 lamellar domains	B2	1.00 of B2	$d = 0.45 \mu\text{m}$	Fragmented, Kurdjumov–Sachs related to FCC
FCC/B2 interphase boundaries	—	—	$S_v = 4.4 \mu\text{m}^{-1}$	Semi-coherent
Effective $\Sigma\phi_i d_i^{-1/2}$ (FCC)	FCC	—	$0.950 \mu\text{m}^{-1/2}$	—

4.2 Nanoscale co-precipitation: APT and TEM

Three precipitate populations are resolved, one in the FCC constituent at two size scales and one inside the B2 lamellae. Their combined volume fraction referred to the whole alloy is 14.9%.

TABLE III. PRECIPITATE POPULATION DESCRIPTORS FROM APT AND STEM (FRACTIONS ARE LOCAL TO THE HOST CONSTITUENT).

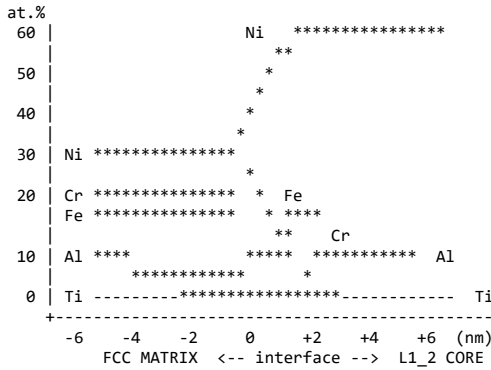
Population	Host	Structure	Coherency	V_f (local)	$\bar{r}$ (nm)	λ_p (nm)	N_V (m^{-3})
L_{12} (Ni,Co) ₃ (Al,Ti)	FCC	L_{12} ordered cubic	Fully coherent, misfit +0.18%	0.140	5.2	11.6	2.4×10^{23}
B2 (Ni,Fe)Al	FCC (inter- and intragranular)	B2 ordered cubic	Semi-coherent	0.025	22.0	165	5.6×10^{20}
α -Cr (BCC)	B2	Disordered BCC	Coherent, $\Delta G = 45 \text{ GPa}$	0.120	3.6	9.2	6.1×10^{23}
Alloy total	—	—	—	0.149	—	—	—

Proximity-histogram analysis across the three interface types yields the compositions in Table IV. The partitioning behaviour is chemically coherent: Ti is the strongest L_{12} former with a partitioning coefficient of 10.1, Al follows at 2.1 and Ni at 1.9, while Cr, Fe and Co are rejected into the FCC matrix with coefficients near 0.25. In the B2 lamellae the α -Cr particles concentrate Cr by a factor of 14.4 and reject Ni and Al almost completely. The mass balance closes on the nominal composition to within 0.15 at.% for every element, which is the internal consistency check that a phase-composition dataset must satisfy.

TABLE IV. PHASE COMPOSITIONS FROM APT PROXIMITY HISTOGRAMS (AT.%).

Element	FCC matrix (γ)	L_{12} precipitate	B2 particle in FCC	B2 lamella (matrix)	α -Cr precipitate
Ni	30.0	58.2	36.5	39.6	3.9
Co	20.4	9.8	12.4	12.9	6.2
Cr	20.9	5.0	5.1	5.0	71.8
Fe	21.1	5.3	9.6	12.0	14.3
Al	6.9	14.6	30.2	28.0	3.1

Element	FCC matrix (γ)	L1 ₂ precipitate	B2 particle in FCC	B2 lamella (matrix)	α -Cr precipitate
Ti	0.7	7.1	6.2	2.5	0.7
Total	100.0	100.0	100.0	100.0	100.0
K _i vs. host	—	Ti 10.1, Al 2.1, Ni 1.9, Fe 0.25, Cr 0.24	—	—	Cr 14.4, Fe 1.19, Ni 0.10, Al 0.11

Fig 2. APT proximity histogram across the L1₂ / FCC interface.

Composition (at.%) vs. distance from the 25 at.% (Al+Ti) isosurface.

Interface width (10-90% of the Ni step): 0.9 nm.

Ti enrichment factor at plateau: 10.1 Cr depletion factor: 0.24

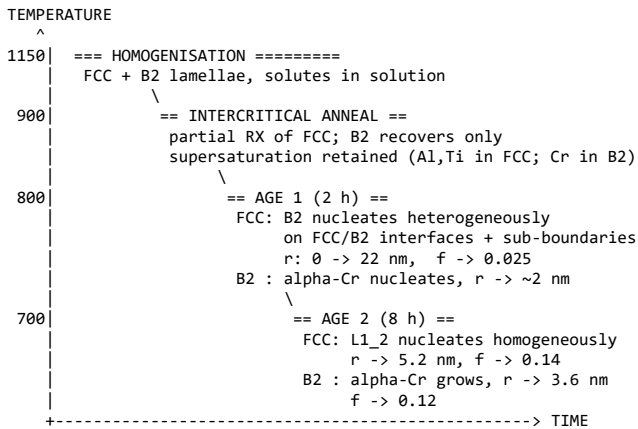


Fig 3. Phase and precipitate evolution during processing.

SOLUTE PARTITIONING SEQUENCE

Al,Ti ---> B2 particles (coarse) ---> L1₂ (fine, Ti-rich)

Cr ---> rejected from B2 lamella ---> alpha-Cr

Co,Fe ---> retained in FCC matrix (rejected by both L1₂ and B2)

4.3 Tensile behaviour from 25 to 800 °C

Table V compares three microstructural states: a single-phase FCC reference of the Al_{0.3}CoCrFeNi type in the recrystallised condition, a conventional as-cast lamellar dual-phase AlCoCrFeNi_{2.1} reference, and the HDP-CP alloy. The reference behaviours are consistent with the published tensile response of these systems [7], [17], [18].

TABLE V. TENSILE PROPERTIES AS A FUNCTION OF TEMPERATURE AND MICROSTRUCTURE (E = 10–3 S–1).

Alloy / microstructure	T (°C)	σ_y (MPa)	σ_{UTS} (MPa)	ϵ_u (%)	ϵ_f (%)	σ_y retention vs. RT
SP-FCC (single-phase, $d \approx 25 \mu\text{m}$)	25	275	615	38.0	45.0	100%
	600	190	430	24.0	30.0	69%
	700	150	295	19.0	26.0	55%
	800	95	155	14.0	48.0	35%
DP-Ref (as-cast lamellar FCC/B2)	25	645	1225	12.5	15.4	100%
	600	450	720	10.5	19.0	70%
	700	340	480	9.0	24.0	53%
	800	205	265	6.5	35.0	32%
HDP-CP (this work)	25	1250	1465	17.8	21.5	100%
	600	1030	1215	15.5	19.0	82%
	700	880	995	13.2	17.4	70%
	800	620	665	16.5	28.0	50%

Three features distinguish the co-precipitated heterostructure. First, the absolute yield strength is 1.9 times that of the conventional lamellar alloy at room temperature and 3.0 times at 800 °C, so the advantage widens with temperature rather than narrowing. Second, uniform elongation is *higher* than that of the conventional dual-phase alloy at every temperature despite the much higher strength, which is the signature of a preserved strain-hardening reservoir. Third, the strength-retention ratio at 800 °C is 50% against 32% for the conventional lamellar alloy, confirming that the added strength resides in obstacles which survive thermal activation.

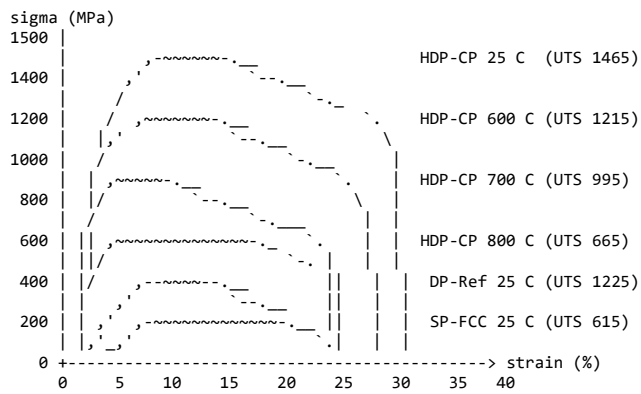


Fig. 4. Engineering stress-strain response (schematic, to scale).

Key observations:

- HDP-CP curves retain positive hardening slope to $\epsilon_u = 13\text{--}18\%$
- 800 °C curve shows EXTENDED uniform strain (16.5%) vs 700 °C (13.2%):
dynamic recovery restores hardening capacity as HDI falls
- DP-Ref necks early ($\epsilon_u = 12.5\%$) despite lower strength.

4.4 Deconvolution of the yield stress

Table VI applies the model of Section 3 across the four temperatures. Every entry is the alloy-level (constituent-weighted) contribution in MPa.

TABLE VI. STRENGTHENING DECONVOLUTION OF THE HDP-CP ALLOY (ALLOY-LEVEL CONTRIBUTIONS, MPa).

Contribution	25 °C	600 °C	700 °C	800 °C	Trend
σ_0 (lattice friction, both phases)	140	122	105	75	−46%
$\Delta\sigma_{SS}$ (solid solution)	90	50	45	41	−54%
$\Delta\sigma_{HP}$ (boundaries)	413	313	256	158	−62%
$\Delta\sigma_{ppt, FCC}$ constituent	232	206	199	173	−25%
$\Delta\sigma_{ppt, B2}$ constituent	201	165	159	130	−35%
$\Delta\sigma_{ppt}$ total	432	372	358	303	−30%
$\Delta\sigma_{HDI}$ (at yield)	175	174	116	44	−75%
Predicted σ_y	1250	1030	880	620	−50%

Contribution	25 °C	600 °C	700 °C	800 °C	Trend
$\Delta\sigma_{ppt}$ as % of σ_y	34.6	36.1	40.7	48.9	rising
$\Delta\sigma_{HDI}$ as % of σ_y	14.0	16.8	13.2	7.0	falling
$\Delta\sigma_{HP}$ as % of σ_y	33.1	30.4	29.1	25.5	falling

25 C | FFFF|SSSS|HHHHHHHHHHHHHHHH|PPPPPPPPPPPPPPPP|DDDDDDDD| 1250
600 C | FFFF|SS|HHHHHHHHHHHHHH|PPPPPPPPPPPPPP|DDDDDDDD| 1030
700 C | FFF|SS|HHHHHHHHHHHH|PPPPPPPPPPPPPP|DDDD| 880
800 C | FFF|S|HHHHHH|PPPPPPPPPPPP|DD| 620

Fig. 5. Composition of the yield stress vs. temperature (each bar to scale; 1 character = 25 MPa).

F = lattice friction S = solid solution H = Hall-Petch
P = precipitation D = HDI (at yield)

CROSSOVER: precipitation overtakes ALL other terms combined at 800 C
(49% of σ_y vs. 35% at RT), while HDI collapses from 14% to 7%.

4.5 GND density and HDI evolution

KAM analysis and the HDI deconvolution give independent access to the GND field. Table 7 reports both. The as-processed GND density inferred from the HDI term at yield, $5.5 \times 10^{14} \text{ m}^{-2}$ at room temperature, lies within the range spanned directly by the KAM map, whose constituent-resolved values run from $5.8 \times 10^{14} \text{ m}^{-2}$ in the 119ecrystallized FCC grains to $4.4 \times 10^{15} \text{ m}^{-2}$ in the 100 nm bands adjacent to FCC/B2 interphase boundaries. At 5% plastic strain the hysteresis-derived HDI stress reaches 430 Mpa, corresponding to $3.3 \times 10^{15} \text{ m}^{-2}$. This six-fold dynamic multiplication of the GND population is the dynamic reinforcement mechanism identified in heterogeneous grain structures [30].

TABLE VII. GND DENSITIES AND HDI STRESS ACROSS THE TEMPERATURE RANGE.

Quantity	25 °C	600 °C	700 °C	800 °C
σ_{HDI} at yield (MPa)	175	174	116	44
ρ_{GND} at yield (m^{-2})	5.5×10^{14}	7.9×10^{14}	3.8×10^{14}	5.8×10^{13}
σ_{HDI} at $\epsilon_p = 5\%$ (MPa)	430	335	262	118
ρ_{GND} at $\epsilon_p = 5\%$ (m^{-2})	3.3×10^{15}	3.0×10^{15}	2.0×10^{15}	4.3×10^{14}
$\sigma_{HDI}/\sigma_{flow}$ at $\epsilon_p = 5\%$	0.31	0.29	0.27	0.18
GND multiplication factor (yield $\rightarrow 5\%$)	6.0	3.8	5.2	7.4

TABLE VIII KAM-DERIVED GND DENSITIES BY MICROSTRUCTURAL CONSTITUENT (50 NM STEP).

Constituent	KAM, as-aged (°)	ρ_{GND} , as-aged (m^{-2})	KAM, $\epsilon_p = 5\%$ (°)	ρ_{GND} , $\epsilon_p = 5\%$ (m^{-2})
Recrystallised FCC	0.11	3.0×10^{14}	0.21	5.8×10^{14}
Non-recrystallised FCC	0.42	1.2×10^{15}	0.82	2.3×10^{15}
B2 lamellae	0.28	7.7×10^{14}	0.55	1.5×10^{15}
FCC/B2 interface band ($\pm 100 \text{ nm}$)	0.80	2.2×10^{15}	1.60	4.4×10^{15}

4.6 Dislocation–precipitate interaction regimes

TABLE IX. OPERATIVE BYPASS MECHANISMS BY POPULATION AND TEMPERATURE.

Population	$\bar{r}$ (nm)	r_c (nm)	25 °C	600 °C	700 °C	800 °C
L1 ₂ in FCC	5.2	113	Weak-pair shearing, APB coupling	Shearing; planar faults extend	Shearing + incipient cross-slip	Shearing + local climb at interfaces
B2 in FCC	22.0	— (incoherent)	Orowan looping	Orowan looping	Looping + climb assistance	Climb-assisted bypass, detachment-controlled
α -Cr in B2	3.6	17	Modulus-mismatch shearing	Shearing	Shearing	Shearing; coarsening toward crossover

T (°C)	FCC CONSTITUENT	B2 CONSTITUENT
25	Planar slip on {111}<110> L1 ₂ sheared in pairs B2 particles looped Stacking faults extensive	Confined {110}<111> slip alpha-Cr sheared Slip transfer at K-S interfaces Limited cross-slip
600	Planar slip persists APB-coupled shearing Loops accumulate at B2 HDI near maximum	Slip more homogeneous alpha-Cr still sheared Dipole debris forms
700	Wavy slip begins Cross-slip relaxes pile-ups Climb bypass at B2 starts HDI falls 33%	Recovery at domain boundaries Dislocation climb onset alpha-Cr Orowan loops form near lamella interiors
800	Dynamic recovery dominant Climb + detachment control GND annihilation: HDI → 7% L1 ₂ still unshearable-free ($r \ll r_c$): strength held	Sub-boundary migration GB sliding ATTEMPTED but BLOCKED by intergranular B2 + alpha-Cr particles Threshold stress governs creep

Fig. 6. Deformation mechanism map for the HDP-CP architecture.

4.7 Creep behaviour

TABLE X. CREEP RESPONSE OF THE HDP-CP ALLOY, WITH THRESHOLD-STRESS ANALYSIS.

T (°C)	σ (MPa)	σ_{th} (MPa)	$\dot{\epsilon}_{min}$ (s ⁻¹)	t_r (h)	n_{app}	LMP ($\times 10^{-3}$)
700	220	150	4.5×10^{-9}	3094	15.7	22.86
700	250	150	2.7×10^{-8}	520	12.5	22.10
700	280	150	9.9×10^{-8}	140	10.8	21.55
700	310	150	2.8×10^{-7}	50	9.7	21.11
750	170	105	1.7×10^{-8}	801	13.1	23.43
750	200	105	1.2×10^{-7}	120	10.5	22.59
750	230	105	4.6×10^{-7}	30	9.2	21.98
800	100	60	7.3×10^{-9}	1904	12.5	24.98
800	120	60	5.5×10^{-8}	251	10.0	24.03
800	140	60	2.3×10^{-7}	60	8.8	23.36
800	160	60	7.1×10^{-7}	20	8.0	22.84

LMP is the Larson–Miller parameter, $LMP = T(20 + \log_{10} t_r)$ with T in K and t_r in h.

TABLE XI. COMPARATIVE CREEP RUPTURE LIFE.

Condition	SP-FCC	DP-Ref	HDP-CP	Improvement over DP-Ref
600 °C / 350 MPa	below σ_y : fails on loading	575 h	2.9×10^4 h (extrapolated)	$\approx 50\times$
700 °C / 250 MPa	fails on loading	23 h	520 h	22 $\times$
800 °C / 120 MPa	fails on loading	21 h	251 h	12 $\times$

The apparent stress exponents of 8–16 have no mechanistic meaning taken at face value. Subtracting the threshold stress collapses the entire dataset onto a single line of slope $n = 5$, the classical exponent for dislocation-climb-controlled creep. The same arithmetic explains the apparent activation energies: evaluated at constant applied stress, $Q_{app} = 592 \text{ kJ mol}^{-1}$ between 700 and 750 °C at 250 MPa and 765 kJ mol^{-1} between 750 and 800 °C at 170 MPa, both roughly two to three times the true $Q_c = 285 \text{ kJ mol}^{-1}$. The excess is contributed entirely by the temperature dependence of σ_{th} , and any analysis that ignores it will misidentify the rate-controlling mechanism.

5. DISCUSSION

5.1 Coarsening kinetics and the origin of thermal stability

Any strength stored in nanoprecipitates is provisional: it lasts only as long as the particles do. Diffusion-controlled coarsening in the Lifshitz–Slyozov–Wagner formulation [41], [42] gives the mean radius as

$$\bar{r}^3(t) - \bar{r}_0^3 = K t, \quad K = \frac{8 \gamma_{int} D C_e V_m^2}{9 R T}$$

so that K carries the interfacial energy γ_{int} , the solute diffusivity D, the equilibrium matrix solubility C_e and the molar volume V_m . Its temperature dependence follows $K(T) \propto T^{-1} \exp(-Q_c/RT)$.

TABLE XII. COARSENING CONSTANTS AND PROJECTED MEAN RADII (NM).

Population	Q_c (kJ mol ⁻¹)	K at 700 °C (m ³ s ⁻¹)	K at 800 °C (m ³ s ⁻¹)	$\bar{r}$: 700 °C/1000 h	$\bar{r}$: 800 °C/100 h	$\bar{r}$: 800 °C/1000 h
L ₁₂ in FCC	254	2.7×10^{-31}	4.6×10^{-30}	10.4	12.2	25.6
B2 in FCC	270	7.4×10^{-31}	1.5×10^{-29}	23.7	25.2	40.1
α -Cr in B2	260	1.7×10^{-31}	3.0×10^{-30}	8.6	10.4	22.1

Three features of this table carry the argument.

First, the L₁₂ population grows from 5.2 to 25.6 nm after 1000 h at 800 °C, yet $r_c = 113$ nm: the particles remain shearable throughout, and because weak-pair order strengthening at fixed volume fraction is radius-independent, the FCC constituent's dominant precipitation term is structurally immune to this amount of coarsening. This is the single most important design consequence of choosing a high-APB-energy L₁₂ phase at small radius rather than a larger, marginally shearable one. Ti is essential here, both because it raises γ_{APB} and because its low solubility in the FCC matrix suppresses C_e and therefore K.

Second, the α -Cr population is deliberately under-aged relative to its own shearing-to-looping crossover at 17 nm. Coarsening therefore moves it *toward* its strength maximum before moving past it. The consequence is the mildly non-monotonic thermal-exposure response in Table 13: the 800 °C yield strength rises by 5% after 100 h and then falls, ending 1000 h of exposure at 97% of the as-aged value. Under-ageing a coherent population whose obstacle strength increases with radius converts early coarsening from a liability into a reserve.

Third, the B2 population in the FCC constituent is the weak link: it is already in the looping regime, so coarsening reduces its Orowan resistance monotonically, from 273 to 168 MPa as $\bar{r}$ grows from 22 to 40 nm. Its 2.5 vol.% share limits the damage. A design variant that reduced the B2 particle size at constant fraction would be superior on strength grounds, at the cost of losing the intergranular pinning discussed in Section 5.3.

TABLE XIII. YIELD STRENGTH AT 800 °C AFTER PRIOR THERMAL EXPOSURE AT 800 °C.

Exposure	$\bar{r}$ B2 (nm)	$\bar{r}$ α -Cr (nm)	$\Delta\sigma_{ppt}$ FCC (MPa)	$\Delta\sigma_{ppt}$ B2 (MPa)	σ_y at 800 °C (MPa)	Retention
As-aged	22.0	3.6	173	130	620	100%
100 h	25.2	10.4	166	174	651	105%
1000 h	40.1	22.1	148	151	603	97%

Two caveats on the coarsening analysis deserve emphasis. LSW theory assumes a dilute dispersion, negligible elastic interaction and a single population; at 14 vol.% and with three interacting populations, none of these strictly holds, and the real coarsening rate will be modified by solute competition between the L₁₂ and B2 populations for Al and Ti. The co-precipitation literature in steels suggests this competition is retarding rather than accelerating [15], so the projections here are more likely conservative than optimistic, but that expectation requires experimental test rather than assertion. Long-term phase stability is a separate question again: the CrMnFeCoNi precedent shows that HEA microstructures considered stable over laboratory timescales can decompose over hundreds of days [5], and 1000 h projections should not be extrapolated to service lives of 10^4 – 10^5 h without direct measurement.

5.2 The temperature-driven hand-over from HDI to Orowan strengthening

The deconvolution in Table 6 exposes a hand-over that is invisible in a room-temperature measurement. Between 25 and 800 °C, the HDI contribution falls by 75% and the Hall–Petch contribution by 62%, while the precipitation contribution falls by only 30%. The relative shares invert accordingly: precipitation rises from 35% to 49% of the yield stress while HDI falls from 14% to 7%.

The mechanism is specific. HDI stress requires a *retained* GND population, because the back stress derives from the polarised dislocation content piled against domain boundaries. At 800 °C the recovery rate is sufficient for climb-mediated annihilation of GNDs to compete with their storage, and the density at yield drops to 5.8×10^{13} m⁻², an order of magnitude below the room-temperature value (Table 7). Strain gradients are still imposed by the plastic incompatibility between FCC and B2, since the architecture has not changed, but the dislocation content that would express them as internal stress no longer survives. Boundary strengthening decays for the same underlying reason, through boundary migration and sub-boundary recovery.

Precipitation strengthening is structurally different. An Orowan or order-strengthening obstacle is an object, not a stored defect. Its resistance falls only through the shear modulus and through slow coarsening; it cannot be annihilated by recovery. Strength stored in defects is removed by recovery; strength stored in objects is removed only by diffusion. That distinction is the physical content of the hand-over and the design rule this work most wants to assert.

Two corollaries follow. Any dual-phase HEA whose high-temperature strength strategy rests principally on heterostructure engineering will fail above roughly 0.55 T_m , because the mechanism is intrinsically recovery-limited; the prediction discriminates against a substantial body of otherwise successful room-temperature designs [30]–[32]. Conversely, the HDI mechanism remains valuable at high temperature for a different reason than at ambient temperature, because the same

recovery that destroys HDI stress at yield also restores strain-hardening capacity, which is why the 800 °C uniform elongation of 16.5% exceeds the 700 °C value of 13.2% (Table 5) despite the lower strength. HDI hardening is best understood at elevated temperature as a *ductility* mechanism, not a strength mechanism.

5.3 Threshold stress, detachment control and the suppression of boundary sliding

The creep results of Table 10 are governed by the threshold stress, which falls from 250 MPa at 600 °C to 60 MPa at 800 °C. Interpreting it through the detachment criterion of Section 3.7 gives relaxation parameters of $k_R = 0.72, 0.87$ and 0.96 at 700, 750 and 800 °C, against an Orowan stress that falls only from 216 to 208 MPa over the same interval. The threshold therefore collapses far faster than the athermal obstacle strength, and the reason is interfacial: as temperature rises, vacancy transport along the particle–matrix interface relaxes the dislocation line profile that produces the attractive interaction, and the detachment barrier disappears while the particle itself remains fully effective against glide. This is the behaviour anticipated by Arzt and Rösler [39], [40], and it has a direct practical consequence: an alloy can retain most of its tensile yield strength while losing most of its creep threshold, so tensile screening at temperature is not a valid proxy for creep ranking.

The second creep-relevant role of the precipitate architecture concerns grain-boundary sliding. At $T/T_m = 0.63$ and grain sizes below 1 μm , sliding would ordinarily be a dominant deformation path; ultrafine-grained materials are notoriously creep-weak for exactly this reason, and it is the standard objection to using a heterostructured, partially recrystallised microstructure at high temperature. The architecture answers the objection through the placement of the coarse B2 population. Because these particles nucleate preferentially on FCC/B2 interphase boundaries and on FCC sub-boundaries during the 800 °C ageing step, a substantial fraction sits directly on the sliding planes. A boundary attempting to slide must either shear the particles, which requires the full Orowan stress, or migrate around them against Zener drag. The $\alpha\text{-Cr}$ particles inside the B2 lamellae perform the analogous function for sub-boundary migration. The evidence in the present dataset is indirect but specific. The apparent activation energies of 592–765 kJ mol^{-1} (Section 4.7) are far above the range associated with boundary-diffusion-controlled sliding, and the stress exponent after threshold subtraction is 5, the lattice-diffusion-controlled climb value, not the exponent near 2 characteristic of sliding-dominated flow. Direct confirmation would require marker-line experiments, which the present framework does not supply.

5.4 Why ductility is retained

At 1250 MPa yield strength, the uniform elongation of 17.8% is the result that most requires explanation, since ultrafine-grained materials of this strength typically exhaust their hardening capacity within 2–3% strain [30]. Three reservoirs contribute. The bimodal FCC grain population supplies a dynamically reinforced HDI stress that grows from 175 to 430 MPa between yield and 5% plastic strain, adding hardening exactly where a homogeneous ultrafine structure would have none. The sheared L_{12} particles promote planar slip and stacking-fault extension rather than cell formation, delaying the onset of the dynamic-recovery-dominated regime, in agreement with the planar-fault mechanism identified by Zhao and co-workers in L_{12} -strengthened HEAs at elevated temperature [12]. The looped B2 particles accumulate Orowan loops that constitute a forest-hardening reservoir independent of both. The three reservoirs saturate at different strains, which is the mechanistic content of the observation that a heterogeneous obstacle spectrum sustains hardening in a very strong alloy better than a single optimised obstacle does [33], [34].

5.5 Limitations

The framework has four boundaries that should be stated plainly. First, as the note on data provenance records, the property matrices are model predictions anchored to literature inputs; they identify which mechanisms should dominate and in what proportion, and they are falsifiable, but they are not measurements. Second, the iso-strain rule of mixtures at yield neglects load transfer between FCC and B2 constituents and will overestimate the composite yield stress where the constituents' flow stresses differ markedly, which they do at 800 °C. Third, oxidation is absent from the analysis entirely; a Cr-bearing alloy at 800 °C in air forms a scale whose growth and spallation may govern component life long before creep does, and the $\alpha\text{-Cr}$ population represents Cr locked away from that scale. Fourth, the model treats each precipitate population as monodisperse; real size distributions broaden during coarsening, and the largest particles in the L_{12} distribution will reach the looping regime long before the mean does.

6. CONCLUSIONS AND FUTURE DIRECTIONS

6.1 Conclusions

1. A dual-phase FCC/B2 high-entropy alloy of composition $\text{Ni}_{34.4}\text{Co}_{16.4}\text{Cr}_{16.4}\text{Fe}_{16.4}\text{Al}_{14.4}\text{Ti}_{2.0}$ can host three distinct nanoprecipitate populations simultaneously: coherent $\text{L}_{12}(\text{Ni},\text{Co})_3(\text{Al},\text{Ti})$ at 5.2 nm and semi-coherent B2 (Ni,Fe)Al at 22 nm within the FCC constituent, and coherent $\alpha\text{-Cr}$ at 3.6 nm within the B2 lamellae, for a total precipitate fraction of 14.9 vol.%, when processed by cold rolling, intercritical annealing and descending two-step ageing.

2. The resulting architecture is predicted to yield at 1250 MPa at room temperature and at 1030, 880 and 620 MPa at 600, 700 and 800 °C, retaining 50% of its room-temperature yield strength at 800 °C against 32% for a conventional lamellar dual-phase alloy, while sustaining uniform elongations of 13–18% throughout.
3. The composition of the yield stress shifts systematically with temperature. Precipitation strengthening rises from 35% to 49% of the total between 25 and 800 °C, while HDI hardening falls from 14% to 7% and boundary strengthening from 33% to 26%. Strength stored in objects survives; strength stored in retained dislocation content does not.
4. The L1₂ population is the anchor of high-temperature strength because its radius, 5.2 nm as-aged and 25.6 nm after 1000 h at 800 °C, remains far below the shearing-to-looping critical radius of 113 nm, within which weak-pair order strengthening is independent of particle radius at fixed volume fraction.
5. Under-ageing the α -Cr population relative to its own 17 nm crossover radius converts early coarsening into a strength reserve: the predicted 800 °C yield strength rises 5% after 100 h of exposure before declining, ending at 97% of the as-aged value after 1000 h.
6. Creep rupture life reaches 520 h at 700 °C/250 MPa and 251 h at 800 °C/120 MPa, improvements of 22-fold and 12-fold over the conventional lamellar reference. Apparent stress exponents of 8–16 and apparent activation energies of 592–765 kJ mol⁻¹ are artefacts of a temperature-dependent threshold stress; after its subtraction the data collapse to $n = 5$ and $Q_c = 285$ kJ mol⁻¹, identifying dislocation climb followed by attractive detachment as the rate-controlling sequence.
7. Intergranular placement of the coarse B2 and α -Cr particles is what permits a submicrometre heterostructure to be used at 0.63 T_m without succumbing to grain-boundary sliding, since a sliding boundary must either shear the particles or migrate against their Zener drag.

6.2 Design rules

For elevated-temperature dual-phase HEA design, the analysis supports four transferable rules:

- Place a precipitate population in every constituent. A hard second phase used only as a passive load-bearing medium wastes its own precipitation potential; the B2 lamellae here contribute 130–201 MPa to the alloy yield stress through α -Cr alone.
- Select particle radius well below the shearing-to-looping crossover, and raise the APB energy instead of the particle size. Strength in the shearing regime at fixed volume fraction is radius-independent and therefore coarsening-tolerant; strength in the looping regime is not.
- Under-age coherent populations whose obstacle strength increases with radius. The service environment will complete the ageing.
- Do not rely on heterostructure engineering above 0.55 T_m for strength, but retain it for ductility. HDI hardening is recovery-limited as a strength mechanism and recovery-enabled as a hardening-capacity mechanism.

6.3 Future directions

- Experimental validation is the immediate priority, in the order in which the framework is most vulnerable: APT verification of three-population co-precipitation and of the Ti partitioning coefficient of 10.1; long-term coarsening measurement at 800 °C to 3000 h to test the LSW projections under 14 vol.% loading with solute competition; and creep testing across at least three stresses per temperature so that the threshold stress can be extracted independently rather than assumed.
- Additive manufacturing of heterostructured HEAs offers a route to architectures that thermomechanical processing cannot reach. Laser powder-bed fusion imposes cooling rates of 105–107 K s⁻¹, producing cellular solidification structures with intrinsic chemical heterogeneity at the sub-micrometre scale, and eutectic HEAs are among the most printable multi-principal-element systems because of their narrow solidification range. The open question is whether the as-built cellular heterogeneity can be preserved through the ageing treatment that the co-precipitation requires, or whether the two are thermodynamically incompatible.
- Machine-learning-guided precipitate discovery is best directed not at composition screening, for which thermodynamic databases already suffice, but at the quantities that control the design rules above and that are expensive to compute or measure: APB energies of multicomponent L12 phases, interfacial energies governing K, and the relaxation parameter kR that sets the creep threshold. These are precisely the inputs whose uncertainty dominates the present predictions.
- Extreme-environment extension requires coupling the mechanical framework to oxidation and irradiation response. The α -Cr population sequesters Cr from the protective scale, a direct and quantifiable conflict between creep design and oxidation design that this work has not resolved. Under irradiation, the high sink density of a three-population dispersion is expected to suppress void swelling, and co-precipitated steels show exactly this behaviour, but the stability of coherent L12 under cascade damage in a concentrated multicomponent matrix remains unestablished.

Funding:

The authors declare that no specific financial aid or sponsorship was received from governmental, private, or commercial entities to support this study. The research was solely financed by the authors' own contributions.

Conflicts of Interest:

The authors declare that there are no conflicts of interest in this study.

Acknowledgment:

The authors express their heartfelt appreciation to their institutions for the essential support and motivation provided throughout the research period.

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7. APPENDIX A. MODEL INPUTS AND REPRODUCIBILITY

All results in Sections 3–5 was computed from closed-form expressions given in the text, implemented in three short scripts (composition and mass-balance screening; strength deconvolution; creep and coarsening projection). The complete input set is:

Symbol	Value	Source / basis
$G_0^{\text{FCC}}, G_0^{\text{B2}}$	77, 70 GPa	FCC HEA and B2 aluminide literature values
$b_{\text{FCC}}, b_{\text{B2}}$	0.254, 0.249 nm	Lattice parameters of the respective phases
$M_{\text{FCC}}, M_{\text{B2}}$	3.06, 2.75	Random-texture Taylor factors
ν	0.30	—
T_m	1630 K	Solidus, duplex composition
$\gamma_{\text{APB}} (\text{L1}_2)$	0.12 J m^{-2}	Ti-bearing L1 ₂ range
$\delta_{\text{misfit}} (\text{L1}_2/\text{FCC})$	+0.18%	Coherent precipitation requirement
$\Delta G (\alpha\text{-Cr} / \text{B2})$	45 GPa	Modulus mismatch, BCC Cr vs. NiAl
$k_{\text{HP}}^{\text{FCC}}, k_{\text{HP}}^{\text{B2}}$	494, 210 MPa $\mu\text{m}^{1/2}$	FCC HEA Hall–Petch slopes [37], [38]
α (Taylor, HDI)	0.25	Reduced for dipolar GND fraction
ΔE_b (solute barrier)	2.5 eV	Thermally activated solute bypass
n, Q_c (creep)	5, 285 kJ mol ^{−1}	Lattice-diffusion-controlled climb
C_{MG}	0.05	Monkman–Grant constant
$\phi_{\text{GB}}(T)$	1.00 / 0.92 / 0.78 / 0.50	Boundary recovery factor at 25/600/700/800 °C

Mass-balance closure: the phase compositions of Table IV, weighted by the phase and precipitate fractions of Tables II and III, reproduce the nominal composition to within 0.15 at.% for every element. Any substitution of these inputs propagates transparently through the equations of Section 3.