

# Microstructural Evolution of INCONEL X-750 under Additive Manufacturing, Thermal Processing, and Irradiation Environments: A Comprehensive Review

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## Abstract

INCONEL® X-750 (UNS N07750) is a precipitation-hardenable Ni–Cr superalloy strengthened primarily by  $\gamma'$  ( $\text{Ni}_3[\text{Al,Ti}]$ ) and, depending on thermal history, by secondary phases such as  $\eta$ - $\text{Ni}_3\text{Ti}$  and  $\gamma''$ /carbides. Its long-standing roles in aerospace hot-section hardware and nuclear reactor internals rely on a careful balance between high-temperature strength/relaxation resistance, oxidation/corrosion tolerance, and SCC mitigation through microstructural control. Conventional wisdom—codified in AMS and vendor heat-treat schedules—was developed for wrought materials; however, the recent adoption of laser powder bed fusion (PBF-LB) and renewed scrutiny under irradiation challenge many assumptions. This review synthesizes 35+ sources to connect three domains often studied separately: (i) AM-induced microsegregation and defect structures, (ii) irradiation-driven  $\gamma'$  disordering/dissolution and grain-boundary phenomena, and (iii) advanced heat-treatment pathways (solution, stabilization, aging, homogenization) from AMS and post-AM strategies. We show how AM-specific precipitate pathways (including anomalous  $\gamma'$  formation during “solution” and  $\eta$  promotion) interact with irradiation temperature/dose windows to determine  $\gamma'$  stability, hardness, and SCC susceptibility; we map design-relevant process–structure–property linkages and propose tailored post-AM thermal schedules and PWHT guidelines for welded and AM parts destined for high-temperature or LWR environments. **Keywords:** INCONEL X-750; UNS N07750;  $\gamma'$ ;  $\eta$ - $\text{Ni}_3\text{Ti}$ ; additive manufacturing; PBF-LB; irradiation; SCC; AMS 5667/5668/5670; hydrogen water chemistry (HWC)

## 1. Introduction

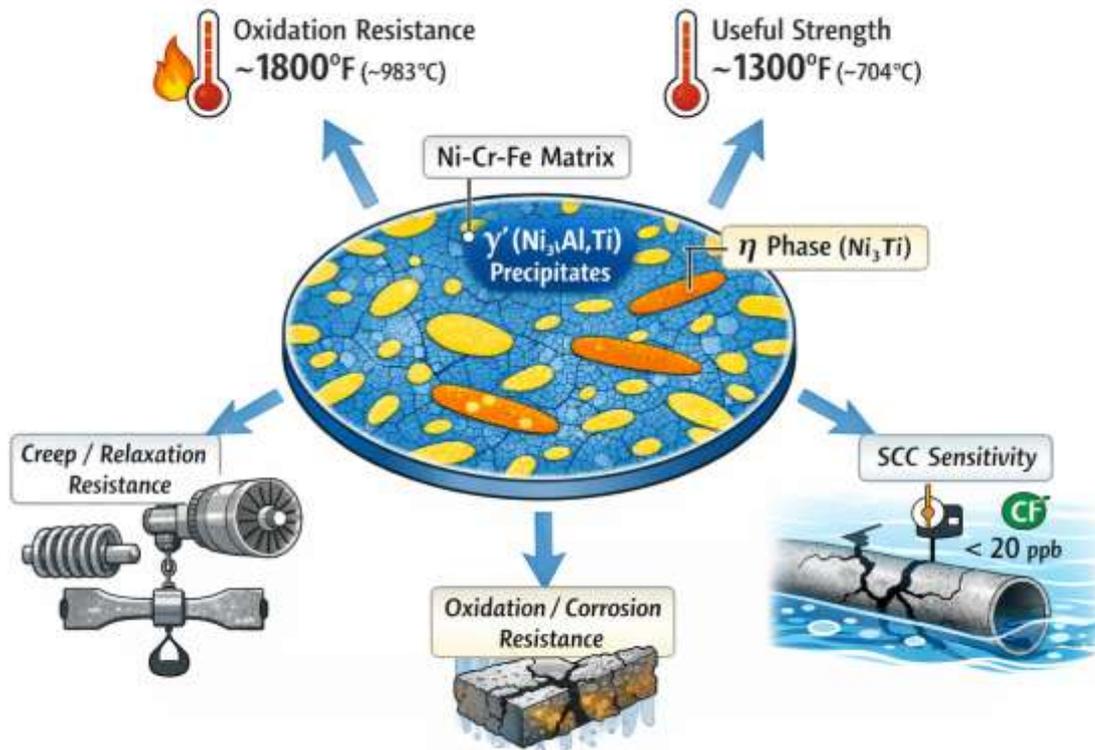
INCONEL X-750 is a Ni–Cr superalloy (Ni+Co  $\geq$ 70%, Cr 14–17%, Fe 5–9%, Ti 2.25–2.75%, Al 0.4–1.0%, Nb+Ta 0.7–1.2%) that derives its elevated-temperature strength from  $\gamma'$  precipitation and exhibits oxidation resistance to  $\sim$ 1800 °F ( $\approx$ 983 °C) with useful strength to  $\sim$ 1300 °F ( $\approx$ 704 °C). These attributes underpin applications from gas-turbine rotors, airframe hot ducting, and thrust reversers to nuclear internals, springs, and fasteners where relaxation resistance is crucial.

Historically, heat-treat windows have been tightly coupled to service regime: (a) “triple” solution ( $\approx$ 2100 °F) + stabilization ( $\approx$ 1550 °F) + age ( $\approx$ 1300 °F) for creep/relaxation  $\geq$ 1100 °F, and (b) equalize ( $\approx$ 1625 °F) + age ( $\approx$ 1300 °F) for maximum room-temperature strength/ductility. These routes are codified across AMS 5667/5668/5670/5671 and vendor data sheets.

Three disruptive developments now motivate a unified, cross-domain review:

1. Additive Manufacturing (PBF-LB): Conventional AMS schedules can misfire in AM X-750 due to segregation, high dislocation density, and cellular/dendritic substructures, giving rise to  $\gamma'$  precipitation during “solution” and  $\eta$ -phase promotion—contrary to wrought behavior—thus requiring AM-specific homogenization/aging designs.
2. Irradiation (LWR-relevant): In-situ ion irradiation studies reveal  $\gamma'$  disordering/dissolution at 333–673 K at very low doses ( $\sim$ 0.06 dpa) and temperature-dependent stability  $\geq$ 773 K, while reactor-relevant experiments show limited effect of irradiation on SCC propagation rates under certain chemistries, complicating microstructure–performance maps.

3. Service environment coupling (BWR/PWR): SCC depends sensitively on electrochemical potential, impurity ppb ( $\text{Cl}^-$ ), heat treatment/microstructure, and loading; HWC can reduce crack growth rates by ~one order of magnitude at 288 °C, while  $\text{Cl}^-$  must be kept below ~20 ppb to avoid SCC in high-T water.

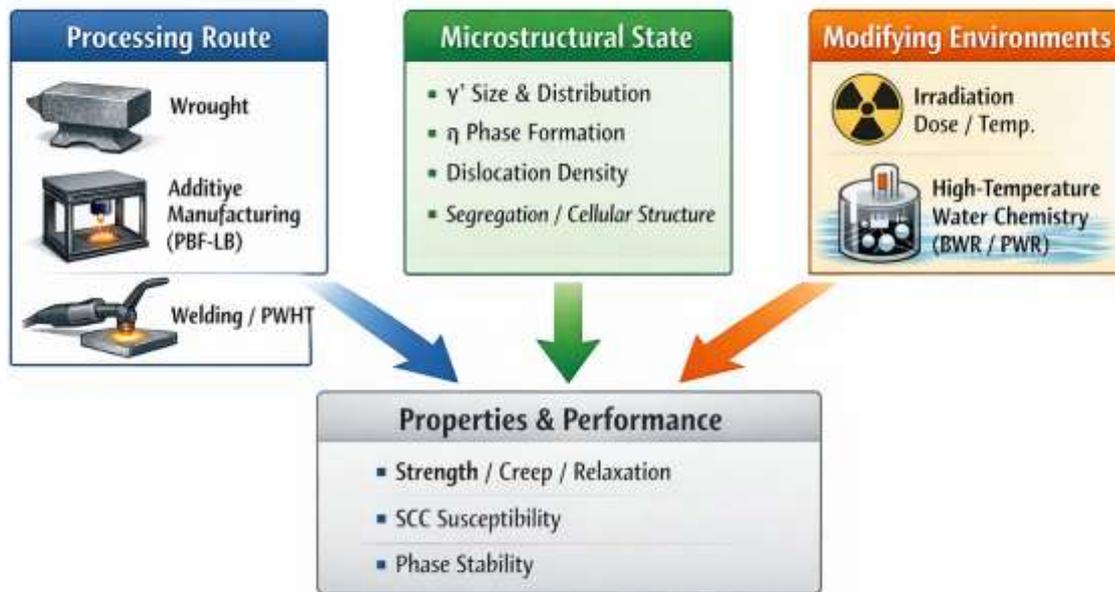


This review integrates these threads to offer design-ready guidance for modern X-750 components.

## 2. Methods: Literature Search and Scope

This review emphasizes establishing clear microstructure–property relationships across three critical domains: (i) additively manufactured versus wrought processing, (ii) irradiation-induced microstructural evolution, and (iii) heat-treatment and post-weld heat-treatment (PWHT) conditions. Literature was evaluated with priority given to studies that explicitly link processing history and environmental exposure to phase stability, deformation mechanisms, corrosion or SCC behavior, and service-relevant performance.

3. Fundamentals: Chemistry, Phases, and Classical Heat Treatments.



## 2.1 Alloy chemistry, phases, and strengthening

The chemistry of INCONEL X-750 is tailored to promote  $\gamma'$  ( $\text{Ni}_3[\text{Al},\text{Ti}]$ ) precipitation as the primary strengthening mechanism. Minor additions of Nb and Ta can contribute to secondary precipitation reactions, including  $\gamma''$  and  $\eta$  phases, depending on thermal history. Grain-boundary carbide formation, predominantly  $\text{Cr}_{23}\text{C}_6$ , plays a critical role in balancing creep resistance and susceptibility to stress corrosion cracking. The alloy exhibits good oxidation and general corrosion resistance, and vendor data consistently report excellent relaxation resistance in springs and fasteners over a wide temperature range, extending from cryogenic conditions to approximately 1200 °F.[4]

## 2.2 Heat-treat windows and property trade-offs

For long-term service above approximately 1100 °F ( $\approx 593$  °C), a three-step heat-treatment sequence consisting of solution treatment near 2100 °F, stabilization at  $\sim 1550$  °F, and aging at  $\sim 1300$  °F is widely recognized to maximize creep and stress-relaxation resistance. In contrast, for service below  $\sim 1100$  °F, a two-step treatment involving stress equalization at  $\sim 1625$  °F followed by aging at  $\sim 1300$  °F is preferred to achieve high yield strength and tensile ductility. These regime-specific heat-treatment strategies are consistently codified in AMS specifications (AMS 5667, 5668, 5670, and 5671) and are corroborated by multiple vendor data sets, which collectively demonstrate convergence toward distinct optima for high-temperature versus intermediate-temperature service.

## 3. Additive Manufacturing (PBF-LB): From “Inherited” Schedules to AM-Specific Thermal Design

### 3.1 AM microstructure: why conventional schedules fail

PBF-LB X-750 exhibits segregation-rich cellular/dendritic structures, high dislocation density, and texture/anisotropy. During nominal standard “solution + stabilization + age”, AM materials show  $\gamma'$  precipitation already during solution,  $\eta$ - $\text{Ni}_3\text{Ti}$  formation on stabilization, coarse precipitates, and anomalous hardness increases—signaling insufficient homogenization and different kinetics vs wrought.

### 3.2 Post-AM thermal pathways: homogenize first, then age

Studies systematically screening >70 post-AM schedules recommend elevated-T homogenization/solution to reduce segregation, promote recrystallization, and suppress  $\eta$ , followed by lower-T aging for fine  $\gamma'$ . The sequence and ramp/cooling controls are critical to avoid  $\eta$  plates and to regain ductility without sacrificing high-T strength.

### 3.3 AM feedstock and quality levers

AM powder suppliers stress spherical, low-oxygen powders for flowability/consistency and recommend post-build heat treatments tailored to X-750's precipitation response; industrial notes highlight AM springs/fasteners/brackets as emerging applications once microstructure is stabilized. Design implication. For AM X-750 intended for 700–815 °C service, prioritize (i) homogenization (high-T/long-hold), (ii) controlled cooling, (iii) aging to generate fine, uniform  $\gamma'$  while avoiding  $\eta$ ; verify via hardness/microhardness, EBSD, APT/TEM.

## 4. Irradiation Effects: $\gamma'$ Stability Windows and Mechanical Impacts

In-situ heavy-ion TEM shows  $\gamma'$  disordering/dissolution at 333–673 K even at  $\sim 0.06$  dpa, whereas  $\geq 773$  K preserves order up to  $\sim 5.4$  dpa; balancing disordering vs thermal recovery defines a  $\gamma'$  stability window and underscores Ti/Al ratio sensitivity. In reactor-relevant programs (INL), X-750 irradiated at  $\sim 288$  °C exhibited changes in tensile/toughness yet no significant acceleration of SCC crack growth rates compared with unirradiated material under equivalent water chemistries—an important nuance when projecting service life for irradiated springs/bolts. Design implication. For BWR-core adjacent components, operating temperature near  $\sim 560$ – $600$  K ( $\approx 287$ – $327$  °C) intersects the sensitive regime for  $\gamma'$  order/disorder; thus chemistry control and microstructure (GB carbides,  $\gamma'$  size) remain decisive for SCC, while irradiation may not dominate CGR in all cases.

## 5. Stress Corrosion Cracking (SCC) in LWR Environments: Chemistry–Microstructure Coupling

### 5.1 Chemistry control: HWC vs NWC and impurity limits

Switching from NWC to HWC reduces CGR by  $\sim 1$  order of magnitude at 288 °C ( $K \approx 28$  MPa $\sqrt{m}$ ), with fracture transitioning from transgranular under fatigue to (pseudo-)intergranular under constant K SCC; at 93 °C under HWC, no growth was observed at tested K. Chloride control is critical: even  $< 200$  ppb affects SCC; immunity requires  $< \sim 20$  ppb in high-T water.

### 5.2 CGR databases and mechanistic models

A 274-point database across heats/heat-treats/orientations analyzed CGR vs potential, purity, KI, periodic unloading; models accounting for Ni/NiO line capture activation energies ( $\sim 134$  kJ/mol) and reconcile BWR vs PWR crack-tip conditions.

### 5.3 Microstructure/heat-treat dependence

Slow-strain-rate tests indicate best IGSCC resistance after  $\sim 704$  °C/20 h aging (GB  $Cr_{23}C_6$  enrichment), while 885 °C/24 h aging degraded resistance;  $Cl^-$  is more aggressive than  $SO_4^{2-}$  in otherwise similar conditions. Design implication. For LWR service, select HTH/AH microstructures with GB carbide coverage (but avoiding continuous films) and maintain HWC (low electrochemical potential) while holding  $Cl^- < \sim 20$  ppb.

## 6. Welding and Post-Weld Heat Treatment (PWHT): HAZ Microstructure and $\eta$ -Phase Risks

X-750's precipitation-hardenable nature raises hot-cracking and HAZ softening/overaging concerns; PWHT is generally required to restore  $\gamma'$  strengthening. A factorial PWHT study (705–845 °C, 2–24 h) showed longer aging  $\rightarrow$  coarser  $\gamma'$ , fewer GB  $Cr_{23}C_6$ , reduced hardness, implying a sweet spot for precipitate size/distribution and GB chemistry.

Process guidance: GTAW/GMAW with ERNiCr family fillers, strict cleanliness and interpass control, and avoidance of autogenous dissimilar joints (documented poor properties) unless fully qualified via mock-ups/PQR. Vendor notes caution that welding in an aged condition is feasible only if subsequent high-T exposures are controlled.

Design implication. For welding AM or wrought X-750, specify filler-assisted procedures, PWHT tuned to restore fine  $\gamma'$  without promoting  $\eta$ ; validate with hardness maps, EBSD/TEM, and relaxation tests for spring components.

## 7. High-Temperature Performance: Creep, Relaxation, and Fatigue Windows

Datasheets and application notes agree that X-750, properly aged, delivers low creep rates and high stress-rupture strength up to  $\sim 816$  °C, with relaxation resistance up to  $\sim 704$  °C underpinning use in springs/fasteners. Property targets for bars (aged) typically reach UTS 160–170 ksi, YS 100–115 ksi, with oxidation resistance to  $\sim 983$  °C; AMS treatment choice must track the intended temperature window.

## 8. Toward a Unified Process–Structure–Property Map for Modern X-750

### 8.1 Synthesis of interactions

- AM → Segregation/Defects → Anomalous  $\gamma'/\eta$  kinetics → Requires homogenization + tailored aging to match wrought-like  $\gamma'$  and suppress  $\eta$ .
- Irradiation (333–773 K) →  $\gamma'$  order/disorder competition; at  $\sim 288$  °C, irradiation influences tensile/toughness but does not necessarily increase CGR under controlled chemistry.
- Environment (BWR/PWR) → Potential +  $\text{Cl}^-$  (ppb) + GB carbides govern SCC; HWC measurably lowers CGR.

### 8.2 A design decision tree (textual)

#### 1. Manufacturing route

- a. Wrought/Cast: Apply AMS 5667/5668/5670 per service window.
- b. PBF-LB: Homogenize/solution at higher T + controlled cool → stabilize only if needed → lower-T aging to refine  $\gamma'$  and block  $\eta$ . Validate via TEM/APT.

#### 2. Service environment

- a. High-T air/steam: Prioritize creep/relaxation; choose triple-treat for  $\geq 1100$  °F long-term.
- b. LWR water: Target GB carbide topology (704 °C/20 h-type aging), HWC, and  $\text{Cl}^- < \sim 20$  ppb.

#### 3. Welded joints

- a. Use ERNiCr fillers, minimize restraint, PWHT to restore  $\gamma'$  and avoid  $\eta$ ; qualify via mock-ups.

## 9. Research Gaps and Future Directions

1. AM-specific homogenization maps for X-750 that couple time–temperature–transformation (TTT) with segregation length scales to reliably suppress  $\eta$  and stabilize fine  $\gamma'$  without over-aging.
2. Irradiation–precipitation coupling models that integrate  $\gamma'$  disordering kinetics with chemical partitioning from AM to predict long-term strength/SCC.
3. Data-driven PWHT design (e.g., Bayesian/ML) for welds and AM parts using multi-modal inputs (EBSD/APT/SECM/CGR) to optimize GB carbide topology +  $\gamma'$  size distribution.
4. Unified SCC predictors fusing APT-measured chemistry, electrochemical potential relative to Ni/NiO, and mechanical loading histories across heats/heat-treat

## 10. Conclusions

This review bridges three active frontiers for INCONEL X-750—AM microstructures, irradiation effects, and advanced heat treatments—to present an integrated framework for microstructure engineering. The key outcome is the recognition that AM X-750 cannot be post-processed by “copy-pasting” wrought AMS schedules; instead, homogenization-first

strategies are needed to erase AM segregation, prevent  $\eta$ , and stabilize fine  $\gamma'$ , which then dovetails with service-specific requirements (creep/relaxation or SCC). Under irradiation near 288 °C,  $\gamma'$  stability is marginal yet CGR may not be drastically affected if chemistry and potential are properly controlled; hence HWC and ppb-level  $\text{Cl}^-$  limits are essential. For welded structures, filler-assisted processes plus PWHT tuned to  $\gamma'$ /GB-carbide balance remain indispensable. These insights transform X-750 from a “legacy” alloy into a platform for high-reliability, modern manufacturing across aerospace and nuclear sectors.

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