

## Influence of Atmospheric Environments on Spark Plasma Sintering Kinetics, and Microstructural Evolution in Inconel 718 and 316 Stainless Steel Alloys

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### 1. Introduction

Inconel 718 (IN718), a precipitation-hardened Ni-Cr-Fe-Ti superalloy, and 316L austenitic stainless steel are both established structural materials in nuclear power systems. IN718 is used for reactor internals subjected to elevated temperature and stress, including bolting, springs, and control-rod components in light-water reactors, and is under active consideration for structural applications in Generation IV reactor concepts due to its retention of tensile strength and creep resistance at reactor operating temperatures [1]. This performance derives from a matrix uniformly strengthened by coherent FCC  $\gamma'$  [Ni<sub>3</sub>(Al,Ti)] and metastable BCT  $\gamma''$  (Ni<sub>3</sub>Nb) precipitates formed via post-heat treatment [2]. 316L, by contrast, is a primary structural material for reactor pressure boundary components, piping, and internals in both light-water and fast breeder reactors, owing to its corrosion resistance and mechanical reliability under prolonged operational exposure [3]. The combined use of these two alloys is increasingly necessary for Generation IV reactor designs and other harsh-environment industrial applications requiring transition joints between high-temperature Ni-superalloy components and stainless steel structural elements [4].

However, joining IN718 to 316L by conventional fusion-based methods remains problematic. During solidification of IN718-containing welds and joints, slow diffusivity of Nb concentrates it into interdendritic regions, terminating solidification through a low-melting  $\gamma$ /Laves eutectic reaction [5]. This brittle intermetallic Laves phase, when present, has been directly shown to control crack growth mechanisms and inhibit crack growth resistance by nucleating micro voids at the matrix-particle interface, with its elimination yielding a 200% higher fatigue crack growth threshold compared to as-fabricated material [6]. Dissimilar-metal joints between IN718 and 316L have been repeatedly reported to develop cracking at the interface under thermal cycling and fatigue loading, a failure mode directly relevant to the cyclic loads experienced in reactor service. As a result, only diffusion-based joining routes — principally brazing

and hot isostatic pressing (HIP) — have been considered viable for producing IN718–316L transition joints, both requiring long processing times and high-temperature exposure that can promote grain coarsening and further interfacial instability [6].

Spark plasma sintering (SPS) offers a promising alternative route for fabricating IN718–316L bimetallic structures directly from powder feedstock. Compared to brazing and HIP, SPS achieves rapid densification through pulsed direct-current heating and simultaneous uniaxial pressure, substantially shortening processing time while enabling fine control over interfacial microstructure and phase development.

Critically, recent work on IN718–316L interfaces has demonstrated that processing atmosphere can be leveraged to directly suppress Laves-phase formation rather than merely tolerate it. In directed energy deposition of IN718–316L bimetallic structures under N<sub>2</sub> atmosphere, in-situ formation of Ti- and Nb-containing nitride precipitates was shown to actively diminish Nb–Mo Laves phase abundance at the interface, with thermodynamic calculation confirming that increasing N<sub>2</sub> partial pressure drives a corresponding decrease in Laves C14 phase fraction.

This nitride-mediated suppression of Laves phase corresponded with measurable crack mitigation at the interface, along with improved yield strength, ultimate tensile strength, elongation, and hardness relative to samples produced under conventional argon atmosphere. Grain size under nitrogen conditions was also reduced, attributed to nitride-driven hindrance of columnar grain growth [7].

These findings indicate that nitride formation, rather than representing an incidental or deleterious byproduct of nitrogen exposure, can function as a beneficial mechanism that outcompetes brittle Laves-phase precipitation at the IN718–316L interface — directly addressing the primary failure mode responsible for conventional joining difficulties in this alloy pair [7].

the role of nitrogen-promoted nitride formation in suppressing Laves-phase-driven cracking has not been investigated for powder-metallurgy joining routes such as SPS, where the absence of a fusion/solidification

pathway already alters phase evolution relative to fusion-based processes.

Accordingly, this study aims to characterize the interfacial stability and phase evolution of IN718–316L bimetallic structures fabricated by SPS, focusing on enhanced nitride formation under nitrogen versus argon atmospheres and the resulting suppression of Laves-phase-driven interfacial instability.

## 2. Experimental Methods

### 2.1. Thermal Profiles of SPS under Ar and N<sub>2</sub>

The Spark plasma sintering (SPS) is a solid-state powder consolidation technique well-suited for producing dissimilar metal joints without introducing unwanted phases or microstructural artifacts at the bimetallic interface. Densification proceeds through three concurrent mechanisms: plasma heating, Joule heating, and uniaxial compression with grain growth initiating once the powder exceeds its recrystallization temperature. Since consolidation occurs via solid-state diffusion, phase transformation can accompany densification.

In this study, IN718 and 316L stainless steel powders were simultaneously consolidated using a Thermal Technology LLC SPS system under industrial-grade argon (Airgas Co.) in a 20 mm-radius die. Sintering temperature was varied from 800–1200 °C with a 16-minute isothermal dwell, a uniaxial pressure rate of 20 MPa/min, and chamber vacuum near 18 torr (**Fig. 1**). For the nitrogen-atmosphere condition, all processing parameters were held identical to the 1200 °C argon condition, with the sole exception of the purging gas, which was substituted with high-purity (99.99%) N<sub>2</sub>.

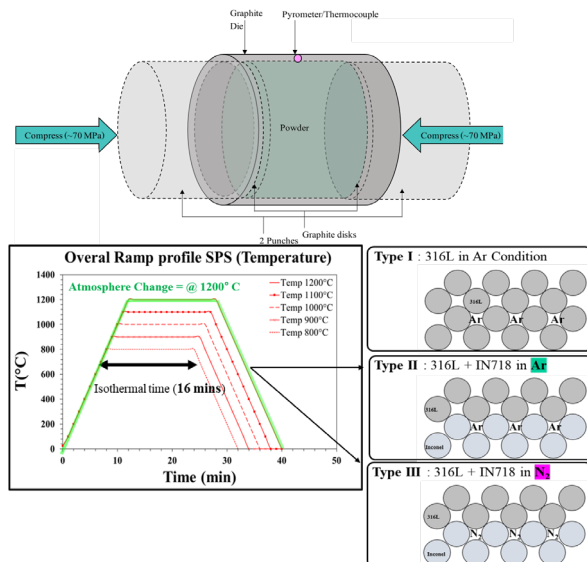


Fig. 1. Thermal profile of Spark plasma sintering process of bimetallic structure both Ar and N<sub>2</sub> atmosphere

## 3. Thermodynamic Approach

In order to characterize compositional evolution across the IN718/316L interface, diffusion coefficients of representative elements (Ti, Nb, and Mo for IN718; Fe for 316L) were calculated at 1000 °C and 1200 °C using ThermoCalc software's diffusion module, corresponding to the sintering temperatures at which near-full densification was experimentally achieved. This thermodynamic approach allowed direct comparison between calculated interdiffusion behavior and the corresponding microstructural interface morphology, revealing a pronounced temperature dependence in interfacial diffusion character.

### 3.1. Diffusion Calculation of Bimetallic Structure

Diffusion coefficients of both alloys during sintering were calculated using ThermoCalc's diffusion module — for IN718, a Ni-based system including Ti, Nb, and Mo; for 316L, an Fe-based system with Fe as the representative element. Diffusion boundaries were calculated at 1000 °C and 1200 °C, the two temperatures corresponding to near-full densification. The interface was faceted at 1000 °C but considerably smoother at 1200 °C, with Nb and Mo also showing a more gradual distribution at the higher temperature (**Fig. 2a**). **Fig. 2b** further shows that Ni and Fe in IN718 diffused considerably faster across the interface than the corresponding elements in 316L. **Fig. 2a** presents the calculated composition profile of the IN718/316L diffusion couple annealed for 16 min at 1000 and 1200 °C, evaluated across five different temperatures. The thermodynamic calculation is consistent with microstructural observations, in that higher temperature produces a more gradually sloped diffusion profile.

Collectively, these results indicate that sintering at 1200 °C promotes more gradual interelement diffusion across the IN718/316L interface, yielding a compositionally continuous and thermodynamically stabilized interfacial region.

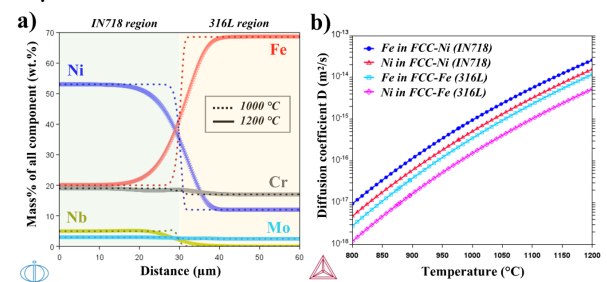


Fig. 2. Diffusion calculation of Ni and Fe in two different temperature via different (a) temperature and (b) interface diffusion coefficient

**Table 1** outlines the phase changes occurring across the sintering temperature range, distinguishing the bulk IN718 and bulk SS316L zones from the interdiffusion region, and together these results show how interdiffusion, phase transformation, and grain evolution interact.

In pristine IN718 powder, sintering between 800–900 °C drove distinct phase changes that shaped microstructural refinement. At 800 °C, the Laves phase already present in the powder acted as reinforcement without converting to another phase. At 900 °C, Laves phase at grain boundaries converted to  $\delta$  phase ( $\text{Ni}_3\text{Nb}$ ), which is compositionally similar to  $\gamma''$ . At 1000 °C, Nb release from the  $\delta$  phase allowed  $\gamma'$  ( $\text{Ni}_3(\text{Ti},\text{Al})$ ) to form as Ti and Al substituted for Nb. Near 1200 °C,  $\gamma'$  and  $\eta$  phase ( $\text{Ni}_3(\text{Nb},\text{Ti},\text{Al})$ ) became compositionally similar despite differing structurally, with  $\eta$  favored by deformed microstructures and retained Nb. Grain refinement in pure IN718 thus stems mainly from interfacial nitride formation and the  $\delta \rightarrow \gamma' \rightarrow \eta$  transformation sequence.

Under nitrogen, the same 1200 °C phases seen under argon persisted, but nitrides also formed uniformly at particle and grain boundaries. This suggests that the  $\eta$  phase, together with nitride-forming elements on both sides of the interface (Ti, Nb, Mo in IN718; Cr in 316L), further promoted recrystallization at the bimetallic interface.

Table I. Identified and Evolved phase of bimetallic component via different atmospheric conditions

| Gas atmosphere | Temp (°C) | Identified and Evolved Phases                                                                     |                                                         |                                                                                          |
|----------------|-----------|---------------------------------------------------------------------------------------------------|---------------------------------------------------------|------------------------------------------------------------------------------------------|
|                |           | Intrinsic region (Bulk IN718, SS316L)                                                             |                                                         | Interdiffusion region (interface)                                                        |
|                |           | IN718 side                                                                                        | SS316L side                                             | Interface                                                                                |
| Ar             | 800       | Nb-Mo Laves phase                                                                                 | $\text{SiO}_2$ , MnO                                    | Nb-Mo Laves Phase                                                                        |
|                | 900       | (Laves $\Rightarrow$ $\delta$ )                                                                   | $\text{SiO}_2$ , MnO                                    | Ti, Nb, Mo-N + (Laves $\Rightarrow$ $\delta$ )                                           |
|                | 1000      | ( $\delta \Rightarrow \gamma'$ )                                                                  | Oxides agglomeration                                    | Ti, Nb, Mo-N + Nb $\Rightarrow$ Ti, Al ( $\delta \Rightarrow \gamma'$ )                  |
|                | 1100      | ( $\delta \Rightarrow \gamma' \Rightarrow \eta$ ) Laves remained                                  |                                                         | Ti-Nb-N and ( $\eta$ )                                                                   |
|                | 1200      | ( $\gamma'$ , $\eta$ ) remained                                                                   |                                                         | Ti-Nb-N network structure ( $\gamma'$ , $\eta$ ) remained                                |
| $\text{N}_2$   | 1200      | Nitride former agent (TiNbMo) forms nitride abundantly, Nitride network promote recrystallization | Nitride former agent (Cr) diffused from dilution region | Strengthened Nitride network developed both (1) Particle boundary and (2) Grain boundary |

## 4. Result and Discussion

This section identifies the sintering condition yielding minimal porosity in the IN718/316L bimetallic structure, followed by SEM- and EPMA-based elemental analysis characterizing interfacial morphology under different atmospheric gas conditions (Ar and  $\text{N}_2$ ) at a fixed temperature of 1200 °C. Finally, a schematic illustration is presented to summarize the microstructural evolution of the IN718–316L system across varying sintering temperatures and atmospheric conditions

### 4.1. Porosity Calculation

The residual density evolution of IN718 and SS316L powders across the 800–1200 °C sintering range. At lower temperatures (800 and 900 °C), both materials retained substantial porosity (~26.7% for SS316L and ~19.8% for IN718 at 800 °C), which decreased markedly with increasing temperature, reaching near-full density above 1000 °C. Further increasing the temperature to 1200 °C produced only marginal additional densification, with samples remaining consistently at or above 99.8% relative density. Under Nitrogen condition, the porosity follows the same as Argon condition.

Interfacial morphology also followed a similar temperature dependence. At 800 and 900 °C, the interface remained clearly defined and retained the original powder morphology whereas above 1000 °C the interface became progressively obscured as residual porosity was eliminated. where enhanced atomic diffusion and grain boundary mobility at elevated temperature promote grain coarsening. At 1200 °C in particular, this effect was most pronounced on the stainless-steel side, which exhibited substantial grain growth (Fig. 3).

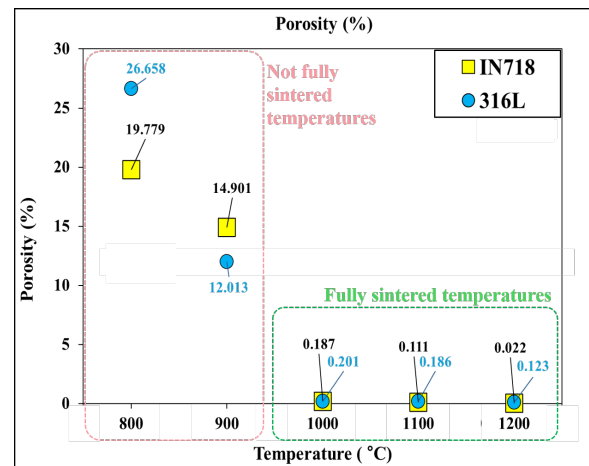


Fig. 3. Porosity calculation via temperatures from 800 to 1200 °C

### 4.2. Phase Evaluation via Characterization

Building on the porosity results, the interfacial region of the IN718/316L bimetallic structure sintered at 1200 °C — the condition yielding minimal porosity — was examined in detail under different atmospheric gas conditions. Fig. 4a shows the interface under argon atmosphere, in which the brighter upper region corresponds to IN718 and the darker region corresponds to 316L. The grain size of IN718 was noticeably finer than that of 316L, and a substantial number of annealing twins were observed within the IN718 region.

Notably, accelerated recrystallization was evident at the interface where the two alloys met, indicating that

limited interdiffusion had occurred during sintering. **Fig. 4b** presents the corresponding interface under nitrogen atmosphere, imaged under identical BSE conditions. In this case, recrystallization was further intensified not only at the bimetallic interface but also within the adjacent IN718 region; this enhanced recrystallization is attributed to accelerated nitride formation at the powder-particle interface, driven by the elevated energy difference associated with the interdiffusion zone.

**Fig. 4c** and **4d** present EPMA nitrogen elemental maps of the Ar- and N<sub>2</sub>-atmosphere interfaces, respectively, allowing direct comparison of nitrogen content between the two atmospheric conditions. The nitrogen signal intensity was measured at approximately 150 CPS under argon atmosphere, compared to approximately 650 CPS under nitrogen atmosphere, indicating markedly coarser and more clearly defined nitride formation at the interface under the nitrogen condition. This enhancement is attributed to the pressurized nitrogen atmosphere promoting nitride formation via the nitride-forming elements inherently present in both IN718 and 316L.

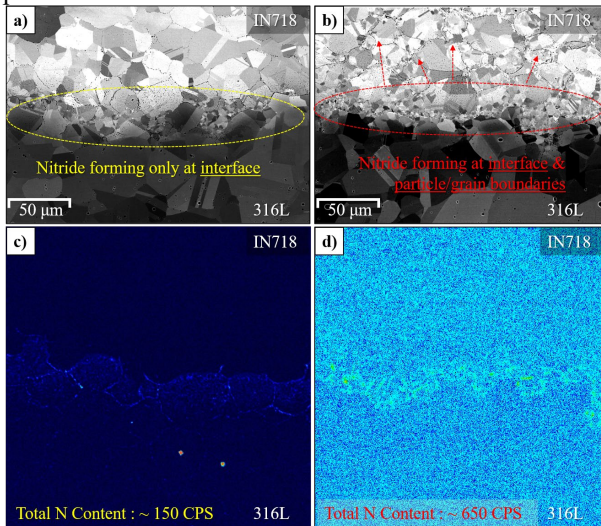


Fig. 4. Interface analysis of bimetallic structure via BSE image (a,b) and EPMA nitride content CPS measurement varied by (c)Ar atmosphere and (d) N<sub>2</sub> atmosphere

#### 4.3. Phase Evolution via Atmospheric Conditions

Based on the above analyses, the phase evolution of both alloys as a function of atmospheric gas condition and sintering temperature is summarized below.

In the pure IN718 region (**Fig. 5a**), the Laves phase at ~800 °C is thermodynamically stable and largely inert, acting as a rigid, strengthening constituent. By 900 °C, Nb redistribution from the Laves phase drives its gradual transformation into  $\delta$  phase (Ni<sub>3</sub>Nb), mainly along grain boundaries;  $\delta$  is chemically similar to the metastable  $\gamma'$  phase, though crystallographically distinct. Near 1000 °C, Nb depletion from  $\delta$ , alongside Ti and Al enrichment, promotes formation of the Nb-deficient  $\gamma'$

phase (Ni<sub>3</sub>(Ti,Al)). Near 1200 °C,  $\gamma'$  may further transform into  $\eta$  phase (Ni<sub>3</sub>Nb(Ti,Al)), especially under deformation or non-equilibrium Nb retention.

In contrast to these established bulk transformation pathways, **Fig. 5b** reveals that the dominant microstructural evolution in the present study occurs at interfaces rather than within the bulk matrix. Phase transformation was found to be strongly localized at particle-particle interfaces formed during sintering, where chemical gradients and diffusion fluxes are significantly enhanced; the transformation sequence from Laves phase to  $\delta$ , and its subsequent progression toward  $\gamma'$  and  $\eta$ , was preferentially initiated and accelerated within these interfacial regions.

Under nitrogen atmosphere, the  $\eta$  phase concentrated at the interface at the same temperature of 1200 °C was closely associated with nitride formation, which not only intensified nitride development at the interface itself but also extended its influence to the surrounding particle boundaries.

As shown in **Fig. 5b**, interfacial nitride formation was consistently observed and functioned as an effective pinning site restricting grain boundary migration, thereby playing a critical role in grain refinement — an effect substantially more pronounced than the comparatively limited contribution of bulk phase transformation under the present sintering conditions. The sequential  $\delta \rightarrow \gamma' \rightarrow \eta$  transformation pathway is therefore not uniformly distributed throughout the material, but is instead concentrated near interfaces, where localized compositional inhomogeneity drives phase instability.

Since pristine IN718 powder containing pre-existing Laves phase, grain refinement is governed primarily by interfacial phenomena — namely (1) nitride formation and (2) localized sequential phase transformation — rather than by conventional bulk phase evolution. Notably, the enhanced nitride formation observed under nitrogen atmosphere appears to drive conversion of the Laves phase, an otherwise deleterious constituent at the bimetallic interface, into more favorable interfacial products. This interface-dominant behavior represents a key distinction from previously reported bulk IN718 alloy behavior and underscores the importance of accounting for powder-specific and interface-driven mechanisms during sintering process.

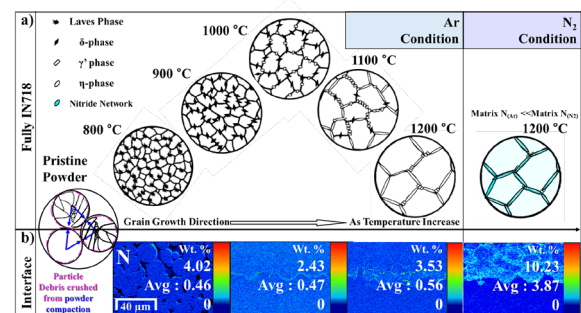


Fig. 5. Schematic summarization of phase evolution in bimetallic structure of IN718 and 316L (a) at fully IN718 region and (b) at the interface

## 5. Conclusion

This study demonstrates fabrication of IN718–316L bimetallic structures via spark plasma sintering (SPS), a solid-state alternative to fusion-based joining. Near-full densification ( $\geq 99.8\%$ ) was achieved above 1000 °C, and diffusion calculations confirmed a smoother, more compositionally continuous interface at 1200 °C. The sequential Laves  $\rightarrow \delta \rightarrow \gamma' \rightarrow \eta$  transformation in IN718 localized preferentially at particle-particle interfaces rather than throughout the bulk, distinguishing powder-based SPS behavior from previously reported bulk IN718 pathways.

Under nitrogen, interfacial nitride formation was substantially enhanced (EPMA intensity  $\sim 650$  vs.  $\sim 150$  CPS under argon), pinning grain boundaries and converting the deleterious Laves phase into more stable nitrides. This indicates that interfacial nitride formation, rather than bulk phase evolution, primarily governs grain refinement and interfacial stability in SPS-fabricated IN718–316L joints — to our knowledge, the first demonstration of this mechanism in a powder-metallurgy joining route.

Atmosphere-controlled SPS thus offers a promising route for IN718–316L transition joints in demanding applications, including Generation IV nuclear reactors, though future work should address mechanical performance and long-term interfacial stability under service conditions

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