

## Effect of Notch Orientation and Recrystallization on Thermal Fatigue Performance of a Directionally Solidified Cobalt-Based Superalloy (Postprint)

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### Abstract

This study employed plate-shaped thermal fatigue specimens with “V” notches oriented perpendicular and parallel to the solidification direction, and pre-fabricated recrystallized microstructures at the notch locations, to investigate the effects of notch orientation and recrystallization on the thermal fatigue performance of directionally solidified cobalt-based superalloys under thermal cycling between a maximum temperature of 1000°C and room temperature. The results show that: when the notch orientation is perpendicular to the solidification direction, the matrix undergoes cyclic oxidation cracking under stress; when the notch is parallel to the solidification direction, the thermal fatigue performance decreases, and cracks propagate along interdendritic regions. Recrystallization degrades the thermal fatigue performance of directionally solidified cobalt-based superalloys, recrystallized grain boundaries oxidize and crack, and voids formed by the oxidation and spalling of M23C6-type carbides precipitated at grain boundaries during experiments accelerate crack propagation; recrystallized grain boundaries connecting interdendritic carbides become the preferential propagation path for thermal fatigue cracks when the notch is parallel to the solidification direction.

### Full Text

### Introduction

Cobalt-based superalloys exhibit excellent high-temperature corrosion resistance, good thermal conductivity, and low coefficients of thermal expansion, making them suitable for manufacturing aero-engine turbine vanes [1]. During service, guide vanes operate at temperatures approximately 100 °C higher than

turbine blades. The repeated temperature fluctuations during engine start-up, shutdown, or afterburner operation subject guide vanes to significant thermal loads and thermal shocks. The cyclic variation of thermal stress and strain readily induces thermal fatigue cracks in blades, leading to engine failure [2,3]. Consequently, thermal fatigue performance represents a critical metric for evaluating materials for aero-engine guide vanes.

The factors influencing the thermal fatigue performance of superalloys are complex, with the underlying mechanisms closely linked to alloy type. Studies [4-6] have shown that polycrystalline alloys generally fail by intergranular cracking; however, as grain size increases, thermal fatigue cracks transition to a transgranular propagation mode, with reduced growth rates and improved thermal fatigue performance [7]. For directionally solidified (DS) nickel-based superalloys, V-notch specimens with the notch perpendicular to the solidification direction are typically employed to investigate thermal fatigue behavior [8,9], with cracks propagating primarily along crystallographic orientations or maximum shear stress directions [8,10,11]. Carbides and eutectic phases in the alloy can serve as crack initiation sites, and even rafted phases may promote crack propagation [12]. Xiao et al. [12] observed that in DS alloy DZ445 tested with notches parallel to the solidification direction, thermal fatigue cracks propagated mainly along interdendritic regions; increasing the maximum test temperature caused cracks to switch to crystallographic orientation propagation. These findings demonstrate that notch orientation and microstructure significantly influence thermal fatigue crack morphology in DS superalloys. However, current thermal fatigue research predominantly focuses on nickel-based superalloys, with very limited studies on cobalt-based alloys [13], and the effect of notch orientation on the thermal fatigue behavior of DS cobalt-based superalloys remains unreported.

Furthermore, plastic deformation during the casting process of DS columnar-grained superalloy blades—resulting from metal shrinkage, core constraint, and subsequent machining, straightening, and shot peening—can lead to recrystallization (RX) during solution heat treatment or ultra-high temperature service [14,15]. Studies [16,17] have shown that recrystallization, as a defective microstructure, severely compromises the structural integrity of DS superalloys and significantly reduces their high-temperature tensile, stress-rupture, and fatigue properties. However, research on the influence of recrystallized microstructures on the thermal fatigue performance of DS superalloys and the associated mechanisms remains scarce.

Based on this background, the present work investigates a DS cobalt-based superalloy suitable for guide vane applications. Plate-type thermal fatigue specimens with V-notches oriented both perpendicular and parallel to the solidification direction were prepared, with recrystallized microstructures pre-induced at the notch locations. The effects of notch orientation and recrystallization on thermal fatigue performance were studied, and the underlying failure mechanisms were analyzed through crack propagation morphology observations. The

results provide theoretical guidance and experimental basis for the processing and engineering application of this alloy in vane components.

## Experimental Procedures

The DS cobalt-based superalloy used in this study had a nominal composition (mass fraction, %) of: Cr 25, Ni 10, W 7.5, Al 1.2, C 0.45, Ta 0.4, Mo 0.35, Ti 0.3, Zr 0.25, B 0.018, and balance Co. DS plates were fabricated by the high-rate solidification (HRS) method. Two groups of plates measuring 25 mm  $\times$  17 mm  $\times$  3 mm were cut from the DS plate by wire electrical discharge machining, with the long edges oriented perpendicular and parallel to the DS direction, respectively.

Local deformation was introduced at positions approximately 3 mm inward from the midpoint of the short edge on both sides of each plate using a Brinell hardness tester with a 5 mm diameter indenter under a 1500 kg load for 10 s. The locally deformed plates were then heat-treated at 1250 °C for 90 min followed by air cooling. Based on preliminary experiments [18], this procedure produced a recrystallized region with a radius of approximately 2 mm in the 3 mm thick alloy plates, with recrystallization occurring through the thickness direction. Both sides of the plates were ground to remove the indentations, and standard thermal fatigue specimens were machined with the V-notch centered at the indentation location, as shown in [Figure 1: see original paper].

Specimens with the V-notch perpendicular to the DS direction were designated transverse samples ([Figure 1: see original paper]a), while those with the notch parallel to the DS direction were designated longitudinal samples ([Figure 1: see original paper]b). To evaluate the effect of recrystallization, transverse and longitudinal samples without plastic deformation were also prepared and subjected to the same heat treatment (1250 °C, 90 min, air cooling). Five specimens were tested for each of the four conditions. All samples were mechanically polished prior to testing and examined under an optical microscope to ensure no pre-existing cracks were present near the notch.

Thermal fatigue testing was conducted in a box-type muffle furnace. The thermal cycle consisted of heating to 1000 °C, holding for 3 min, then water quenching to room temperature. After a predetermined number of thermal cycles, specimens were mechanically ground, polished, and etched for measurement of the primary crack length using a reading microscope. The primary crack, located at the V-notch, was identified as the longest and widest crack among multiple potential cracks. Reported crack lengths represent averages of the primary crack lengths measured on ten surfaces (both sides of five specimens) under identical conditions. Microstructural characterization and crack morphology observations were performed using an Axiovert200MAT optical microscope (OM) and an S-3400N scanning electron microscope (SEM) with backscattered electron (BSE) imaging. Energy-dispersive spectroscopy (EDS) and X-ray diffraction (XRD) were employed for compositional and phase analysis. The chemical etchant

used was a solution of 4 g  $\text{CuSO}_4 + 80 \text{ mL HCl} + 20 \text{ mL C}_2\text{H}_5\text{OH}$ .

## Results

### 2.1 As-Cast and Recrystallized Microstructures

[Figure 2: see original paper] shows the as-cast microstructure of the DS cobalt-based superalloy. The alloy grew along the  $\langle 001 \rangle$  direction, with primary dendrite arms parallel to the DS direction. Numerous carbides were distributed along columnar grain boundaries and interdendritic regions. [Figure 3: see original paper] presents SEM and BSE images of the carbides. Three types of carbides were identified: blocky and irregularly shaped  $\text{M}_2\text{C}$  and  $\text{MC}$  carbides, with fine  $\text{M}_2\text{C}$  carbides dispersed around these primary carbides ([Figure 3: see original paper]a). The Cr-rich  $\text{M}_2\text{C}$  carbides exhibited lower brightness, while the  $\text{MC}$  carbides containing heavy elements such as Ta, Ti, Zr, and W appeared brighter ([Figure 3: see original paper]b). EDS analysis of the  $\text{M}_2\text{C}$  and  $\text{MC}$  carbides is shown in [Figure 4: see original paper]. The primary dendrite arm spacing was approximately  $242 \mu\text{m}$ , and the total carbide content was about 7.3%.

XRD analysis of carbides extracted from as-cast DS samples after annealing at  $1250^\circ\text{C}$  for 90 min revealed that both  $\text{M}_2\text{C}$  and  $\text{MC}$  carbides remained present ([Figure 5: see original paper]). [Figure 6: see original paper] compares the microstructures of as-cast and indented-deformed samples after annealing at  $1250^\circ\text{C}$  for 90 min. In the as-cast sample, fine  $\text{M}_2\text{C}$  carbides nearly completely dissolved, while the originally irregular  $\text{M}_2\text{C}$  carbides in the interdendritic regions transformed into blocky morphologies. The  $\text{MC}$  carbides, containing high-melting-point elements, dissolved more slowly and retained their fine strip-like morphology ([Figure 6: see original paper]a). In the indented sample, the deformed region underwent complete recrystallization with relatively straight RX grain boundaries. The  $\text{M}_2\text{C}$  carbides also nearly completely dissolved, while some residual  $\text{M}_2\text{C}$  and  $\text{MC}$  carbides remained distributed along the grain boundaries ([Figure 6: see original paper]b). Carbide content measurements indicated that after annealing at  $1250^\circ\text{C}$ , the carbide volume fraction decreased to approximately 2.1% in the as-cast sample and about 2.0% in the recrystallized region, showing no significant difference between the two conditions.

### 2.2 Crack Propagation Behavior

When V-notched DS cobalt-based superalloy specimens were water-quenched from high temperature, the notch region contracted, generating stress concentration at the notch tip. After multiple thermal cycles, all four specimen groups—with and without recrystallization, in both transverse and longitudinal orientations—developed thermal fatigue cracks to varying degrees. [Figure 7: see original paper] plots the relationship between average primary crack length ( $L$ ) and thermal cycle number ( $N$ ). After 5 cycles, all four groups remained intact. At

15 cycles, the transverse and longitudinal samples without recrystallization were still crack-free, whereas both recrystallized groups had initiated cracks. After 30 cycles, cracks had formed in the non-recrystallized transverse and longitudinal samples as well. Crack lengths increased significantly with continued cycling. Within 75 cycles, the recrystallized longitudinal specimens exhibited the longest primary crack extension, followed by the non-recrystallized longitudinal specimens, then the recrystallized transverse specimens, with the non-recrystallized transverse specimens showing the shortest cracks.

Further analysis revealed that after 75 thermal cycles, the average crack length in recrystallized longitudinal samples reached 1.7 mm, approximately 2.8 times that of recrystallized transverse samples. In the non-recrystallized groups, the longitudinal sample average crack length (1.14 mm) was 4.4 times greater than that of transverse samples (0.26 mm). Additionally, the average crack lengths in recrystallized and non-recrystallized transverse samples were 0.61 mm and 0.26 mm, respectively. At equivalent cycle numbers, the presence of recrystallization also increased crack length and growth rate in longitudinal samples. Notably, both longitudinal groups consistently exhibited greater crack lengths and growth rates than the two transverse groups. These results demonstrate that thermal fatigue performance is always superior when the notch orientation is perpendicular to the solidification direction, and this trend persists even with recrystallization. Under identical orientation conditions, recrystallization significantly accelerates crack propagation and degrades thermal fatigue performance.

### 2.3 Crack Morphology Observations

[Figure 8: see original paper]a shows the thermal fatigue crack morphology in a non-recrystallized transverse DS cobalt-based superalloy after 30 thermal cycles. Two cracks were present at the notch: a shorter crack perpendicular to the primary dendrite arm direction (i.e., normal to the principal stress direction) and another crack oriented at  $45^\circ$  to the primary dendrite growth direction, propagating along the maximum shear stress direction. After 75 cycles, the crack along the maximum shear stress direction grew slowly, while the crack perpendicular to the primary dendrite arms extended inward and widened, becoming the dominant crack ([Figure 8: see original paper]b). SEM observation of the primary crack front revealed that the crack arrested within the alloy matrix ([Figure 9: see original paper]a). EDS analysis indicated high oxygen content near the crack tip, suggesting oxidation of the matrix ahead of the crack ([Figure 9: see original paper]b).

[Figure 10: see original paper]a illustrates crack propagation in a recrystallized transverse sample after 15 thermal cycles. Unlike matrix cracking in non-recrystallized transverse specimens ([Figure 9: see original paper]a), cracking initiated at recrystallized grain boundaries near the notch tip and propagated along these boundaries. After 75 cycles, multiple cracks formed at the notch with branching during propagation, though the primary extension directions generally aligned with the principal stress and maximum shear stress directions

([Figure 10: see original paper]b). The longest crack extended along the principal stress direction (perpendicular to primary dendrite arms). SEM imaging ([Figure 10: see original paper]c) revealed numerous nanoscale M<sub>23</sub>C<sub>6</sub> secondary carbides precipitated within recrystallized grains. Thermal fatigue cracks propagated along recrystallized grain boundaries, with M<sub>23</sub>C<sub>6</sub>-depleted zones observed near the crack path. Blocky carbides on grain boundaries exhibited cracking and severe oxidation. [Figure 10: see original paper]d shows that M<sub>23</sub>C<sub>6</sub> secondary carbides precipitated along recrystallized grain boundaries; however, at the crack front, previously precipitated M<sub>23</sub>C<sub>6</sub> carbides detached, pre-forming intergranular voids that accelerated crack propagation.

[Figure 11: see original paper]a depicts crack initiation and propagation in a non-recrystallized longitudinal DS sample after 30 thermal cycles. Cracks nucleated in interdendritic regions at the notch tip. After 75 cycles ([Figure 11: see original paper]b), the primary crack propagated along interdendritic regions. The SEM image of the primary crack front ([Figure 11: see original paper]c) shows large blocky M<sub>23</sub>C<sub>6</sub> carbides and smaller strip-like MC carbides distributed densely along the crack path. During propagation, interfacial decohesion between primary carbides and the matrix caused carbide detachment, leaving voids, with the crack eventually arresting near MC carbides.

[Figure 12: see original paper] shows crack initiation, propagation, and carbide morphology near cracks in recrystallized longitudinal specimens. After 15 thermal cycles, stress concentration at the V-notch tip caused cracking of recrystallized grain boundaries near the notch, with carbide cracking on grain boundaries promoting crack extension ([Figure 12: see original paper]a). After 75 cycles, the primary crack extended parallel to the primary dendrite direction from the notch into the specimen interior ([Figure 12: see original paper]b). The SEM image of the primary crack front ([Figure 12: see original paper]c) indicates crack propagation along recrystallized grain boundaries from left to right. Secondary cracks formed at triple junctions along recrystallized grain boundaries, oriented approximately parallel to the principal stress direction, but arrested at carbide-matrix interfaces. Additionally, carbides on grain boundaries exhibited interfacial cracking under the influence of the crack tip stress field, with larger carbides impeding crack propagation.

## Discussion

### 3.1 Effect of Notch Orientation on Thermal Fatigue Performance

Research on the influence of notch orientation on thermal fatigue performance of DS superalloys is limited. Previous studies on nickel-based alloy DZ22 reported that thermal fatigue performance decreased significantly when the angle between notch direction and solidification direction was less than 45° [19]. Robert and Richard [20] found that DS nickel-based alloys exhibited superior thermo-mechanical fatigue life when notches were perpendicular to the solidification direction, whereas cracks propagated along columnar grain boundaries near the

notch when oriented parallel to the solidification direction. Similar observations were made in the present study. The alloy contained numerous blocky MC and M C carbides distributed along columnar grain boundaries and interdendritic regions ([Figure 6: see original paper]a), which served as crack initiation and propagation paths ([Figure 11: see original paper]a and b). However, when the notch was perpendicular to the solidification direction ([Figure 8: see original paper]b), cracks propagated through dense dendrite cores perpendicular to the principal stress direction, with minimal influence from microstructural features, indicating fundamentally different failure mechanisms between the two notch orientations.

Xia et al. [21] reported that in nickel-based alloy DZ319, cracks propagated by linking oxidation voids through dense dendrite cores. In contrast, the present work on transverse samples with notches perpendicular to the solidification direction ([Figure 8: see original paper]b) observed oxidation near cracks but no oxidation voids ahead of the crack front ([Figure 9: see original paper]a), possibly attributable to the excellent oxidation resistance of cobalt-based alloys. Research [22] has shown that static oxidation products of cobalt-based alloys below 1000 °C consist primarily of continuous, dense CoO and Cr O scales. In the current work, under severe temperature fluctuations and stress concentration at the V-notch, brittle oxide films fractured, exposing fresh alloy surfaces at the crack tip. Reuchet and Remy [23] calculated that oxygen diffusion to the crack tip in cobalt-based alloys at 900 °C in air reaches saturation in only  $5 \times 10^{-4}$  s. Kang et al. [24] found that stress can accelerate oxygen diffusion into the alloy, exacerbating high-temperature oxidation. Simultaneously, oxidation of Al, Ti, and Cr in the matrix continuously consumed these strengthening elements, reducing alloy strength. The combined effects resulted in the most severe oxidation at the V-notch, with the oxide layer repeatedly forming and fracturing during thermal cycling, leading to continuous oxidation of the crack tip. This cyclic oxidation mechanism represents the primary cracking mode for transverse samples with notches perpendicular to the solidification direction, as shown in [Figure 8: see original paper].

For longitudinal samples with notches parallel to the solidification direction, microstructural effects on crack initiation and propagation were significant in addition to oxidation. The coefficient of thermal expansion (CTE) of cobalt-based superalloys at 980 °C is approximately  $1.7 \times 10^{-6}$  °C<sup>-1</sup> [25], higher than that of Cr-rich M C or M C carbides ( $1.1 \times 10^{-6}$  °C<sup>-1</sup> [25]) and approximately twice that of MC carbides rich in Ta, Ti, Zr, and W ( $0.7 \times 10^{-6}$  to  $0.8 \times 10^{-6}$  °C<sup>-1</sup> [26]). The CTE mismatch between the matrix and carbides caused compressive stresses on carbides during heating and tensile stresses during cooling, degrading the carbide-matrix interfacial strength [6]. When cracks encountered carbides during propagation, the crack tip stress exceeded the interfacial bonding strength, causing interfacial decohesion. The high density of carbides distributed in interdendritic regions of DS cobalt-based alloys provided low-energy propagation paths compared to crack propagation through dense dendrite cores in transverse samples ([Figure 8: see original paper]), resulting in preferential interdendritic

cracking in longitudinal samples ([Figure 11: see original paper]b).

### 3.2 Effect of Recrystallization on Thermal Fatigue Performance

Recrystallization generally causes drastic degradation of DS superalloy properties. Xie et al. [16] reported that in DS nickel-based superalloy DZ125L containing surface recrystallization, grain boundaries perpendicular to the stress axis readily cracked during high-temperature stress-rupture testing. Zheng et al. [27] suggested that dendrite cores constitute the primary load-bearing structure in DS alloys, and that recrystallized layers have extremely low load-bearing capacity. If dendrite cores are intersected by recrystallized grain boundaries, these regions become weak links in the alloy. In the present study of transverse samples, recrystallized grains were pre-induced in the notch region, with numerous grain boundaries intersecting dendrite cores and disrupting the original columnar grain structure ([Figure 6: see original paper]b). Thermal cycling induced periodic thermal stress variations, causing cracks to initiate and propagate along recrystallized grain boundaries ([Figure 10: see original paper]). However, as shown in [Figure 7: see original paper], the crack growth rate in recrystallized transverse samples was lower than that in non-recrystallized longitudinal samples, indicating that recrystallized grain boundaries in DS cobalt-based superalloys retain some load-bearing capacity.

Unlike nickel-based alloys, cobalt-based alloys under cyclic stress during thermal fatigue testing generate extensive slip and dislocation activity, promoting Cr segregation to slip planes and grain boundaries where it reacts with C in the matrix [28,29], precipitating nanoscale secondary M<sub>23</sub>C<sub>6</sub> carbides in both the matrix and grain boundaries ([Figure 10: see original paper]d). The presence of recrystallized grain boundaries facilitates secondary M<sub>23</sub>C<sub>6</sub> precipitation, which can enhance high-temperature stress-rupture strength [30]. However, grain boundaries also exhibit high solubility and diffusivity [31,32], providing rapid diffusion pathways for oxygen that reduce grain boundary strength and promote thermal fatigue crack propagation. Cr in cobalt-based alloys is chemically active and prone to oxidation at high temperatures [22]. In the present experiments, oxidation-induced Cr depletion near grain boundary cracks caused previously precipitated M<sub>23</sub>C<sub>6</sub> carbides to redissolve, creating M<sub>23</sub>C<sub>6</sub>-depleted zones near cracks ([Figure 10: see original paper]c). Simultaneously, M<sub>23</sub>C<sub>6</sub> carbides on recrystallized grain boundaries at the crack front decomposed, and their interfacial bonding with the matrix weakened, leading to carbide detachment and intergranular void formation ([Figure 10: see original paper]d). Crack propagation by linking these intergranular voids reduced propagation resistance and accelerated cracking, degrading the thermal fatigue performance of recrystallized transverse samples shown in [Figure 10: see original paper].

In the two recrystallized specimen groups, longitudinal samples with notches parallel to the solidification direction exhibited the highest crack propagation rate ([Figure 7: see original paper]), and crack morphology ([Figure 12: see original paper]b) differed significantly from that of transverse samples ([Figure 10: see

original paper]b). This indicates that the original microstructure substantially influences the thermal fatigue performance of recrystallized samples with different notch orientations. After introducing local recrystallization, primary MC and M C carbides became interconnected through recrystallized grain boundaries ([Figure 6: see original paper]b, [Figure 11: see original paper]a), creating carbide-dense grain boundary paths. Since primary carbides distribute along interdendritic regions, these grain boundary paths were approximately aligned with the solidification direction. As previously discussed, the interfacial strength between carbides and the matrix degraded under cyclic stress-strain conditions. Concurrent with grain boundary oxidation and cracking, secondary M C carbides precipitated on grain boundaries detached under stress and oxidation, accelerating grain boundary fracture. According to the principle of minimum energy for crack propagation, these combined factors caused cracks in recrystallized longitudinal samples to preferentially propagate along recrystallized grain boundaries connecting interdendritic carbides ([Figure 12: see original paper]b), resulting in the lowest thermal fatigue performance.

In summary, notch orientation and recrystallization induce different thermal fatigue damage mechanisms in DS cobalt-based superalloys. The performance difference between notch orientations depends on whether cracks propagate along interdendritic primary carbides, and this effect also influences the two recrystallized specimen groups. However, as shown in [Figure 7: see original paper], even the crack growth rate in recrystallized transverse samples was lower than that in non-recrystallized longitudinal samples, demonstrating that regularly aligned primary carbides along the notch direction constitute a critical factor reducing thermal fatigue performance, regardless of recrystallization. Based on these results, for aero-engines and ground gas turbines employing DS cobalt-based superalloy components, comprehensive consideration of stress state, alloy orientation, surface recrystallization, and service environment is essential. Preventing surface recrystallization and avoiding component loading directions perpendicular to or at large angles with the columnar grain direction are crucial for ensuring thermal fatigue performance.

## Conclusions

- (1) Within 75 thermal cycles, the primary crack extension length in recrystallized longitudinal samples of the DS cobalt-based superalloy was the longest, followed by non-recrystallized longitudinal samples, then recrystallized transverse samples, with non-recrystallized transverse samples exhibiting the shortest cracks.
- (2) When the notch orientation was perpendicular to the solidification direction, the alloy failed by cyclic oxidation cracking under stress, with cracks propagating perpendicular to the solidification direction and exhibiting superior thermal fatigue performance. When the notch was parallel to the solidification direction, thermal fatigue cracks propagated along interdendritic regions with dense distributions of primary carbides along the crack

path.

- (3) Recrystallization degraded the thermal fatigue performance of the DS cobalt-based superalloy through oxidation cracking of recrystallized grain boundaries. During thermal fatigue, M<sub>23</sub>C<sub>6</sub> carbides precipitated on grain boundaries, and their oxidative detachment created intergranular voids that accelerated crack propagation. Recrystallized grain boundaries connecting interdendritic carbides served as preferential crack propagation channels when the notch was parallel to the solidification direction.

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