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Post-Print: Anisotropy of Tensile Properties in DD9 Single Crystal Superalloy

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Date: 2023-03-19T00:00:00+00:00

Abstract

The microstructures of third-generation single-crystal superalloy DD9 with [001], [011], and [111] orientations were investigated using optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM), and the tensile properties of the three orientations at 760 and 1100 ℃ were tested on a tensile testing machine. The results show that on cross-sections perpendicular to the crystal growth direction, the as-cast dendrite morphologies and the shapes of the g phase in the heat-treated state differ among DD9 alloys with [001], [011], and [111] orientations. With increasing temperature, the tensile strength and yield strength of the alloys decrease, and the anisotropy weakens. Except that the yield strength of the [001] orientation at 1100 ℃ is slightly lower than that of the [011] orientation, the tensile strength and yield strength of the DD9 alloy with [001] orientation are respectively higher than those of the alloys with [011] and [111] orientations. The tensile fracture surfaces of DD9 alloys with [001], [011], and [111] orientations at 760 ℃ exhibit quasi-cleavage characteristics, while the fracture surfaces at 1100 ℃ show dimple fracture characteristics. At 760 ℃, the DD9 tensile specimens contain dense dislocations in the matrix channels, with stacking faults appearing within the g phase in the [001] orientation. At 1100 ℃, the [001] and [111] orientations accumulate a large number of dense dislocation networks in both the matrix channels and the g phase, while the [011] orientation exhibits a large number of deformation twin bands.

Full Text

Preamble

ACTA METALLURGICA SINICA
Vol. 51, No. 10, October 2015, pp. 1253-1260

ChinaRxiv

Tensile Anisotropy of DD9 Single Crystal Superalloy*

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Abstract

The microstructures and tensile properties of the third-generation single crystal superalloy DD9 with [001], [011], and [111] orientations were investigated using optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and tensile testing at 760 and 1100 °C. The results show that the as-cast dendritic structures and heat-treated γ phases of DD9 alloy differ among the three orientations on sections perpendicular to the crystal growth direction. With increasing temperature, both the ultimate tensile strength and yield strength decrease, while tensile anisotropy diminishes. Except for the yield strength of [001] orientation being slightly lower than that of [011] orientation at 1100 °C, the ultimate tensile strength and yield strength of DD9 alloy with [001] orientation are higher than those with [011] and [111] orientations. The fracture characteristics transform from quasi-cleavage at 760 °C to dimple fracture at 1100 °C. At 760 °C, very high density dislocations appear in the matrix channels for all three orientations, but stacking faults are present only in γ particles with [001] orientation. At 1100 °C, high-density dislocation networks accumulate in both matrix channels and γ particles for [001] and [111] orientations, while a large number of deformation twins are observed in samples with [011] orientation.

Keywords: single crystal superalloy DD9, tensile property, anisotropy, fracture surface, dislocation

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1. Introduction

Nickel-based single crystal superalloys are widely used in key hot-section components of advanced aero-engines due to their superior high-temperature mechanical properties. During service, turbine blades experience complex temperature and stress states that frequently generate multi-axial stresses. Consequently, the mechanical properties of three principal orientations must be thoroughly investigated. However, most existing research has focused on first- and second-generation single crystal superalloys, with limited reports on third-generation alloys.

DD9 is a third-generation single crystal superalloy independently developed in China. Its rhenium content is lower than that of foreign third-generation superalloys, yet it offers high-temperature strength, excellent comprehensive properties, microstructural stability, and good castability, with performance comparable to CMSX-10, René N6, and TMS-75 [?]. Building upon previous studies of tensile properties in [001]-oriented DD9, this work investigates the tensile behavior of [011]- and [111]-oriented DD9 single crystal superalloy to elucidate the mechanism of tensile anisotropy and provide design guidance for engine designers.

Previous studies on earlier generation superalloys have established the importance of orientation effects. Shah and Duhl [?] demonstrated that PWA1480 alloy exhibits higher tensile properties in [001] orientation than in [011] orientation at 593 °C. Dalal et al. [?] investigated orientation effects on physical and mechanical properties, reporting tensile anisotropy at room temperature where [111] orientation showed maximum tensile and yield strength, while [011] orientation exhibited the best ductility; however, this anisotropy disappeared at 982 and 1093 °C. Liu et al. [?] studied the anisotropy of creep properties in a nickel-based single crystal superalloy, finding that [001] orientation had the best ductility, [111] orientation the longest life, and [011] orientation the lowest performance, attributing these differences to the number and mode of activated slip systems. MacKay and Maier [?] reported that for MAR-M247 and MAR-M200 single crystal alloys, creep life decreased in the order of [111], [001], and [011] at 774 °C. Research on DD6 single crystal superalloy [?] revealed tensile anisotropy, with [001] orientation showing higher tensile and yield strengths than [011] and [111] orientations above 850 °C. These studies collectively demonstrate that first- and second-generation single crystal superalloys exhibit both tensile and creep property anisotropy.

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2. Experimental Methods

DD9 master alloy was vacuum-melted using high-purity raw materials with the following composition (mass fraction, %): Cr 1.5-5.0, Co 5.5-9.5, Mo 0.5-3.0, W 6.0-8.5, Al 5.2-6.2, Ta 5.5-9.0, Re 3.5-5.0, Hf 0-0.5, Nb 0-1.5, C 0-0.04, and Ni balance. The alloy was remelted, cast, and directionally solidified in a vacuum directional solidification furnace. Single crystal bars of [001], [011], and [111] orientations were prepared using a seed crystal method. Bars with growth directions deviating less than 10° from the principal stress axis were selected and subjected to standard heat treatment.

Metallographic samples were sectioned perpendicular to the solidification direction for both as-cast and heat-treated conditions. Dendritic structures and γ phases were examined using optical microscopy and S-4800 field emission SEM. Heat-treated γ phases and tensile fracture surfaces were observed via SEM, while dislocation configurations in tensile specimens were analyzed using JEM-2000FX TEM. Heat-treated single crystal bars were machined into tensile test specimens, and tensile properties of [001], [011], and [111] orientations were evaluated using an Instron 2300 electromechanical testing machine to characterize the tensile anisotropy of DD9 alloy.

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3. Experimental Results

3.1 Microstructure of [001], [011], and [111] Oriented DD9 Alloy

[Figure 1: see original paper] shows the as-cast dendritic structures on transverse sections of DD9 alloy with [001], [011], and [111] orientations. For [001] orientation (Fig. 1a), the as-cast structure exhibits a regular dendritic pattern on the section perpendicular to the crystal growth direction, with primary dendrite arms growing along [001] and secondary arms along [100] and [010], forming a well-aligned cruciform pattern. In contrast, [011] orientation (Fig. 1b) shows dendrites tending to align linearly with parallel dendrite columns of unequal arm lengths. The [111] orientation (Fig. 1c) displays irregular dendritic patterns with asymmetric secondary arms intersecting at approximately 60° angles, creating parallel secondary dendrite arms between primary trunks.

[Figure 2: see original paper] presents the heat-treated microstructures on sections perpendicular to the growth direction. The γ phase morphology varies by orientation: square for [001], rectangular for [011], and polygonal for [111].

3.2 Tensile Properties of [001], [011], and [111] Oriented DD9 Alloy

Typical stress-strain curves at 760 and 1100 °C are shown in [Figure 3: see original paper]. At 760 °C, the [001] oriented alloy exhibits rapid stress increase after yielding until fracture. The [111] orientation shows gradual flow stress increase with deformation, followed by slow stress decrease after reaching ultimate strength. The [011] orientation displays a serrated stage after yielding before reaching maximum stress, after which stress drops rapidly. This characteristic, also observed in René N4 alloy [**?**], is attributed to single slip system activation during tensile deformation. At 1100 °C, stress levels decrease overall, with [111] orientation showing maximum ductility. The [001] orientation exhibits a gradual stress decline after peak stress, similar to behavior at 760 °C, while [011] orientation shows the fastest stress drop.

[Figure 4: see original paper] summarizes the tensile properties. Ultimate tensile strength and yield strength decrease with increasing temperature ([Figure 4: see original paper]a, b). Except for [001] orientation having slightly lower yield strength than [011] at 1100 °C, [001] orientation demonstrates higher tensile and yield strengths than [011] and [111] orientations. At 760 °C, [111] orientation exhibits better tensile properties than [011], but this reverses at 1100 °C where [011] outperforms [111]. The anisotropy in tensile and yield strengths weakens with increasing temperature, consistent with Dalal et al. [**?**] who reported disappearance of tensile anisotropy at 1093 °C.

Elongation and reduction of area ([Figure 4: see original paper]c, d) show consistent trends: lowest for [001], intermediate for [011], and highest for [111]. As with other single crystal superalloys, [001] oriented DD9 exhibits low elongation and reduction of area at 760 °C, which increase significantly at 1100 °C. The [011] and [111] orientations show minimal temperature dependence in ductility parameters.

3.3 Fracture Morphology of [001], [011], and [111] Oriented DD9 Alloy

[Figure 5: see original paper] shows fracture surfaces and longitudinal morphologies after tensile testing at 760 °C. Both [001] and [011] orientations exhibit elliptical fracture surfaces with river patterns (Figs. 5a1, b1). Longitudinal sections (Figs. 5a2, b2) reveal wedge-shaped fracture planes whose normals form 50-60° angles with the stress axis. The [011] specimen shows significant necking, attributed to its four equivalent {111}<110> slip systems—the fewest independent slip systems among the orientations—resulting in lower deformation resistance and coordination, forcing crystal rotation and substantial deformation. The [111] orientation (Figs. 5c1, c2) also shows a wedge-shaped fracture composed of multiple intersecting slip planes, indicating activation of at least two dominant slip systems. Overall, all three orientations exhibit quasi-cleavage fracture characteristics at 760 °C.

At 1100 °C ([Figure 6: see original paper]), all three orientations display cup-cone macroscopic fractures with obvious necking and dimple features. Dimples connect through tear ridges or form secondary cracks upon meeting, similar to DD6 alloy fracture morphology [?]. The [011] orientation shows severe elliptical distortion due to fewer effective slip systems, lower deformation resistance, and poor coordination, likely associated with lattice rotation during deformation—a phenomenon also observed in SRR9 single crystal alloy [?, ?].

3.4 Dislocation Morphology of [001], [011], and [111] Oriented DD9 Alloy

Dislocation structures were analyzed for specimens tested at 760 and 1100 °C. [Figure 7: see original paper] presents TEM images of dislocation configurations. At 760 °C (Figs. 7a, c, e), all three orientations show high dislocation densities in matrix channels, with stacking faults appearing only in γ particles of [001] orientation, similar to other single crystal superalloys [?, ?]. No stacking faults were observed in [011] and [111] orientations.

At 1100 °C, [001] and [111] orientations accumulate dense dislocation networks in both matrix channels and γ particles (Figs. 7b, f), resulting from activation of multiple slip systems at elevated temperature. The [011] orientation at 1100 °C shows numerous twin-like bands, identified as deformation twins formed during deformation [?, ?, ?]. Although FCC metals typically have sufficient independent slip systems to avoid twinning, the limited slip system activation in [011] orientation makes twinning a necessary complementary deformation mechanism.

4. Discussion

4.1 Effect of Orientation on DD9 Alloy Microstructure

The dendritic structure observations reveal that when the heat flow direction parallels [001], primary dendrites form aligned arrays with short, thick secondary arms. Solidification latent heat dissipates slowly due to perpendicular orientation of secondary arms relative to the solidification direction and is affected by high solute concentration in interdendritic regions. For [011] orientation, both [001] and [010] dendrites form 45° angles with the solidification direction, providing equal thermal gradient advantages for both to develop as primary trunks. When these meet during solidification, solute rejected by the faster-growing arm enriches the solidification front of the slower arm, impeding its growth and causing annihilation [**?**], resulting in intersecting dendrite trunk morphologies.

For [111] orientation, three <001> dendrite directions share identical angles with the solidification direction, giving equal growth advantages. Consequently, dendrite trunk networks develop within three {100} planes, forming a hexagonal "cage-like" dendritic structure in [111]-oriented single crystals [**?**]. Thus, differences in the number of crystallographically equivalent dendrite growth directions produce the distinct dendritic morphologies observed among [001], [011], and [111] orientations.

After standard heat treatment, the γ phase should become regular cubes. The different cross-sectional morphologies arise from section orientation relative to the crystal axes. [Figure 8: see original paper] schematically illustrates this: [001] specimen cross-sections are (001) planes showing square γ , [011] sections are (011) planes showing rectangular γ , and [111] sections are (111) planes showing polygonal γ , consistent with [Figure 2: see original paper].

4.2 Effect of Orientation on DD9 Alloy Tensile Properties

With increasing temperature, DD9 alloy shows decreased tensile and yield strengths but increased ductility. At 760 °C, limited atomic diffusion forces dislocations to shear γ particles, creating substantial resistance and strong strain hardening with few active slip systems, resulting in quasi-cleavage fracture, high strength, and low ductility. At 1100 °C, enhanced atomic diffusion enables dislocations to bypass γ particles, reducing resistance. Thermal activation promotes multiple slip system operation, producing dimple fracture, lower strength, and higher ductility.

During tensile deformation, [001] oriented DD9 has eight octahedral slip systems with identical Schmid factors, facilitating deformation through multiple slip and providing high deformation resistance, thus exhibiting superior tensile and yield strengths. The [011] orientation also has four equivalent octahedral slip systems, but these reside on two conjugate slip planes. Since the initial slip plane and direction form 54.7° and 60° angles with the tensile axis (both >45°), lattice rotation from initial slip causes geometric softening of the active system while geometrically hardening the conjugate system, resulting in lower tensile strength [**?**]. The [111] orientation exhibits cubic slip characteristics [**?**], with yield strength determined by the critical resolved shear stress of cubic slip systems. However, cubic slip distributes deformation uniformly, conferring high elongation.

The tensile tests demonstrate strong anisotropy at 760 °C that diminishes at 1100 °C. Enhanced thermal activation at higher temperatures increases active slip systems, reducing anisotropy. The serrated stress-strain curve for [011] orientation at 760 °C ([Figure 3: see original paper]) relates to twinning activation. Deformation twinning requires higher stress than slip, but the limited slip systems and low symmetry in [011] orientation at low temperatures impede slip. When stress accumulates sufficiently, deformation twins nucleate. Since nucleation stress exceeds propagation stress, twin formation causes sudden load drops, creating serrations. Continuous twin formation during deformation produces the characteristic serrated curve. Twinning causes lattice reorientation, bringing some slip systems into favorable orientations for subsequent slip, resulting in smooth curve segments following serrations.

5. Conclusions

1. On sections perpendicular to the crystal growth direction, [001], [011], and [111] oriented DD9 alloys exhibit different as-cast dendritic morphologies: dendritic for [001], linearly aligned for [011], and irregular for [111]. These differences arise from variations in the number of crystallographically equivalent dendrite growth directions.
2. After heat treatment, the γ phase in [001], [011], and [111] oriented DD9 alloy becomes cubic. On transverse sections, γ appears square in [001], rectangular in [011], and polygonal in [111] orientation.
3. With increasing temperature, tensile and yield strengths decrease while anisotropy weakens. Except for [001] orientation having slightly lower yield strength than [011] at 1100 °C, [001] oriented DD9 exhibits higher tensile and yield strengths than [011] and [111] orientations.
4. At 760 °C, fracture surfaces of [001], [011], and [111] oriented DD9 show quasi-cleavage characteristics, while at 1100 °C they exhibit dimple fracture. The [011] orientation, with fewer effective slip systems and poor coordination, undergoes significant lattice rotation.
5. At 760 °C, high dislocation densities exist in matrix channels of all orientations, with stacking faults appearing only in γ particles of [001] orientation. At 1100 °C, multiple slip systems activate, producing dense dislocation networks in matrix channels and γ particles of [001] and [111] orientations, while [011] orientation develops extensive deformation twin bands.

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