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# Effect of porosity and eutectics on the high-temperature low-cycle fatigue performance of a nickel-base single-crystal superalloy

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ABSTRACT

This work investigates the separate influence of porosity and γ/γ′-eutectics on the low-cycle fatigue life of a single-crystal Ni-base superalloy at high temperatures. A conventional vacuum furnace heat-treatment but also integrated heat-treatments in a hot isostatic press are applied to produce different material variants of the same alloy. High-resolution electron microscopy revealed that both pores and γ/γ′-eutectics act as crack starters, thus initiating early failure. Moreover, the results indicate that remaining γ/γ′-eutectics can weaken the fatigue resistance even more than pores. Furthermore, the results confirm the beneficial effect of proper integrated hot isostatic pressing heat-treatments on the fatigue performance.


Damage of Ni-base single-crystal (SX) superalloys that is related to fatigue at high temperatures depends on surface condition and microstructure [1–3]. Internal microstructural heterogeneities such as pores or precipitates act as stress concentration areas and preferred crack initiation zones during cyclic loading, leading to premature rupture [4–7]. Partitioning of alloying elements between dendritic (D) and interdendritic (ID) areas during Bridgman-solidification leads to heterogeneities in the ID regions such as large irregular shrinkage pores and large γ′-precipitates that form at the end of solidification, which are described as γ/γ′-eutectic (eutectic) or peritectic [8, 9]. In order to reduce heterogeneities and to improve the mechanical properties of SX after casting, hot isostatic pressing (HIP) is applied to heal porosity, and a subsequent homogenization treatment dissolves the eutectics and reduces chemical in-homogeneities between D and ID areas. Finally, a controlled thermal treatment provides the well-known small-scale γ/γ′-morphology [1]. There is agreement, that the afore mentioned heterogeneities downgrade the low-cycle fatigue (LCF) properties of SX [4–7, 10–12]. However, the separate role of porosity and of the eutectics is not considered adequately in the literature, and also a beneficial effect of HIP on high-temperature LCF of SX was only emphasized for temperatures up to 700 °C [12, 13]. A novel type of heat-treatment that combines HIP, homogenization and quenching allows integrated heat-treatments (IHT) under pressure. Simultaneously, the chemistry and the γ/γ′-morphology of the SX material becomes uniform whereas the porosity is reduced [14, 15]. This type of IHT has been shown to prolong the creep life of an SX and its potential regarding rejuvenation [16, 17]. The IHT in the HIP opens up new possibilities because temperature, pressure, and cooling rate can be controlled, which means that different microstructures and their particular influence on LCF of SX can be investigated systematically. One microstructural feature is the presence of eutectics that can remain as a result of insufficient homogenization temperatures. This is a key factor for alloys such as CMSX-4 and CMSX-10, in which large amounts of eutectics are present in the as-cast microstructure [18, 19]. The aim of this work is to explain the influence of an IHT on the LCF performance of the second generation SX ERBO/1 and to discern the influence of porosity and eutectics. High-resolution field-emission scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) are employed for the microstructural investigations.

The LCF performance of three different material states of the same alloy is compared:

- Conventional vacuum furnace heat-treated SX having high porosity and a small amount of eutectics, denoted as ERBO/1C
- HIP-heat-treated (IHT) SX having almost no porosity but many eutectics, denoted as ERBO/1-IHT-E
- HIP-heat-treated (IHT) SX with almost no porosity and no eutectics having a virtually defect-free microstructure, denoted as ERBO/1-IHT

The initial material for all states is the CMSX-4 type alloy ERBO/1A (A: as-cast condition). After the conventional heat-treatment in a

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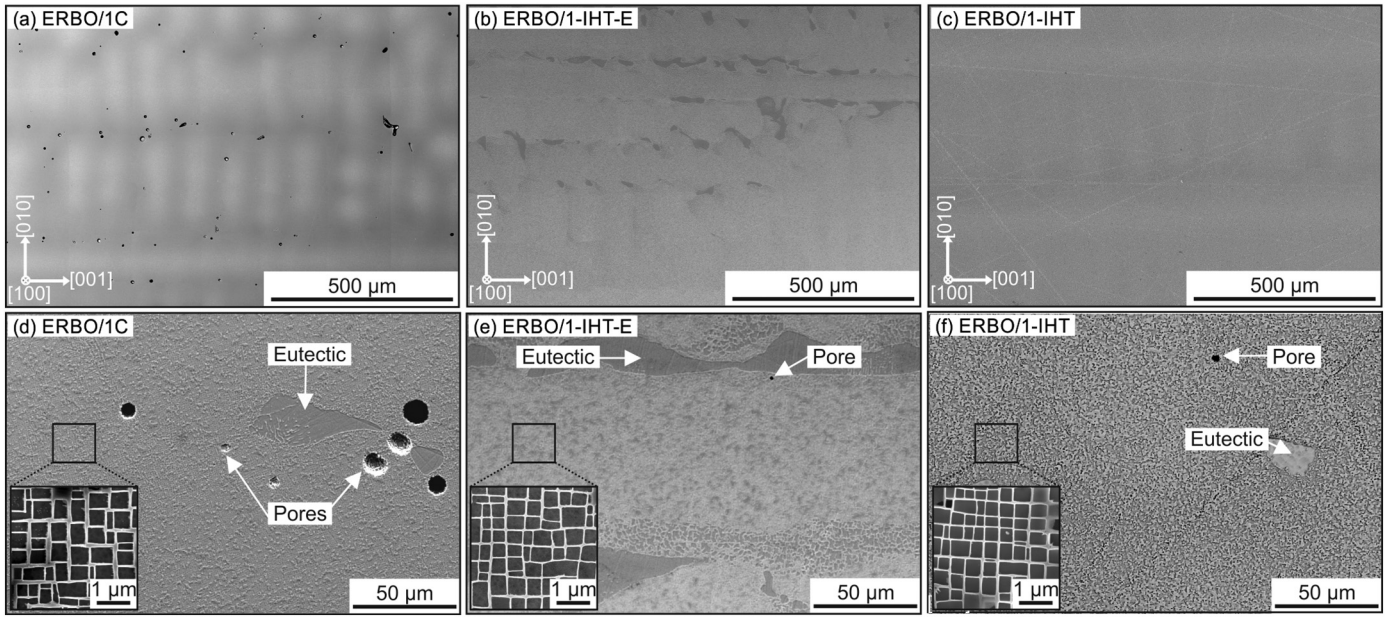


**Fig. 1.** BSE overview image montages showing (a) the microstructure of ERBO/1C, (b) the microstructure of ERBO/1-IHT-E, and (c) the microstructure of ERBO/1-IHT. (d)–(f) show magnified BSE and InLens micrographs: the microstructure of (d) ERBO/1C, (e) ERBO/1-IHT-E, and (f) ERBO/1-IHT.

vacuum furnace (homogenization + aging) the material is referred to as ERBO/1C. All details regarding the chemical composition, homogeneity, microstructure, and creep properties of ERBO/1C are described elsewhere [20]. The initial material for ERBO/1-IHT and ERBO/1-IHT-E was taken from the same ERBO/1A cast-plate segment. The IHT of ERBO/1-IHT-E and ERBO/1-IHT was conducted in a novel HIP (Quintus Technologies) that provides high quenching rates. This has several advantages: With this treatment, it is possible to homogenize the as-cast SX and to simultaneously close both the pre-existing pores and those evolving during heat-treatment. Additionally, the high quenching rate is essential for achieving at low temperature, after HIP-homogenization, a supersaturated solid-solution for the subsequent precipitation-hardening. Thus, a fine and uniform γ/γ′-microstructure can be achieved after one processing step integrated within the HIP. The main difference between the heat-treatments of the two IHT material states was the temperature during homogenization in the HIP. In order to produce a nearly pore-free microstructure and a considerable amount of eutectics (E), the material denoted as ERBO/1-IHT-E was homogenized at 1290 °C. The γ′-solvus temperature for ERBO/1 is 1285 °C [14]. To produce a virtually defect-free microstructure (without eutectics and pores) the homogenization temperature for ERBO/1-IHT was set to 1310 °C. Both materials were homogenized for 6 h at 100 MPa pressure. The quenching rate after homogenization for both HIP-cycles was similar (43 K/s measured in the gas near the specimen) because the difference in temperature (1290 °C, 1310 °C) is minimal and the applied pressure was the same [14, 17]. All details regarding the applied IHT for SX and its benefits on creep properties have been published recently [14–17].

LCF tests were performed until rupture, using miniature specimens with a cylindrical gauge length of 6 mm and a diameter of 2 mm. The total height of the specimens is 20 mm and they were clamped by threads. Precisely oriented SX specimens in the crystallographic [001] direction were prepared using the Laue technique in combination with electro discharge machining similar as reported in [21] for miniature creep specimens. The final specimen contour was turned, and the surface stayed as machined. LCF tests were conducted under load control in air at 950 °C using a servo-hydraulic testing machine. The waveform was triangular with a frequency of 0.25 Hz and a stress ratio of $R \approx 0.6$. Two different stress levels were considered (mean stresses 550 and 590 MPa) while the stress amplitude was kept constant at 130 MPa.

Specimen for SEM (Leo 1530 VP, Ultra 55, Zeiss) analysis were sectioned parallel to the longitudinal axis and prepared by metallographic preparation procedures as described in [14]. The amount of porosity and eutectics before LCF (area %, density, average diameter) was examined in a {100} plane, parallel to the D growth direction [14, 15]. EBSD was performed using the EBSD unit of TexSem Laboratories. Evaluation of the collected EBSD raw-data was supported by OIM Data Collection software.

The initial microstructures mainly differ in two aspects: the amount of porosity and the amount of eutectics. Only a slight difference can be found in the size of the γ/γ′-morphology. Fig. 1 shows microstructural overviews of larger and smaller-scales. The corresponding microstructural parameters relating to the porosity and eutectics are shown in Table 1. Fig. 1a–c compares SEM back-scatter electron (BSE) overview sections from conventional vacuum heat-treated ERBO/1C and both IHT HIP materials. The dendrites appear in a lighter gray than ID regions. Fig. 1a shows pores appearing as black spots in the corresponding micrograph of ERBO/1C. The pores line up alongside the primary dendrites and between the dendrite arms. After HIP, the two material variants exhibit almost no porosity. The corresponding micrograph of ERBO/1-IHT-E (Fig. 1b) shows large amounts of dark shaded areas that mark the remaining eutectics, which are located in the ID areas. The associated micrograph of ERBO/1-IHT (Fig. 1c) is almost free of both pores and eutectics. Fig. 1d–f compares the microstructures of each specimen at a higher magnification. ERBO/1C (Fig. 1d) contains significantly more and larger pores than both materials after HIP. Larger, irregularly shaped pores that evolved during solidification co-exist with smaller spheroidal homogenization pores. The summarized pore-values in Table 1 show that the IHT is very efficient in reducing porosity in both material states, ERBO/1-IHT-E and ERBO/1-IHT. The ID area of ERBO/1C contains only

**Table 1**
Size and area fraction of pores and eutectics of the tested materials.

| Material | Porosity | | | Eutectic | | |
|---|---|---|---|---|---|---|
| | Area | Quantity | Mean Ø | Area | Quantity | Mean Ø |
| | % | $mm^{-2}$ | μm | % | $mm^{-2}$ | μm |
| ERBO/1C | 0.3881 | 48.9 | 10.3 | 0.161 | 6.4 | 25 |
| ERBO/1-IHT-E | 0.0004 | 1.8 | 2.9 | 3.124 | 47 | 48 |
| ERBO/1-IHT | 0.0006 | 1.7 | 2.8 | 0.002 | 0.7 | 8 |