ENVIRONMENTALLY ASSISTED BRITLLE FRACTURE OF NICKEL-BASE SUPERALLOYS AT HIGH TEMPERATURES

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ABSTRACT

Brittle intergranular cracking of nickel-based superalloys has been observed in many cases to be a consequence of superimposing high-temperature exposure and a high tensile load. In the present paper, hold-time cracking of the Ni-based superalloy IN718 at 650°C and quench cracking of the Ni-base superalloy René 95 were studied applying tensile tests under fixed displacement conditions in various environments. In both cases, testing in air yielded very high cracking rates, up to 50µm/s, while in vacuum crack propagation was substantially slower and mainly determined by power law creep or ductile rupture. The governing damage mechanism for this kind of environmentally-assisted brittle crack propagation was recognized as "dynamic embrittlement", i.e., oxygen grain-boundary diffusion into the elastic stress field ahead of the crack tip followed by deformation-less intergranular decohesion. Load-relaxation experiments on bicrystalline and thermomechanically-processed 4pt-bending specimens support the observation that the susceptibility to dynamic embrittlement depends substantially on the crystallographic orientation relationship of the affected grain boundaries.

1 INTRODUCTION AND THEORETICAL BACKGROUND

Polycrystalline nickel-based superalloys are typically used for aero-engine applications, such as gas-turbine discs or blades. In order to withstand high creep and fatigue loading these alloys are strengthened by $\gamma'$and/or γ'' precipitates. Due to their high strength, Ni-based superalloys are prone to oxygen-induced brittle cracking, either during rapid quenching from the solution-heat treatment temperature or during dwell times in tension under in-service conditions; this is known as *hold-time* cracking. It has been reported by Browning et al. [1] and Hayes et al. [2] that water vapour in the atmosphere, probably acting as an additional source of atomic oxygen at high temperatures, has an additional detrimental effect on the degradation of the mechanical properties of Ni-base superalloys at high temperatures.

The latter crack-propagation phenomenon is often attributed to oxidation effects, termed as stress-accelerated grain-boundary oxidation (SAGBO) [3]. In the case of IN718 it is assumed that an enrichment in niobium at the grain boundaries (GBs) leads to a fast formation of a massive Nb-oxide layer along the GBs, which fail in a brittle manner when a tensile load is applied. Since oxidation rates of Ni-base superalloys, at least at low temperatures, are extremely small [4], it is more likely that the GBs are weakened by inward-diffusing oxygen. Molins et al. [5] and Andrieu et al. [6] suggest an alternative mechanism where GB oxidation ahead of the crack tip in an early epitactic state leads to vacancy injection and therefore eased cracking of the affected grain boundary.

A catastrophic decrease in fracture toughness was found by Chang [7] in the form of quench cracking of René 95. Instead of ductile rupture, specimens failed by brittle intergranular fracture throughout the cross section without any indication of plastic deformation. It is hard to believe that this kind of very fast intergranular cracking is due to the formation of thermally-grown oxides along the GBs.

A reasonable explanation for environmentally-assisted brittle fracture is found in the *dynamic-embrittlement* mechanism [8]. As schematically shown in Fig. 1, an embrittling species, either provided from the surrounding environment or a surface-active element from the material itself (e.g. in the case of S-induced stress-relief cracking in steel [9]), can diffuse into grain boundaries that are exposed to high tensile stresses acting perpendicular to the crack/GB faces. This is analogous to the diffusive cavity growth model according to Hull and Rimmer [10]. Penetration of the embrittling species into the grain boundary lowers the interfacial cohesion and, hence, results in crack advance.

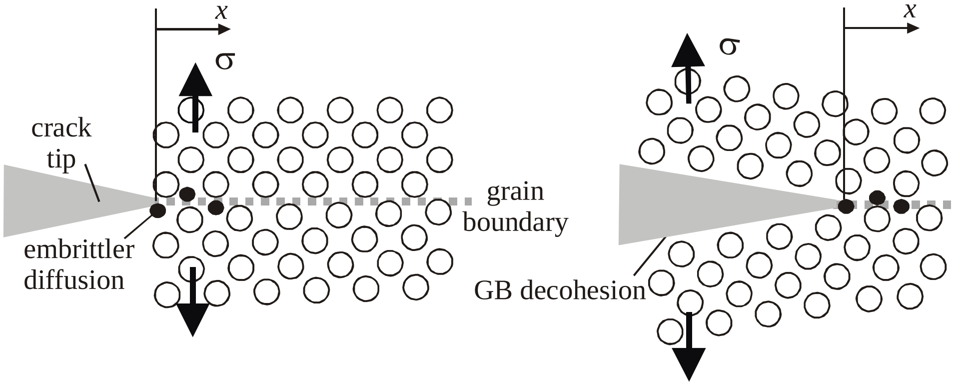


Figure 1: Schematic representation of the dynamic-embrittlement mechanism: (a) diffusion of an embrittling element into a stressed GB and (b) crack advance by stepwise GB decohesion.

Generally, dynamic embrittlement can be described by the governing equation for stress-assisted GB diffusion

$$\frac{\partial c}{\partial t} = D_{\mathrm{GB}} \frac{\partial^2 c}{\partial x^2} - \frac{D_{\mathrm{GB}}\Omega}{kT} \frac{\partial}{\partial x}\left(c \frac{\partial \sigma}{\partial x} \right) \tag{1}$$

where c is the concentration and D_{GB} the GB diffusion coefficient of the embrittling agent, σ the tensile stress ahead the crack tip and Ω the atomic volume of the bulk material. By solving this partial differential equation in a simplified form [11], two important conclusions can be drawn: (1) cracking by dynamic embrittlement occurs within a process zone that lies within a distance of a few nm ahead of the crack tip, and (2) the main parameters affecting crack propagation are the stress level and the grain-boundary diffusivity in the cohesion zone ahead of the crack tip. Therefore, the local microstructure, i.e., geometry and crystallographic orientation relationships, should play a major role for the susceptibility to environmentally-assisted brittle crack propagation at elevated temperatures.

2 EXPERIMENTAL DETAILS

Two commercial Ni-based superalloys, IN718 and René 95, with a chemical composition according to Table 1, were used to study cracking by dynamic embrittlement. Pre-cracked and completely heat-treated IN718 single-edge-notched bending specimens (SENB) were tested under fixed-displacement conditions at 650°C, which corresponds to the typical in-service temperature of gas-turbine discs, and various oxygen partial pressures in a vacuum chamber. Crack-propagation rates *da/dt* were evaluated by use of compliance calibration curves, i.e., fixed-displacements tests were interrupted and the crack length was correlated with the corresponding load level.

To study the phenomenon of intergranular brittle cracking during quenching of René 95, cylindrical notched tensile specimens were heated to the typical solution heat treatment temperature of 1175°C in an INSTRON mechanical testing machine at approximately zero stress.

Subsequently, the specimens were cooled down rapidly in air and in vacuum, holding the grips fixed in place.

Additional fixed-displacement experiments were carried out on IN718 bicrystals and thermo-mechanically-processed IN718 specimens, exhibiting a two-times-higher fraction of CSL low-Σ grain boundaries than the as-received material (for more details see [12]).

Microstructure and fracture surfaces were studied by means of scanning electron microscopy in combination with automated electron back-scattered diffraction (EBSD and orientation imaging microscopy OIM™).

Table 1: Chemical compositions and heat treatment (IN718) of the materials used in this study (in wt.%)

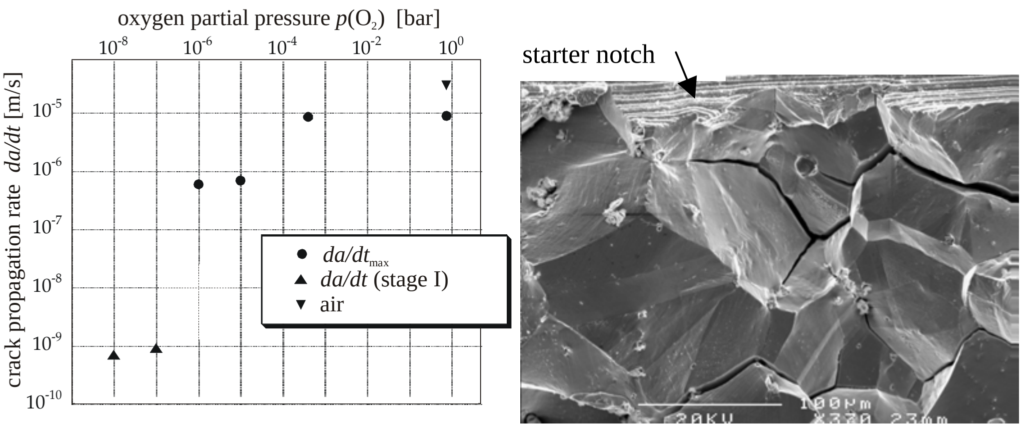
	Ni	Fe	Cr	Nb	Mo	Ti	Al	Co	W	C
IN718	Bal.	18.7	18.2	5.2	3.0	1.0	0.5	0.1	--	0.04
René 95	Bal.	--	12.9	3.3	3.4	2.4	3.6	7.8	3.4	0.06
Solution heat treatment (IN718):				1050°C (1h) water-quenched						
Ageing (IN718)				720°C (12h) furnace-cooled						
				620°C (12h) air-cooled						

3 RESULTS AND DISCUSSION

3.1 Oxygen-Induced Quasi-Brittle Crack Propagation in IN718

Load-relaxation experiments on 4pt-bending specimens of IN718 at 650°C revealed very high cracking rates up to 50µm/s for oxygen partial pressures above approximately $p(O_2)=10^{-4}$bar (Fig. 2a, ref. [13]). As it can be seen clearly in Fig. 2b, the prevailing part of the corresponding intercrystalline fracture surfaces do not show any apparent indication of plastic deformation or oxidation attack.

For lower oxygen partial pressures there is a substantial contribution of power-law creep to dynamic embrittlement occurring in a stepwise manner interrupted by long time intervals during which oxygen is build up at the crack tip resulting in very low crack-propagation rates. In fact, no pronounced load drop was observed any more for tests carried out at $p(O_2)<10^{-6}$bar.



a **b**

Figure 2: (a) Maximum crack propagation rates during load relaxation of IN718 4pt-bending specimens as a function of the oxygen partial pressure at 650°C (ref. [13]) and (b) intergranular fracture surface in the vicinity of the starter notch of an IN718 SENB specimen.

Taking these results into consideration (for details see [13]), it seems reasonable to postulate that fast and deformation-less separation of grain boundaries is a consequence of interfacial oxygen diffusion, leading to an embrittlement of the cohesion zone ahead of the GBs. Since the embrittled zone propagates as the crack advances this damage process has been termed "dynamic embrittlement".

From literature (e.g. [14]) it is known that GB diffusion is strongly affected by the GB structure. GB diffusion coefficients are particularly low for low-angle GBs and CSL GBs with a high fraction of coincident lattice sites of the neighbouring grains, i.e., low Σ values. Since dynamic embrittlement is governed by GB diffusion, it can be assumed that specimens with a high fraction of CSL GBs exhibit a high resistance against brittle intergranular fracture. Figure 3a shows the load relaxation curves of IN718 4pt bending specimens in the as received (AR) and in a grain-boundary-engineering-type thermomechanically processed condition (TMP, 4 sequences of 20% cold rolling followed by a 1h anneal at 1050°C). TMP resulting in a 100% increase of CSL GBs, as shown by OIM™, led to a strong increase of the incubation time before the onset of fast intergranular crack propagation and to a decrease of the maximum crack-propagation rate *da/dt* by one order of magnitude. This result was confirmed by preliminary fixed-displacement tests on IN718 bicrystalline 4pt-bending specimens, the results of which are depicted in Fig. 3b. It is obvious that the resistance of a special CSL GB - in this case a symmetrical Σ5 (031)[001] tilt boundary - is substantially higher than the resistance of a random high-angle grain boundary.

A further quantification of the significance of the grain boundary structure for cracking by dynamic embrittlement, and also for cyclic-loading conditions is a subject of ongoing research.

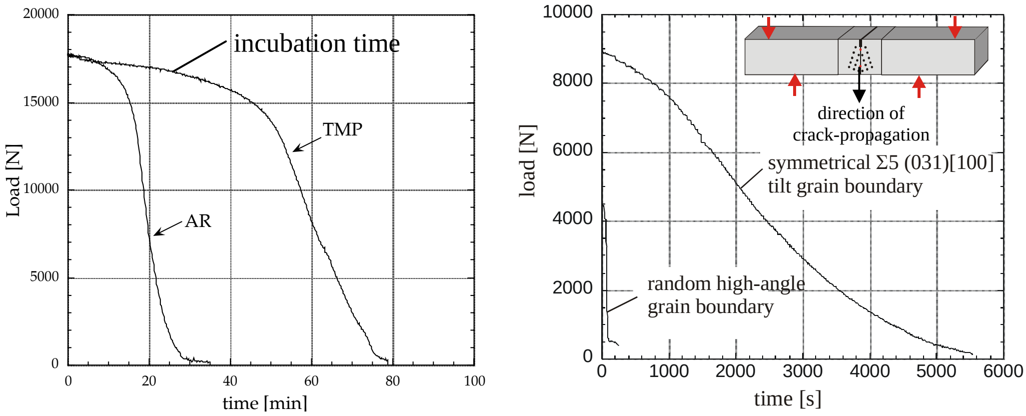


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Figure 3: Load relaxation due to quasi-brittle crack propagation vs. time at 650°C for (a) IN718 SENB specimens in the as-received and thermomechanically-processed condition, and (b) for IN718 bicrystalline SENB specimens with a random high-angle and a special Σ5 grain boundary.

3.2 Quench Cracking of René 95

Recent results on the quench-cracking phenomenon observed for the Ni-base superalloy René 95 [15] support the postulate that oxygen-induced brittle cracking involves neither the formation of oxide products nor long-range oxygen diffusion within the cohesive zone ahead of an intergranular crack tip. Fig. 4a shows the stress increase within a notched René 95 tensile specimen during fast-cooling in air from 1175°C while the grips were hold in place. Fast crack propagation set in suddenly when the load exceeded a critical value of approx. 2950 lbs, continuing only for a few seconds until the specimen failed completely. The fracture surface shown in Fig. 4b reveals that the initial cracking mode was intercrystalline to a large extent without plastic deformation to be involved.

Even though, the initiation phase of intercrystalline cracks might have started already during the quench-induced load increase, the mechanism of crack propagation seems to be the same as proposed in section 3.1 for the hold-time-cracking phenomenon of IN718, i.e., dynamic embrittlement. Carrying out the same experiment - cooling a notched René 95 tensile specimen in fixed grips - in vacuum (for details see [15]), revealed no indications of intercrystalline damage, and failure occurred completely by ductile rupture. Therefore, one can conclude that no intrinsic effects, like GB embrittlement by sulphur, is responsible for the brittle intercrystalline failure in air.

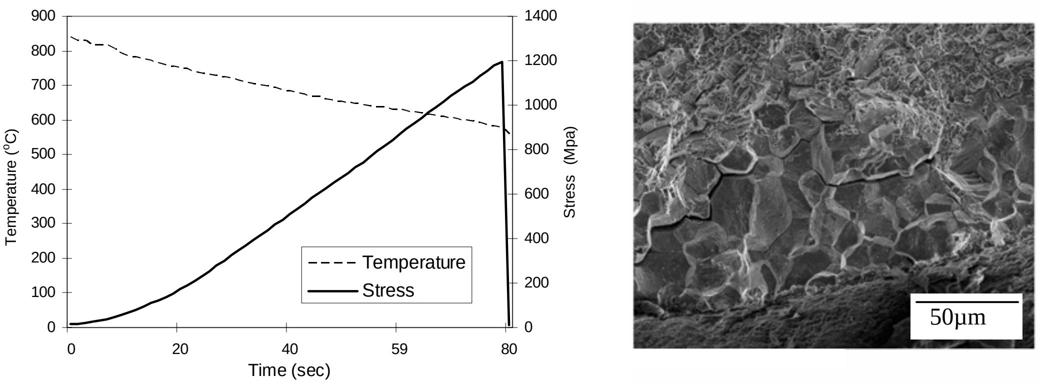

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Figure 4: (a) Temperature and stress vs. time during fixed-grip cooling of a notched tensile specimen, and (b) transition from intergranular brittle to transgranular fracture near the notch tip.

Kane et al. [15] have shown that brittle intercrystalline quench cracking can be effectively suppressed simply by nickel plating of the components before solution heat treatment. By carrying out the heat treatment in a non-oxidizing atmosphere for a period as short as possible, the Ni layer protects the surface from the embrittling reaction.

4 CONCLUDING REMARKS

Hold-time cracking during LCF loading at temperatures between 500°C and 700°C as well as quench-cracking during cooling from the solution heat treatment temperature are discussed as typical failure mechanisms of polycrystalline high-strength Ni-based superallyos, e.g., René 95 and IN718. Load-relaxation and fixed-grips experiments representing the hold-time or quenching situation revealed very high crack propagation rates, up to 50µm/s, that again depend strongly on the environment. In the case of hold-time cracking of IN718 switching to vacuum conditions leads to a transition to very slow crack propagation mainly governed by power-law creep. In air, failure generally occurs in an intercrystalline manner by brittle grain-boundary decohesion. This has been attributed to the generic damage mechanism "dynamic embrittlement", which is governed by short-range GB diffusion of the embrittling species into the cohesion zone ahead of an advancing crack tip followed by decohesion. There is evidence from results on thermomechanically-processed specimens and bicrystals that an increase of the fraction of special CSL grain boundaries can increase the resistance of a material to quasi-brittle intergranular cracking by dynamic embrittlement.

5 ACKNOWLEDGMENTS

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