Elevated temperature fatigue crack propagation in relation with crack-tip microstructure in the AD730® Nickel-base superalloy.

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CONTEXT AND OBJECTIVES OF THE THESIS

CHAIRE ANR SAFRAN OPALE

Optimisation of the polycrystalline nickel-base superalloys' properties by controlling the microstructure after processing.

PROCESSING ⟷ MICROSTRUCTURE ⟷ MECHANICAL PROPERTIES

CEMEF — PPRIME

Objectives

- **To reproduce** test conditions leading to similar crack propagation rates in air and vacuum on the **AD730**
- To investigate at 750°C the difference between **bulk microstructure aging** and **enclosed microstructure aging** at the crack tip
- To understand how do **interactions** between creep, fatigue, oxidation and dynamic microstructure aging at the **crack tip** impact crack propagation rates

The rim of aeroengine turbine disks is a highly loaded region that must be resistant to :

- **Crack nucleation**
- **Fatigue damage**
- **Creep damage**

at

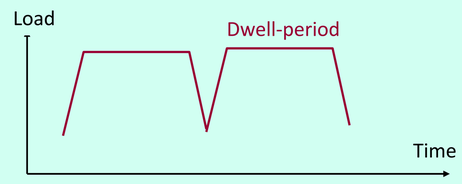
- **High temperatures (> 650°C)**
- **In an atmospheric environment**

➡ Nickel-base superalloys provide such properties

To get closer to reality

⬇

DWELL-FATIGUE tests are performed in air



Usually,

dwell-periods at max load [1]
+
oxidizing environment at high temperatures [2]
=
Increase of the crack propagation rate

BUT

At 750°C, it has been shown on Astroloy that by applying different levels of **initial ΔK value**, crack propagation rates **in air** and with dwell-periods could be similar to those **in vacuum**. [3][4]

⬇

Suggested explanations back then :

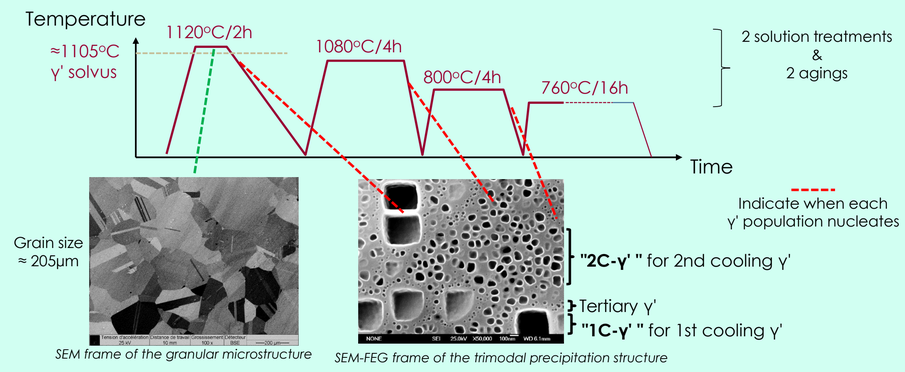
- **Stops of the crack inducing aging of the microstructure towards a better crack propagation resistant one [5]**
- **Growth of a protective layer of oxides at the crack-tip**
- **Change of the microstructure at the very crack-tip due to oxidation**

Initial framework

Test temperature : 750°C

Studied material : Nickel-based superalloy **AD730** γ/γ' (respectively matrix/precipitates)

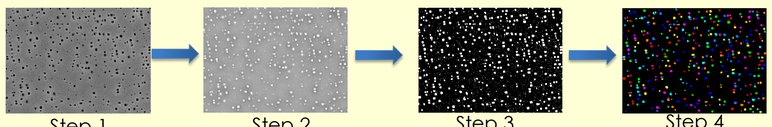
Initial microstructure : A four steps heat treatment is performed so as a coarse grained microstructure with a trimodal precipitation is obtained



SEM frame of the granular microstructure — *SEM-FEG frame of the trimodal precipitation structure*

Aging of the γ' precipitates at 750°C

Method : Image analysis via Visilog



➡ **area fraction, mean diameter and distance between particles**

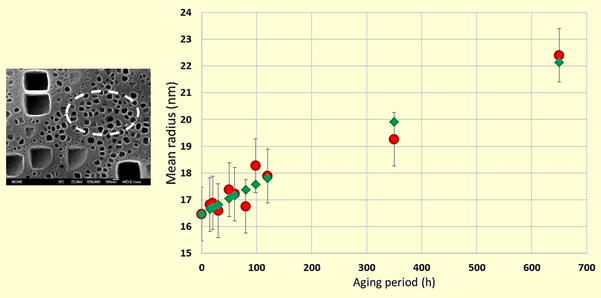
Describes the isothermal growth of small precipitates by **Ostwald Ripening** : diffusion controlled process

Common LSW Law [6]

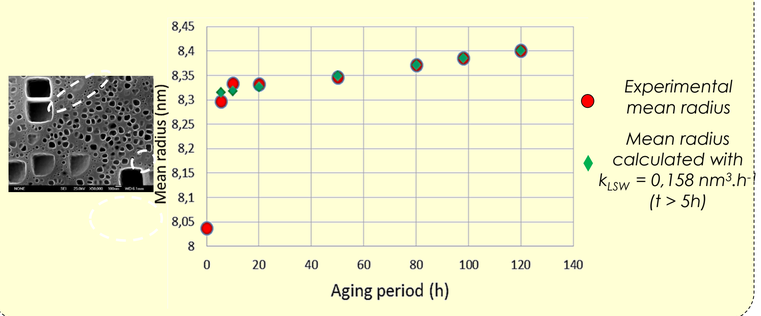
$$R^3 = kL_{SWt} + R_0^{\,3}$$

R = mean radius
R_0 = initial mean radius
t = isothermal aging time
k_{LSW} = volume rate constant

Stress-free aging of the 2C-γ'



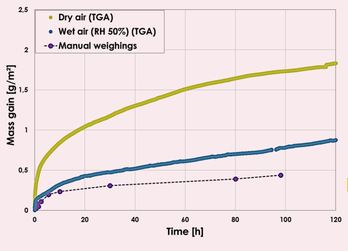
Stress-free aging of the tertiary γ'



Oxidation of the AD730 at 750°C

Thermogravimetric Analysis (TGA)

Technique to measure the time dependent mass gain due to the growth of oxide layers



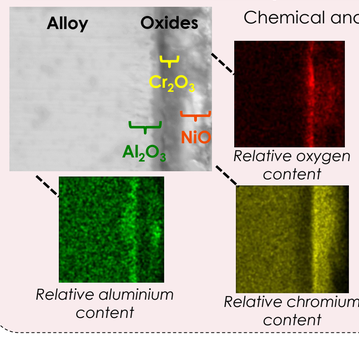
In the steady state, the oxide growth kinetic is described by

$$\left(\frac{\Delta m}{S}\right)^n = kt$$

Δm = mass gain
S = exposed surface
t = isothermal exposing time
K & n = constants

- n_{app} ≈ 2,5 for the three cases with t>20h
- The wet air test result is closer to the manual weighings : k_{app} ≈ 4,90^{E}-14 IU

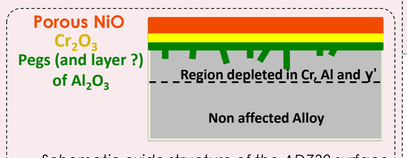
Structure of the oxide layers and changes in the microstructure below



Chemical analysis via Energy Dispersive Spectroscopy (EDS)

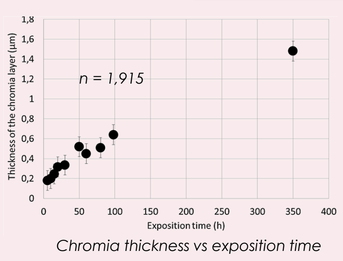
Ni	Cr	Co	Fe	Mo	W	Al	Ti	Nb	B	C	Zr
Base	15.7	8.4	4.0	3.1	2.7	2.3	3.4	1.1	0.011	0.015	0.035

Chemical composition (% mass.) of AD730 [7]

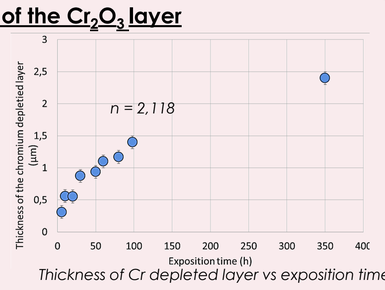


Schematic oxide structure of the AD730 surface exposed a long time at high temperature

Growth kinetic of the Cr_2O_3 layer



Chromia thickness vs exposition time



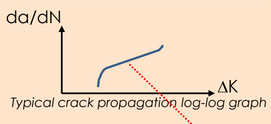
Thickness of Cr depleted layer vs exposition time

As expected for a protective oxide, the growth kinetic is parabolic, as well as the Cr depletion in the sub-layer : the chromia layer growth is diffusion controlled

Crack propagation at 750°C

Basic concepts

- da/dN



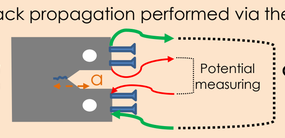
Typical crack propagation log-log graph

K = Stress intensity factor (MPa√m)
= f(stress, geometry, imperfection size)
ΔK = $K_{max} - K_{min}$ in a cyclic test

- Paris' domain : $$\frac{da}{dN} = C\Delta K^m$$

with *da/dN* the crack propagation per cycle, *m* the Paris coefficient, *C* a constant

- Measurement of the crack propagation performed via the **Potentiel Drop Method** :

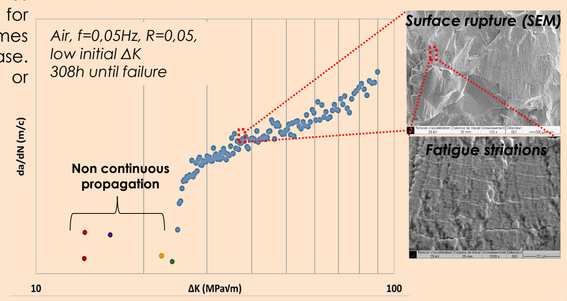


*Potentiel drop method on a Compact Tension specimen with a crack **a**.*

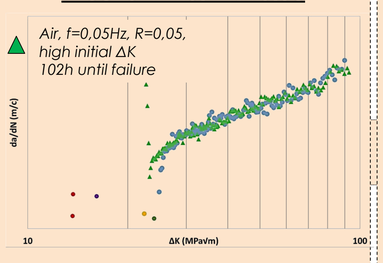
When crack length ↗, potential ↗

Referencial test

- **Micro-propagation** :
- the crack propagates
- then remains stagnant for several cycles, sometimes despite ΔK value increase. Oxydation effects or preckracking effects ?
- The crack propagates continuously and accelerates

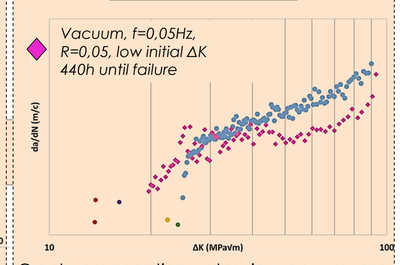


Effect of initial ΔK value



No influence of the level of initial ΔK value on the Paris' domain. The steady state propagation regime is delayed with the use of low initial ΔK value.

Effect of oxidation



Crack propagation rates in vacuum are similar to those in air. The propagation begins at the same ΔK value and is immediately continuous in vacuum.

Conclusion and outlooks

- Aging of the two smallest AD730 γ' populations at 750°C up to 120h shows a **growth of the γ' precipitates** following the mechanism of Ostwald Ripening. Aging under loading with and without a stress concentration are to be studied. We will also have to investigate the differences between the bulk aging and the enclosed crack-tip aging of the γ' precipitates.
- The oxide layers growing during exposition in air would be organized as follows : 1st **nickel oxides**, 2nd **chromium oxides** and finally **aluminium oxides**. Further analysis have to be performed to confirm these assumptions. Besides, **the γ' depletion size** in the sub-area of the oxide layers and its evolution versus time of exposition are to be determined.
- We have carried out the first crack propagations tests at 750°C on the AD730. The introduction and effects of dwell-periods at maximum load is the next step of the study. This campaign should provide tools to better understand interactions at the crack tip between creep, fatigue, oxidation and microstructure.

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[1] R. Jiang, N. Gao, M. Ward, Z. Aslam, J. C. Walker, and P. A. S. Reed, *Superalloys 2016*, pp. 11–15, 2016
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[7] A. Devaux, W. Li, C. Crozet, J.M. Lardon, *Superalloys 2016*, pp 469-477, 2016.

Acknowledgments :
Jonathan CORMIER for the lot in advice and conseul he has given to me.
Guillaume BENOIT, Florence HAMON, Jacques LEFORT, Laurent JOUVANNEAU and everyone involved in the this work..