

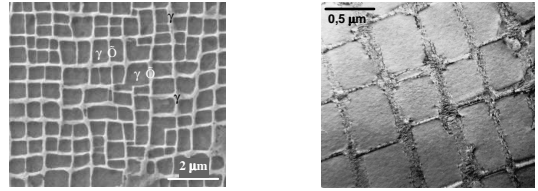
OXIDATION INDUCED MICROSTRUCTURAL EVOLUTION IN A MULTIPHASE ALLOY.

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Single-crystal nickel based superalloys are used for gas turbine blades, due to their advanced mechanical properties and oxidation resistance. The considered superalloy is based on two phase microstructure consisting of an γ fcc Ni matrix hardened by γ' $\text{Ni}_3(\text{Al}, \text{Ti}, \text{Ta})$ as cube shaped coherent precipitates. Moreover, multiphase coatings are also used to protect these superalloys for high temperature applications (TBC) in order to obtain a better efficiency and durability. The knowledge of the mechanical properties of thermal barrier coatings requires informations about the alloy oxidation behaviour. So, the objective of this study is to report the microstructural evolution of the single-crystal superalloy observed during oxidation : the oxide nature and morphology, the development of a γ' depleted subsurface, the adherence of the oxide/substrate interface according to the sulphur content of the superalloy.

Isothermal oxidation tests were carried out at 1100°C for 1h, 5h, 10h, and 50h under ambient atmosphere. The oxide formation and the substrate evolution were followed and analysed using SEM, TEM, WDS and SIMS in order to identify the oxidation mechanisms. Cross section observations show that, whatever the S content of the substrate, the oxide layer closed to the oxide/substrate interface is only composed of α alumina (under an outer layer of complex oxides) and a modified subsurface zone appears beneath the alumina scale. The thickness of the γ' depleted layer depends on the oxidation time as the consequence of the aluminium diffusion to ensure alumina growth. Between the γ' dissolution zone and the frontier of the non-affected alloy, an intermediate layer including small spherical γ' precipitates was observed. Its thickness depends again on the oxidation conditions. WDS profile analyses show an homogenisation of tungsten and chromium concentrations in the γ' dissolution zone, as well as an increase of titanium and tantalum concentrations in the spherical γ' precipitates.

In the case of the standard sulphur superalloy substrate, oxide spallation after heat treatment is macroscopically observed, which is not the case for the low sulphur substrate. In both cases, spallation was related to the growth of cavities at the oxide/superalloy interface. Moreover, sulphur segregation at this interface was investigated using SIMS for various oxidation times. Results show that sulphur concentration at the interface is four times higher in the case of the standard substrate than the low S alloy. The sulphur segregation is considered at the origin of increased cavities formation and loss of adherence.



Figures 1 and 2 : Phases γ and γ' .

	Ni	Co	Cr	Mo	W	Ta	Al	Ti	C	Fe
min.	bal	6	7	1,8	5	7,5	5,1	1		
max.	bal	7	8	2,2	6	8,5	5,5	1,4	0,01	0,2

Table 1 : Single-crystal composition in weight%.

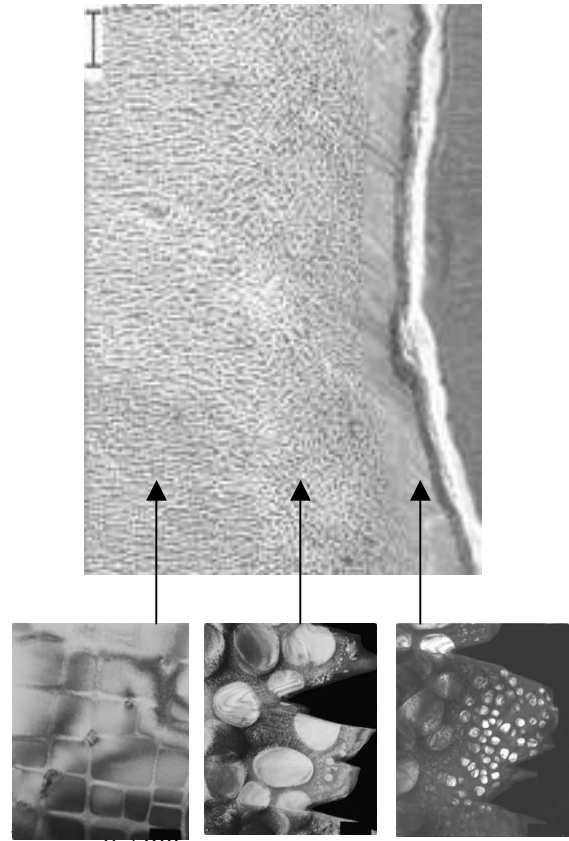


Figure 3, 4, 5, 6: Microstructural evolution after heat treatment.