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# Influence of Environment on Creep Properties of MC2 Single Crystal Superalloy at 1150°C

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**Keywords:** single crystal superalloy, creep test, controlled atmosphere, high temperature oxidation.

**Abstract.** In order to reveal the effect of oxidation on thin blade walls, a new machine allowing tests up to 1250°C under controlled atmosphere has been designed. Creep tests were performed on MC2 single crystal superalloy at 1150°C, under hydrogenated argon and dry air, but also with a switch from one atmosphere to the other after reaching the steady state creep stage. The results point out the decrease of the minimum creep rate in case of tests performed or at least started under hydrogenated argon, compared with the value obtained under synthetic dry air. This effect of oxidation was attributed to the protective oxide scale formed under hydrogenated argon. The low growth rate of alumina layer leads to a thinner zone affected by metal consumption, which is assumed to be non bearing, and prevents from vacancy flux toward the alloy. The second point results in slowing down creep mechanisms controlled by diffusion and therefore dislocation motion and microstructure evolution. Thermogravimetric tests confirm the difference of oxidation kinetics regards to environment (hydrogenated argon and dry air). However, oxide scales have different microstructures on thermogravimetric and creep samples when tested under air.

## Introduction

Ni- base single crystal superalloys have been designed for turbine blades in aeroengines since they can withstand high centrifugal loads at temperatures up to 1100°C. Many factors can damage these parts. Thus, the blades are protected by MCrAlY or aluminides coatings from high temperature corrosion and oxidation. However, creep is the main life limiting phenomenon for uncooled blades in engine certification conditions.

In order to take into account thin blade walls, new creep test specimens were designed (thin parallepipedic specimen, about 1 mm thick). First creep test results [1,2] showed that the rupture life decreases almost from a factor 2 when going from a standard test specimen geometry (massive bars) to this new sample configuration. This behaviour is usually explained as an effect of environment (oxidation and/or corrosion) on the superalloy creep rate, which is particularly significant for thin bare specimens [3,4].

For a better understanding of the oxidation effect, a new creep test machine has been designed to allow an accurate strain measurement at temperatures up to 1250°C and complex test conditions (atmosphere and temperature variations). This paper is a microstructural complementary study of the results presented in ref. [5].

## Materials and experimental procedures

**Materials.** Single crystal plates of MC2 alloy (cf. Table 1) were obtained from SNECMA foundry.

| Ni | Cr | Co | Al | Mo | Ti | W | Ta |
|---|---|---|---|---|---|---|---|
| bal. | 7.8 | 5.2 | 5 | 2.1 | 1.5 | 8 | 5.8 |

Table 1. Chemical composition of MC2 alloy (wt %)

Thin sheets of 1.2 mm of thickness were cut from these plates, subjected to the full aging heat treatment and finally ground to 1 mm. Thin creep specimens were then machined by spark erosion in the longitudinal [001] direction of these sheets. They were surface polished with SiC paper and diamond paste down to 3 µm to remove affected zone by rectification.

**Creep Tests.** Creep tests were carried out at 1150°C under hydrogenated argon (argon + 5 % vol. $H_2$) and synthetic dry air on a MTS electromechanical machine. The specimens were heated by a radiation furnace which allows a thermal gradient along the gauge length of the specimen lower than 20°C and fast heating speed. Three thermocouples were spot welded : one on the middle of the gauge length to regulate the temperature (± 2 °C) and the two others to evaluate the thermal gradient. Windows let a laser beam go through the furnace which allows measurement of the displacement between two L-flags spot welded on the gauge length. The laser accuracy is 1 µm at room temperature but 5 µm at 1150°C because of the lamps light disturbance. Specimen geometry and flags locations are shown on Fig. 1.

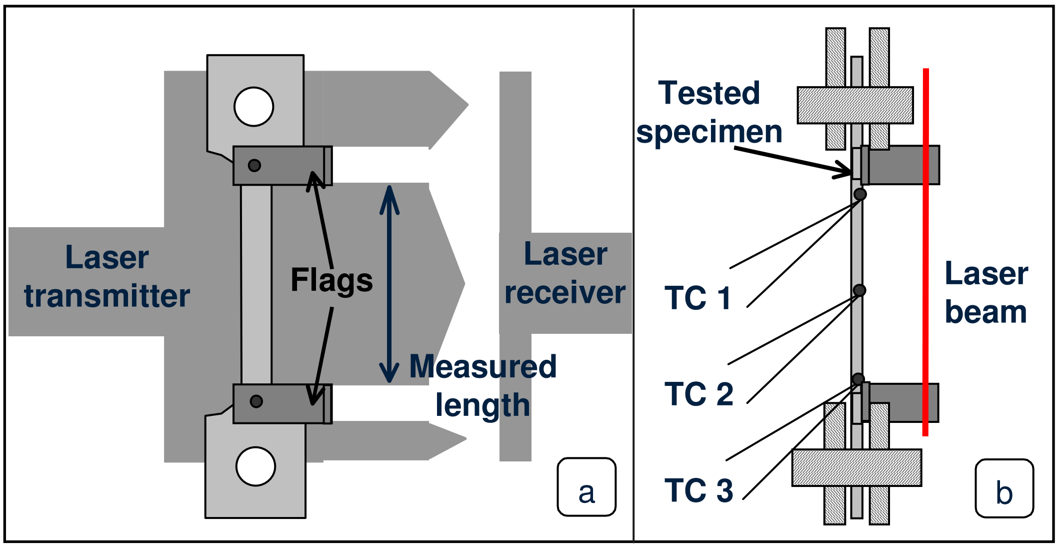

Fig. 1. Specimen geometry with flags and thermocouples location, a) Front view of the tested specimen and location of the laser beam, b) Side view of the assembly

Creep tests were carried out to rupture at 1150°C and a given stres $\sigma_0$, both under synthetic dry air and hydrogenated argon.

Dispersion on creep results may however be significant with single crystal superalloys. In order to free oneself from this dispersion, two other kinds of creep tests have been performed : the first one was started under hydrogenated argon until reaching secondary creep stage , and then the atmosphere was changed to dry air until rupture. The second one has been conducted under dry air up to the steady state creep stage and finished under hydrogenated argon. In each case, before switching the environment, a primary vacuum was made to ensure a faster transition in atmosphere.

**Thermogravimetric Tests.** Oxidation tests were performed with an automatic recording thermobalance SETARAM TAG 24S equipped with a double symmetrical furnace which provides a very stable signal and minimizes the drift error to less than 1 µg for a test duration of 24 hr. The parabolic rate constants were calculated using the method described in ref. [6]. This method also allows to estimate a rate constant during the transient oxidation stage.

## Results

**Creep results.** Fig. 2 shows the creep curves obtained at 1150°C-$\sigma_0$ MPa under dry air, hydrogenated argon and two tests with changing atmosphere. Table 2 put together all the minimum creep rates and times to rupture. Times to rupture are normalized with the time to rupture of the test performed under synthetic air and the minimum creep rates are normalized with the average creep rate of tests begun under hydrogenated argon.

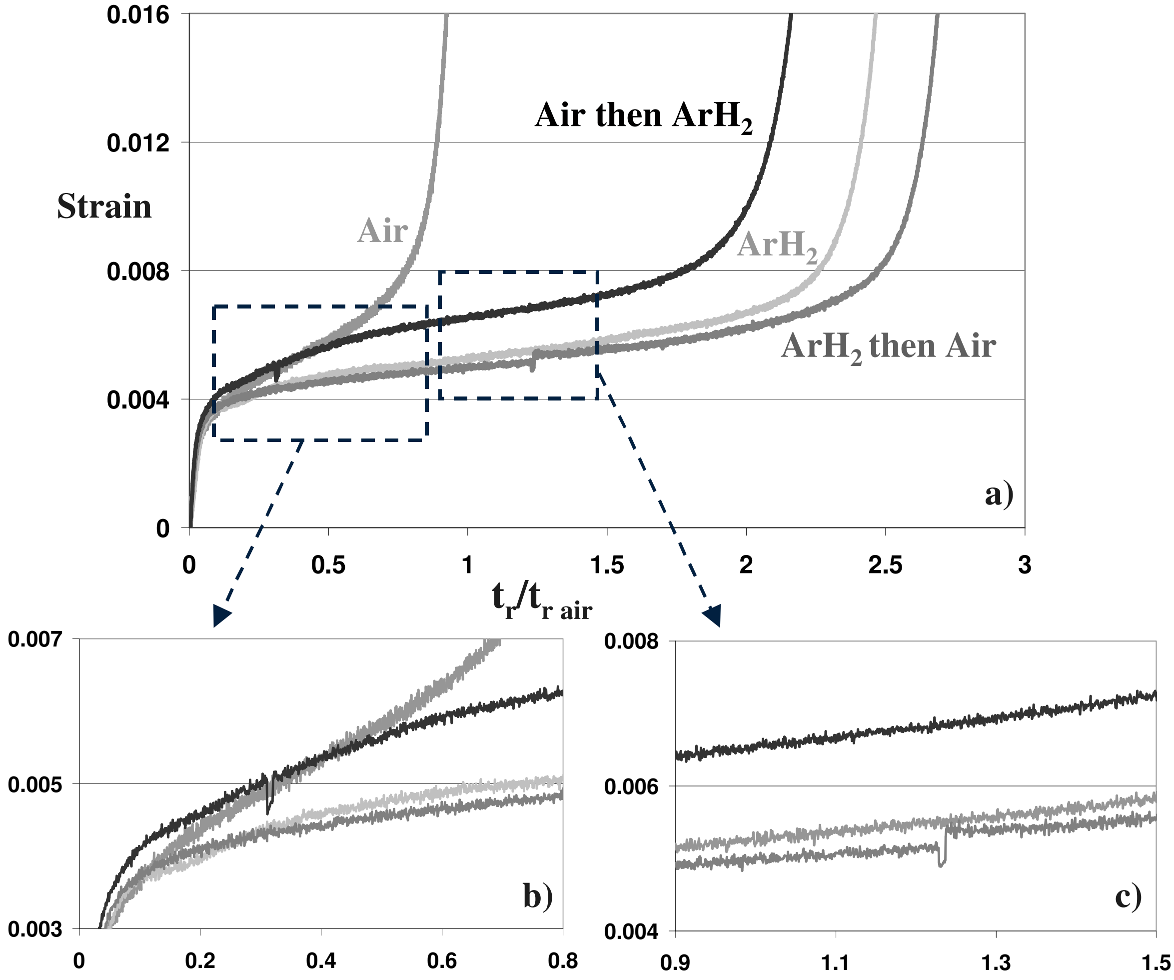

Fig. 2. Normalized creep curves obtained at 1150°C- $\sigma_0$ MPa under different atmospheres, a): complete curves, b) and c) zooms around atmosphere switches

| First Atmosphere | $\frac{\dot{\varepsilon}_{min}}{\dot{\varepsilon}_{min\ ArH_2}}$ | Second Atmosphere | $\frac{\dot{\varepsilon}_{min}}{\dot{\varepsilon}_{min\ ArH_2}}$ | $\frac{T_{rupture}}{T_{rupture\ air}}$ |
|---|---|---|---|---|
| Air | 5.9 | | | 1 |
| Air | 6.7 | $ArH_2$ | 3.2 | 1.9 |
| Air | 5.3 | $ArH_2$ | 1.6 | 2.2 |
| average for air | 6 | | 2.4 | |
| $ArH_2$ | 1.3 | | | 2.5 |
| $ArH_2$ | 1 | Air | 1.2 | 2.7 |
| $ArH_2$ | 0.85 | Air | 1.3 | 3.3 |
| average for $ArH_2$ | 1 | | 1.25 | |

Table 2. Normalized minimum creep rates and times to rupture at 1150°C- $\sigma_0$ MPa

This table points out the fact that the average minimum creep rate of tests begun in dry air is roughly six times the one of tests started in hydrogenated argon. It appears also that changing atmosphere from air to hydrogenated argon leads in the two cases to a decrease of the minimum creep rate. The consequence is an increase in the life time to rupture. Lastly, the opposite switch, from hydrogenated argon to dry air, has almost no effect on minimum creep rate and therefore times to rupture are similar for all the tests begun under hydrogenated argon.

In order to correlate these environmental effects on creep curves with microstructure evolution, S.E.M. observations of the oxide scale and the resulting affected zone were performed. The major part of the oxide scale spalled during cooling to room temperature. The remaining pieces of oxide layer were often visible but not in contact with the substrate.

The micrographs in Fig. 3 show that the oxide scale after creep under synthetic air adopts a wrinkled morphology. The direction of the applied stress, which was also the direction of grinding, is a preferred axis for wrinkling. When the oxide layer is strongly convoluted (Fig. 3a), alumina is quite the only oxide formed whereas in regions less undulated, Ti and Cr oxides are also observed. Ni-based single crystal superalloy oxidation leads to the appearance of a depleted zone in $\gamma'$ precipitates because of the consumption of aluminum to form alumina. In the case of the sample crept under air, the average thickness of this depleted zone is about 15 µm. The micrographs section of the Fig. 4 show however that in region where oxidation is highly developed, the affected zone may reach a thickness of 60 µm.

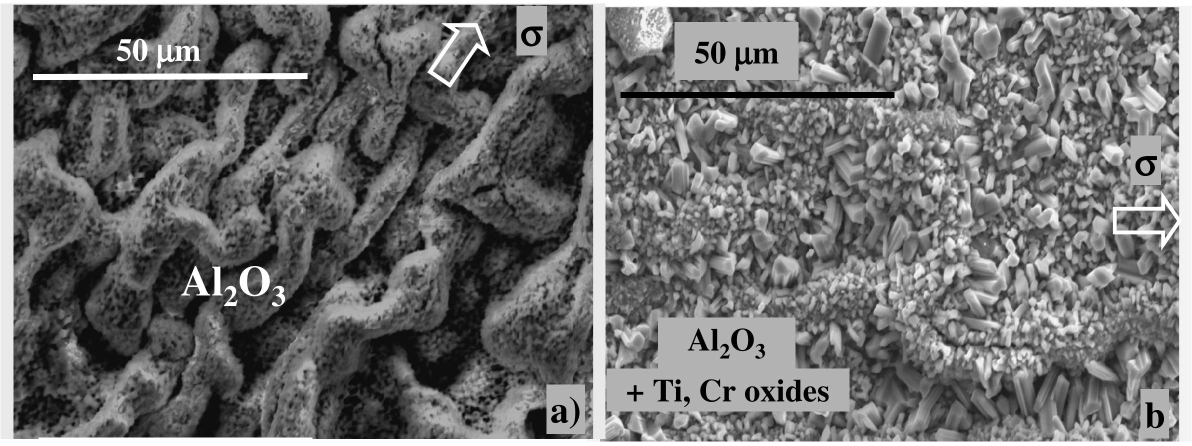

Fig. 3. Oxide layer at the surface of the specimen crept at 1150°C- $\sigma_0$ MPa under synthetic dry air, a) area with other oxides than $Al_2O_3$ , b) area with a convoluted alumina layer

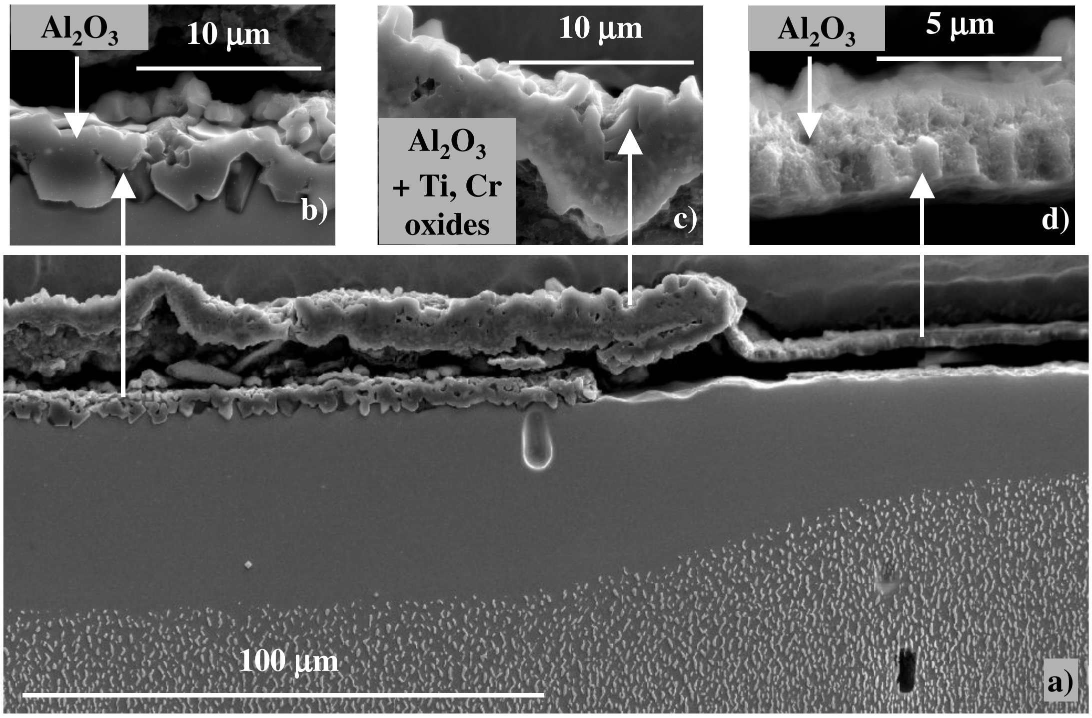

Fig. 4. S.E.M. micrographs of the sample crept at 1150°C- $\sigma_0$ MPa under synthetic air a) general view of the depleted zone and the oxide layer, b), c) and d) enlarged views of the oxide layer at different location

The morphology and the composition of the oxide scale formed during the creep test performed under hydrogenated argon are very different (Fig. 5). The oxide scale is homogenous, slightly convoluted and composed quite only of alumina. Its thickness is about 3 µm and the resulting depleted zone, very homogenous too, is 10 µm thick.

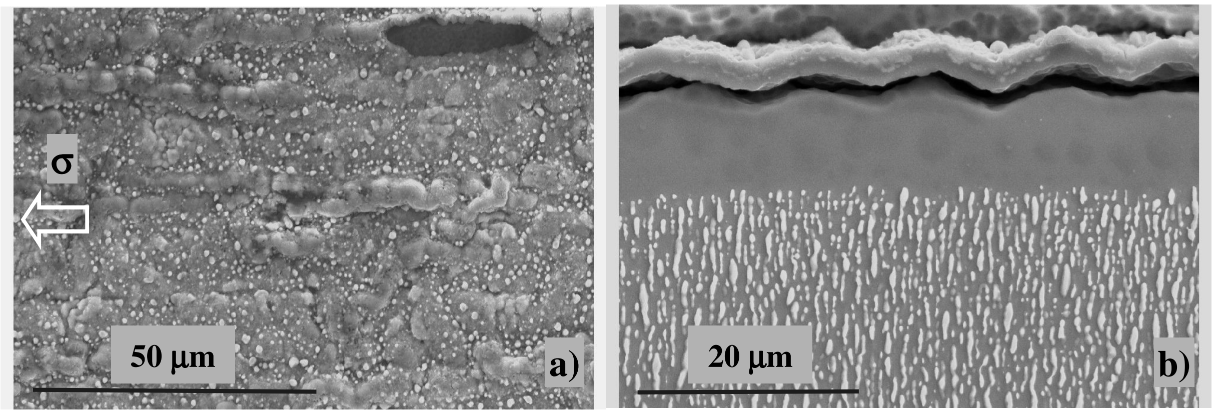

Fig. 5. S.E.M. micrographs of the specimen crept under hydrogenated argon, a) view of the surface of the oxide scale, b) cross section view of the depleted zone and the oxide layer

As one can see on figure 6a, the oxide layer developed after creep test with a switch from hydrogenated argon to synthetic air is similar to the one formed under hydrogenated argon only (Fig. 5b). The depleted zone is again homogeneous and is 15 µm thick. Lastly, the micrograph on Fig. 6b shows that the feature of the oxide scale after creep with a switch from synthetic air to hydrogenated argon is intermediate compared to a single environment. The duplex oxide layer is composed of an inner alumina scale and an outer scale rich in Al, Ni, Cr and Ti. The thickness of the depleted zone varies from 15 to 25 µm.

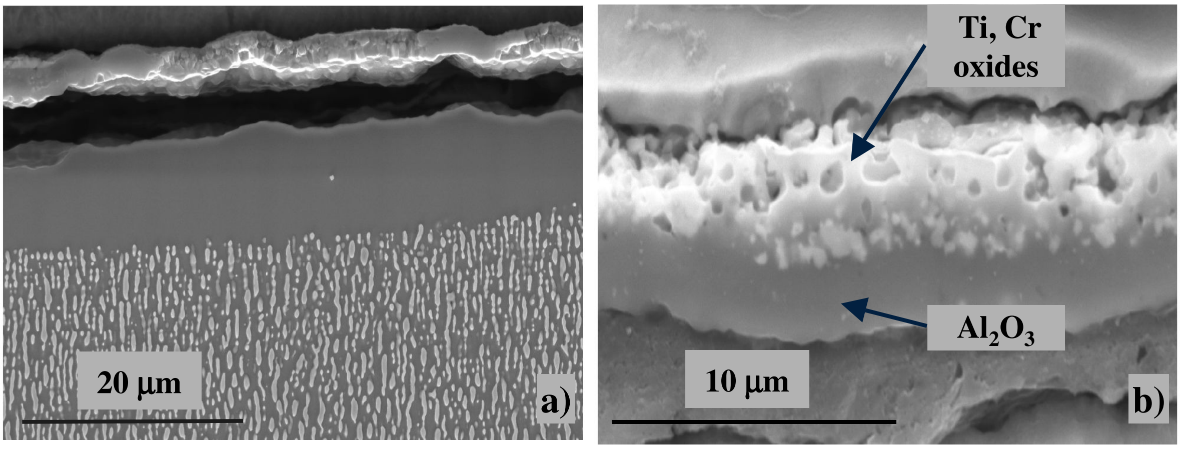

Fig. 6. S.E.M. micrographs, a) depleted zone of the specimen crept first under synthetic air and then under hydrogenated argon, b) oxide layer of the specimen crept first under hydrogenated argon and then under synthetic air

**Thermogravimetric Test.** The mass gain curves obtained from thermogravimetric tests are presented in Fig. 7a. The parabolic rate constants calculated from these curves have been plotted in the Arrhenius diagram of Fig. 7b with an estimation of these constants during the transient stage.

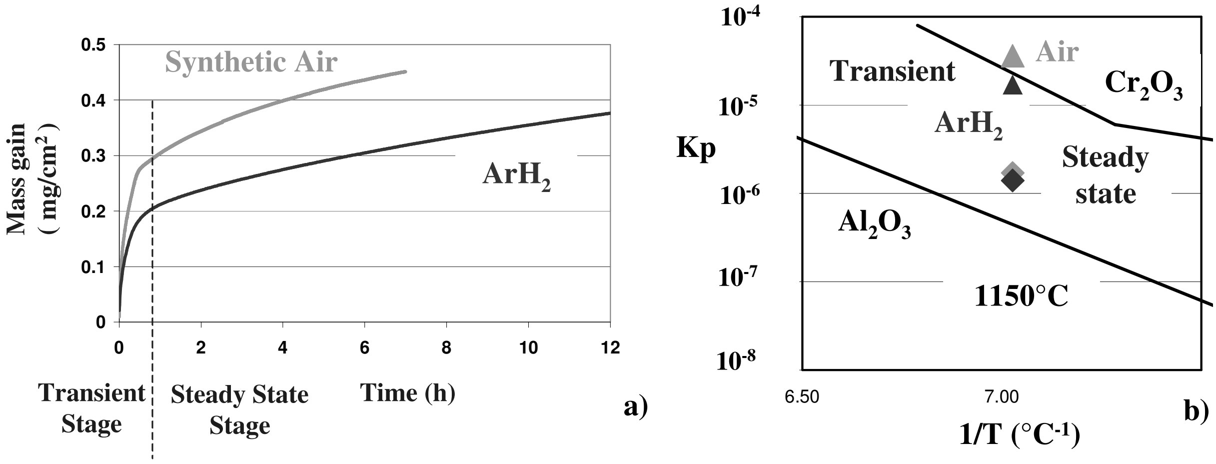

Fig. 7. a) mass gain curves resulting from the thermogravimetric tests, b) calculated kp in an Arrhenius diagram

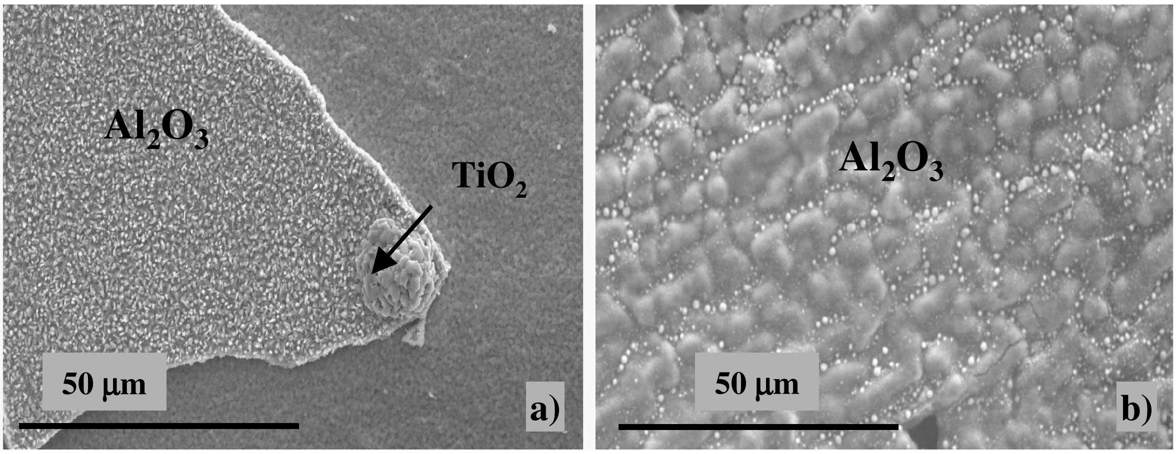

Fig. 8. S.E.M. micrographs of the surface of the oxide layer after thermogravimetric test, a) under synthetic dry air, b) under hydrogenated argon

Parabolic rate constants are the same for the two atmospheres whereas the oxide layer grows faster during the transient stage under synthetic air than under hydrogenated argon. The micrograph on Fig. 8a reveals that the morphology of the oxide layers formed under synthetic air after creep and thermogravimetric tests are very different for the same duration of test. For the thermogravimetric tests, the alumina layer is smooth and of uniform thickness with little other oxide grains at its surface and locally piles of oxides rich in Ti or Ni. Under hydrogenated argon, the same slightly convoluted scale is observed after thermogravimetric test and creep test (Fig. 8b).

## Discussion

**Differences in creep results under synthetic air and hydrogenated argon.** Micrographs of Fig. 3, 4 and 5 demonstrate that oxidation during creep test is radically different under synthetic air and hydrogenated argon. The affected zone by oxidation may be considered as non load bearing since the strengthening γ' precipitates have disappeared. The thicker depleted zone developed under synthetic air leads to an increase of the minimum creep rate. Using an appropriate Norton flow law, we estimated however that the ratio of 6 between the minimum creep rates measured under air and hydrogenated argon is consistent if the difference between the depletion zone thickness is about 75 µm. The average variation measured is only of 5 µm and the thicker zones observed locally under air are not spread enough to change drastically average creep rate. Therefore, the deleterious effect of the depleted zone cannot explain creep results. Another argument against this hypothesis is brought by the decrease of the minimum creep rate after switching from air to hydrogenated argon. Whatever the atmosphere, the oxygen partial pressure is high enough so that alpha alumina is stable once formed. The development of the depleted zone can be then slowed down by a change of atmosphere but its thickness cannot decrease. The evolution of the depleted zone cannot thus explain a decrease of the minimum creep rate after switching the atmosphere. Another way to explain the deterioration of the mechanical properties by oxidation is the injection of point defects. Studies [7,8,9] have shown that formation of an oxide scale by outward cationic diffusion towards the external surface of the substrate might induce vacancy injection into the alloy and so accelerate diffusion processes. The appearance of cavities below the surface of the specimens confirms the possibility of a such phenomenon. In single crystal superalloy, diffusion is involved in many creep mechanisms : dislocation climb to bypass γ' particles, morphological changes of γ' precipitates by migrations of γ / γ' and shearing of the precipitates [10]. Oxidation may then speed up microstructure evolution and dislocation motion which lead to higher creep rates and shorter times to rupture.

**Formation and growth of oxide scales.** The comparison between thermogravimetric tests and creep tests indicates that the applied stress influences the morphology and the composition of the oxide scale formed under synthetic air. This intensive oxidation during creep test performed under synthetic air explains the variation of creep rate regards to the atmosphere.

Wrinkling is generally associated with relaxation of the compressive stress which appears because of lateral growth of the oxide scale [11,12]. The creep deformation of the specimen under air probably increases this compressive stress. It leads to enhance the convoluted shape of the oxide layer in some areas and to the appearance of fissures in others areas which has for effect to allow the formation of transient oxides during all test duration. The oxide layer under hydrogenated argon stays slightly convoluted even when a stress is applied. The growth of transient oxides under air may lead to modify the stress state in the oxide layer and intensify undulation. Another explanation of the different stress states may be due to a better adhesion of the oxide scale formed under hydrogenated argon than under air. Looking at the surface of the metal where the oxide scale have spalled, we can indeed notice that under air there are areas with no imprints left by the alumina grains which indicates that the oxide scale was not adherent.

## Conclusion

The tests performed under controlled atmosphere have shown the drastic oxidation effect on creep properties of MC2 at 1150°C. This effect is not due to a poor oxidation resistance of the alloy but to the applied stress which modifies the composition and the morphology of the oxide scale under synthetic air. The decrease of the minimum creep rate after switching the atmosphere from synthetic air to hydrogenated argon proves that the consideration of a non load bearing affected zone cannot explain oxidation effect. Another explanation would be the injection of vacancies in the substrate alloy which may activate mechanisms of creep depending on diffusion. These results confirm the necessity of tests with thermomechanical loading more complex than usual and closer to the situation of blades in service since spalling due to thermal cycling may highly increase oxidation effect.

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