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MICROSTRUCTURAL EVOLUTION OF RENÉ N4 DURING HIGH TEMPERATURE CREEP AND AGING

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Abstract

The main scope of this work is to describe the microstructure evolution of single-crystal (SX) superalloy René N4 during creep and static aging at high temperatures, in function of time, stress and temperature. During creep at high temperatures, SX microstructure evolves from a dense and ordered distribution of cuboidal γ' particles to a configuration characterized by alternate rafts of γ' phase and γ matrix, through a process known as rafting. The microstructural evolution of superalloys is very important to derive models able to predict service conditions of a component through microstructural analysis. In this work two microstructural parameters were identified and analyzed for René N4: matrix channels width w along the [001] lattice direction and periodicity width λ, given by the sum of w and the width of the γ' precipitates along [001]. Both parameters were measured on some creep-damaged and some statically aged specimens, as well as on the virgin material to analyze their trends in function of time, temperature and stress. In particular, the parameter $\Delta\lambda$ looks independent of both the stress level and the microstructural morphology and could be used in future works to develop microstructural evolution model of René N4 in function of service time and temperature.

Keywords: Single-crystal, Superalloy, Microstructure, René N4

Introduction

Ni-based superalloys are widely used for manufacturing turbine buckets for civil and military aircraft gas turbine engines and for stationary gas turbines, due to their good fatigue resistance, both high (HCF) and low (LCF) cycles, to oxidation and corrosion resistance and especially to their excellent creep properties. Single-crystal (SX) superalloys are a family of Ni-based alloys that have greater high temperature strength and creep resistance. René N4 is a first generation SX superalloy developed by General Electric and used in high pressure stages of the most recent and advanced industrial gas turbines.

The fundamental property these components are required to have is a high creep resistance, which is mainly given by the γ' phase precipitation. The chemical composition of the material used in this study is shown in Table 1, where weight percentage of all elements is reported.

Table 1 – Chemical composition of René N4 superalloy.

Element	Cr	Co	Al	Ti	W	Mo	Ta	C	B	Nb	Hf	Ni
wt%	9.79	7.39	4.18	3.48	5.91	1.48	4.73	0.06	0.004	0.49	0.13	Bal.

Analyzing the chemical composition of Table 1, it is interesting to observe the total high amount of Cr, Co, W, Ta, and, in lower quantity, Mo, which are used for solid solution strengthening. Also, the high amount of Cr gives a higher corrosion resistance compared to other SX superalloys, while the high amount of Co gives a good microstructural stability at high temperatures. It is interesting to note the total absence of Re, typical of first generation SX superalloys, the substantially equal fraction of Al and Ti (both γ' formers) and the very low presence of C and B (carbides and borides formers).
The virgin microstructure of René N4 after the primary heat treatments is characterized by a dense and ordered distribution of cuboidal γ' precipitates, separated by narrow channels of γ matrix, typical configuration of all single-crystal superalloys [1].
Figure 1 shows a SEM image of the virgin microstructure of René N4 at 20000X magnifications.

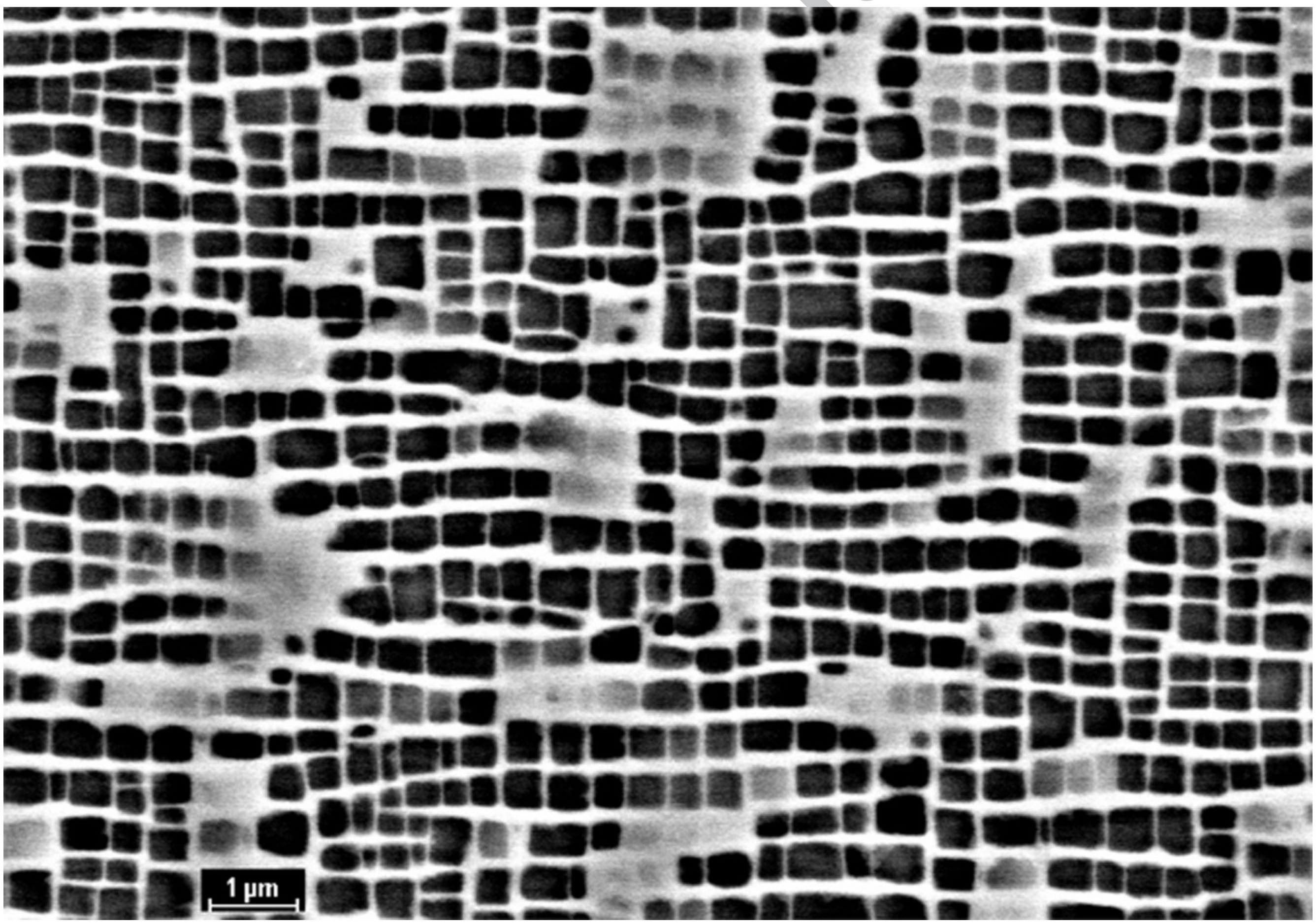

Figure 1 – Virgin microstructure of René N4 at dendrite core.

A long service exposure at high service temperatures and stresses brings to an alteration in shape and dimension of γ' precipitates [2,3]. Microstructural damage heavily influences components life duration and, once that these alterations reach a too high level, mechanical properties are impaired [4,5].

Once a model is able to describe the microstructural evolution, it is possible to determine the service conditions of a component through some specific microstructural analysis. Since the service conditions of a part are very difficult to measure and verify otherwise, the microstructural model plays a key role for the Residual Life Assessment (RLA) of a component.
Unfortunately, microstructure evolution in single-crystal superalloys does not follow the well-known LSW (Lifshitz, Slyozov, Wagner) theory, as the more conventional polycrystalline superalloys do [6-11], and strongly depends not only on service time and temperature, but also on the applied stress.
During high temperature creep, in SX superalloys, γ' phase coarsening first occurs followed by a directional coalescence of the precipitates that finally leads to a rafted microstructure (process known as *rafting*) [12-15].
Figure 2 shows an example of rafted microstructure of René N4 after a creep test.

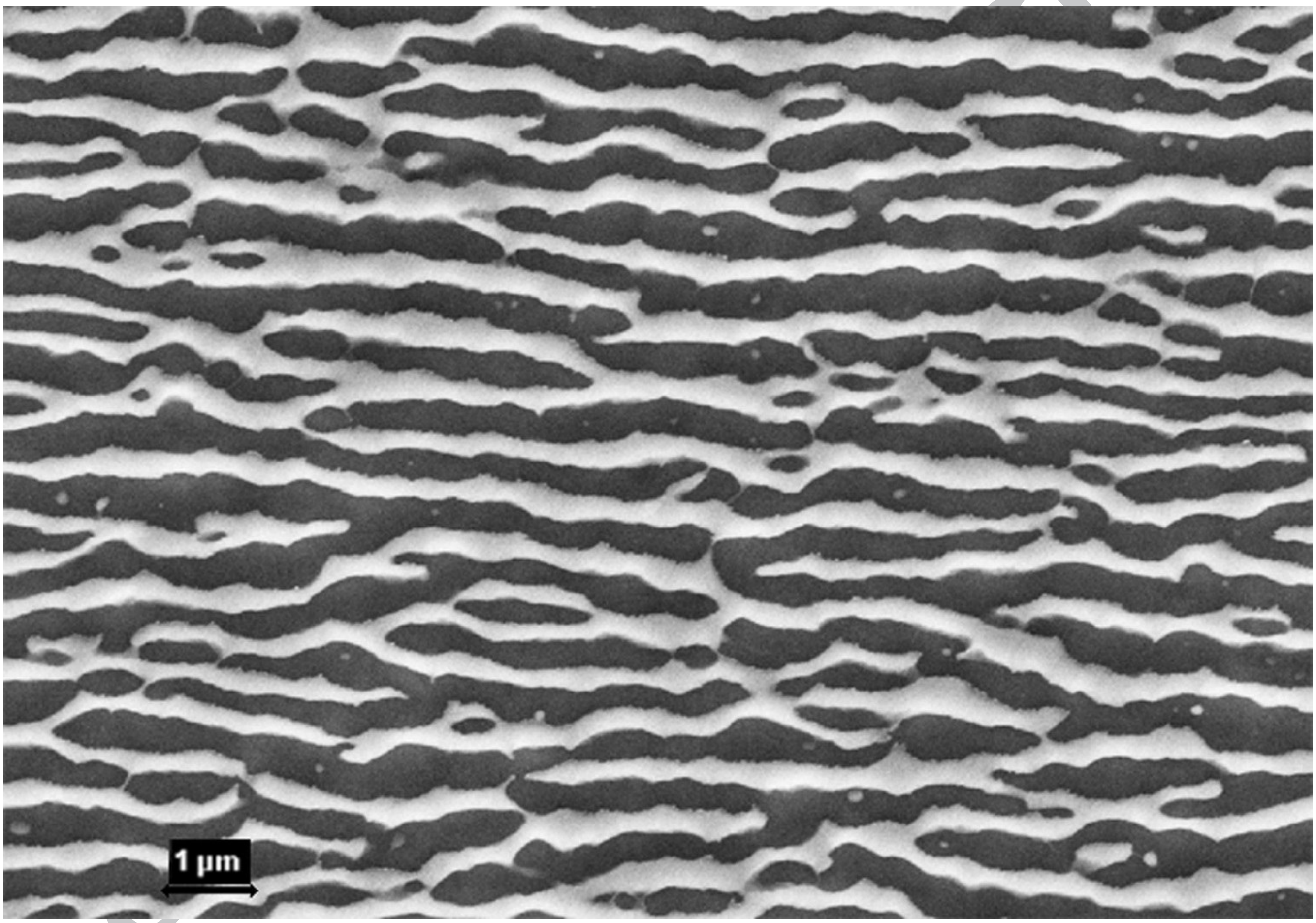

Figure 2 – Microstructure of René N4 characterized by rafting after creep at high temperatures.

It is therefore necessary to identify new microstructural parameters, to assess the operating conditions even for single-crystal components.Some works, that study the microstructure evolution of single-crystal superalloys, are present in the literature. The most interesting ones, briefly described below, all refer to CMSX-4 microstructure evolution during uniaxial creep at high temperatures (over 850°C), with the stress applied along [001] lattice direction [16-20]. The microstructural parameters mostly analyzed are shown in Figure 3. In particular:

- the γ' width is the dimension of the precipitates in the direction parallel to [001]
- the γ' length is the dimension of the precipitates in the direction perpendicular to [001]
- the γ width (also known as w) is the dimension of the matrix channels in the direction parallel to [001]

- the periodicity width λ is defined as the sum of the γ channels width and the γ' precipitates width.

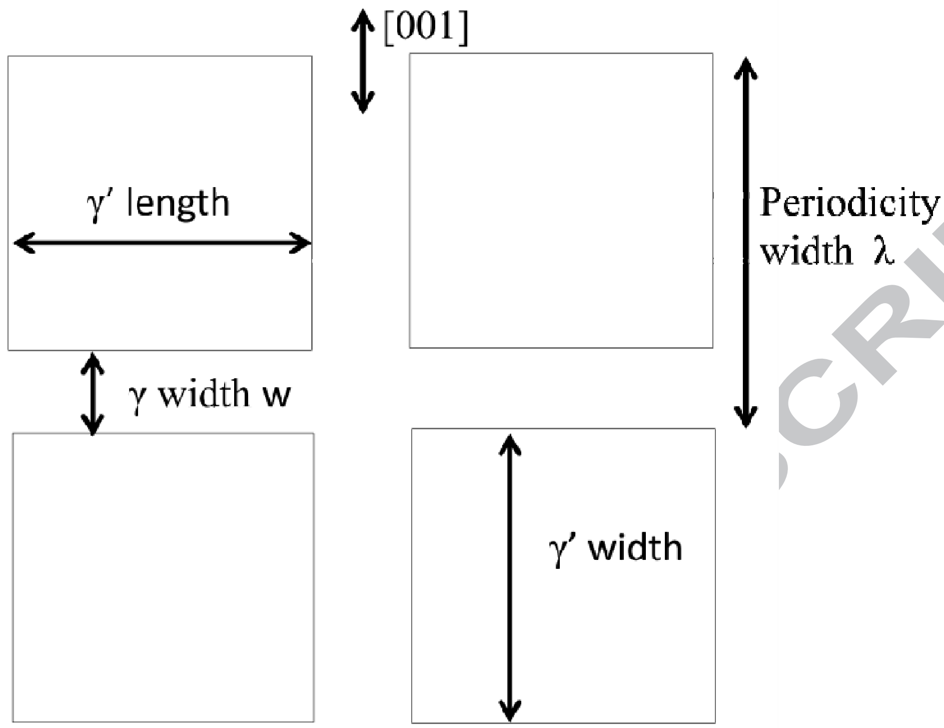

Figure 3 - Microstructural parameters evaluated in [5].

Lapin et al. [16] have performed some creep tests on CMSX-4 superalloy, at a constant temperature of 950° C with different stress and time levels. In their work they have analyzed the average values of the width and the length of the γ' precipitates and the γ channels width. Their results are that the γ' width decreases with the increase of the stress and increases with time, while the γ' length and the γ width increase with both stress and time. Even Matan et al. [17] have studied the trend of γ' length, γ' width and γ width in CMSX-4 superalloy during creep at 950°C. They have also introduced the rafting parameter R, defined as the ratio of γ' length to γ' width, as a good parameter to describe the microstructural evolution. Epishin et al. propose a linear model of evolution of the γ width for CMSX-4. According to their model, the γ channels width depends on both temperature and stress applied and increases linearly as a function of time, with a double slope [18]. Another microstructural model present in literature is the one proposed by Kamaraj et al. [19], according to which the variation of the γ width from the virgin condition is linearly dependent on the square root of time. Furthermore, Fedelich et al. [20] propose two different parameters to model the microstructural evolution of CMSX-4 superalloy during high temperature creep: the periodicity width λ and the γ channels width w. The periodicity width is defined as the sum of the γ channels width and the length of the γ' precipitates in the load direction [001] (Figure 3). In particular they find that the evolution law of the periodicity width λ during creep only depends on time and temperature, and not on stress, while the evolution law of w seems to depend even on the stress applied. Although in literature there are examples of microstructural models developed for CMSX-4 superalloy produced by Cannon-Muskegon, currently there are no studies on René N4. The main purpose of this work is then to describe the microstructure evolution of René N4 during creep and static aging at high temperatures, compare the evolution of the microstructural parameters with the results on CMSX-4, and identify suitable microstructure parameters for this material.

1 – Experimental Procedure

Different types of tests were performed on René N4 superalloy, in the as-received condition (virgin material). Some disks and some cylindrical specimens were cut from bars of the same base material, whose chemical composition is shown in Table 1. Creep specimens were machined according to ASTM E139 from the cylindrical bars, with gauge length of 35 mm and diameter of 6.25 mm. The disks were used to analyze the virgin microstructure and perform some static aging.
Creep tests were performed at temperatures between 870°C and 925°C using different values of stress and time. In creep tests, the load was always applied in [001] direction. The strain levels obtained were different from test to test, with a range between 0.2% and 6.5%.
In addition to creep tests, four static aging were carried out at 925°C for 1000, 2000, 5000 and 12000 hours, and one static aging was performed at 900°C for 10000 hours.
For all tests performed (both static and creep) and for the virgin material, the microstructure analysis was performed using SEM. The metallographic analyses were all performed at the end of the tests. Both creep and static specimens were cut along the diameter in the [001] direction and the SEM analysis were performed on the cutting surface, within the gauge length for the creep specimens. Measurements of λ and w were performed on 20 different fields in the dendrite core area at 20000X magnifications. The analysis of the virgin material also included the measurement of the average equivalent diameter and the total volume fraction V_f of γ' precipitates. The equivalent diameter of a precipitate is defined as the diameter of a circle with the same area of the particle. Table 2 summarizes all tests performed (creep and static aging), with the corresponding values of stress, temperature and time. All the tests were time based to create a microstructural model.

Table 2 – Description of all tests performed.

N.° test	Temp (°C)	Stress (MPa)	Time (h)
S1	925	0	1000
S2	925	0	2000
S3	925	0	5000
S4	925	0	12000
S5	900	0	10000
1	925	207	500
2	925	185	900
3	925	207	900
4	925	230	900
5	900	160	1750
6	900	180	1750
6A	900	180	1750
6B	900	180	1750
7	900	200	1750
8	900	240	1100
9	900	240	1500
10	900	270	1000
11	870	200	1800
12	870	240	1800
13	870	290	1800

2 – Microstructural parameters trend

With reference to previous works done on CMSX-4 superalloy, the candidate parameters chosen to describe the microstructural evolution were the matrix channel width w, and the periodicity width λ. The average dimension of matrix channels width w in the virgin material is 0.198 μm with a standard deviation of 0.011 μm, the average dimension of the periodicity width λ is 0.612 μm with a standard deviation of 0.015 μm, while the average equivalent diameter of γ' precipitates is equal to 0.587 μm. The total volume fraction V_f of γ' precipitates in the virgin material is equal to 69.6%, which is perfectly aligned with the value of 70% typical for SX superalloys [1].

Referring to Table 2, the first exploratory tests to understand microstructural parameters trend were static aging S1 and S2 and the creep tests from 1 to 4. As shown in Table 2, these six tests are all performed at 925°C with different values of time and stress and their main purpose is to observe and verify the dependence of the two microstructural parameters on the applied stress. Figure 4 shows the results of these preliminary tests for λ parameter variation (Δλ) from the virgin condition.

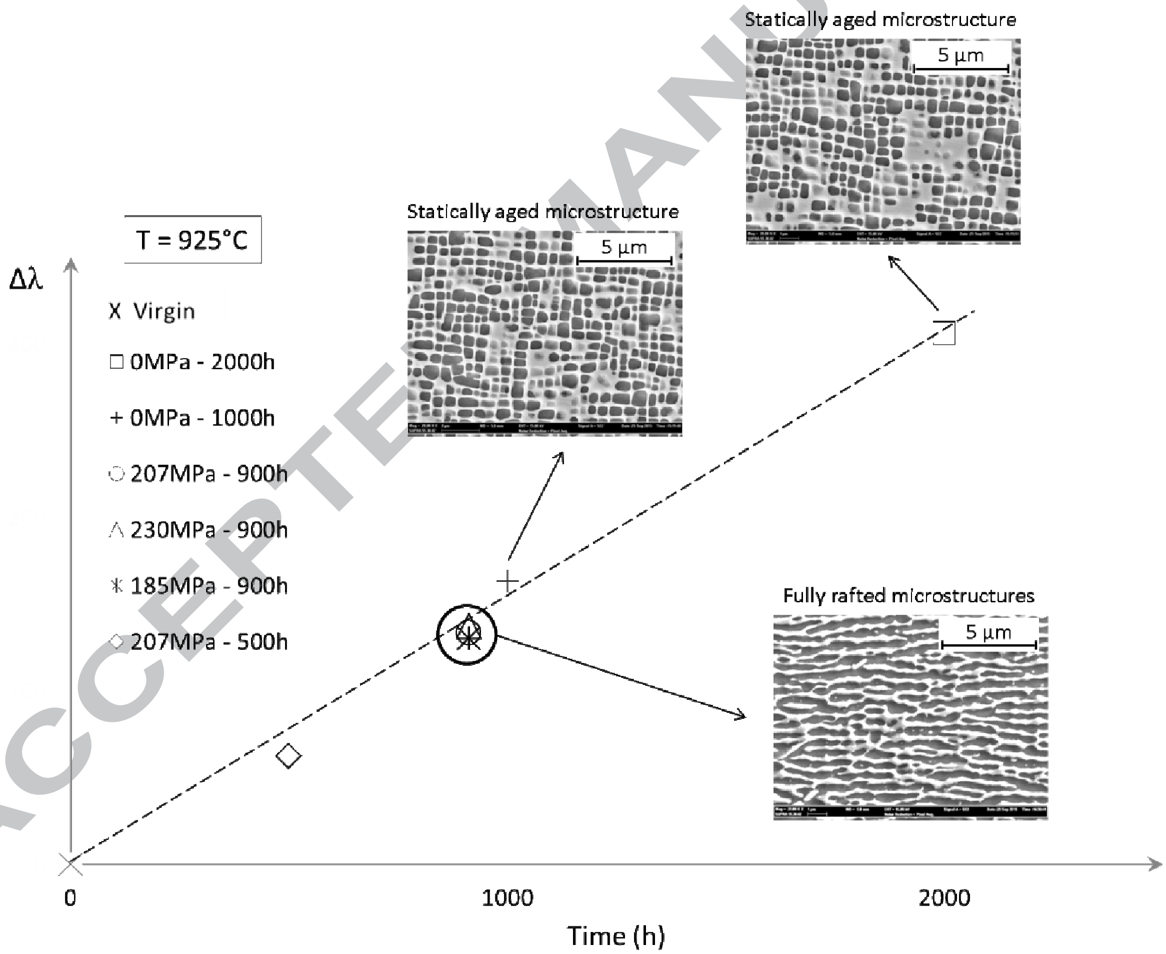

Figure 4 – Results of preliminary tests referred to Δλ with relative microstructure.

Looking at Figure 4, Δλ trend appears to be independent not only on the applied stress level, but also on the microstructural morphology: the parameter does not seem to be affected by the presence or absence of rafting.

The situation is rather different for w parameter and its variation (Δw) from the virgin condition. Figure 5 shows the results of these preliminary tests for Δw parameter.

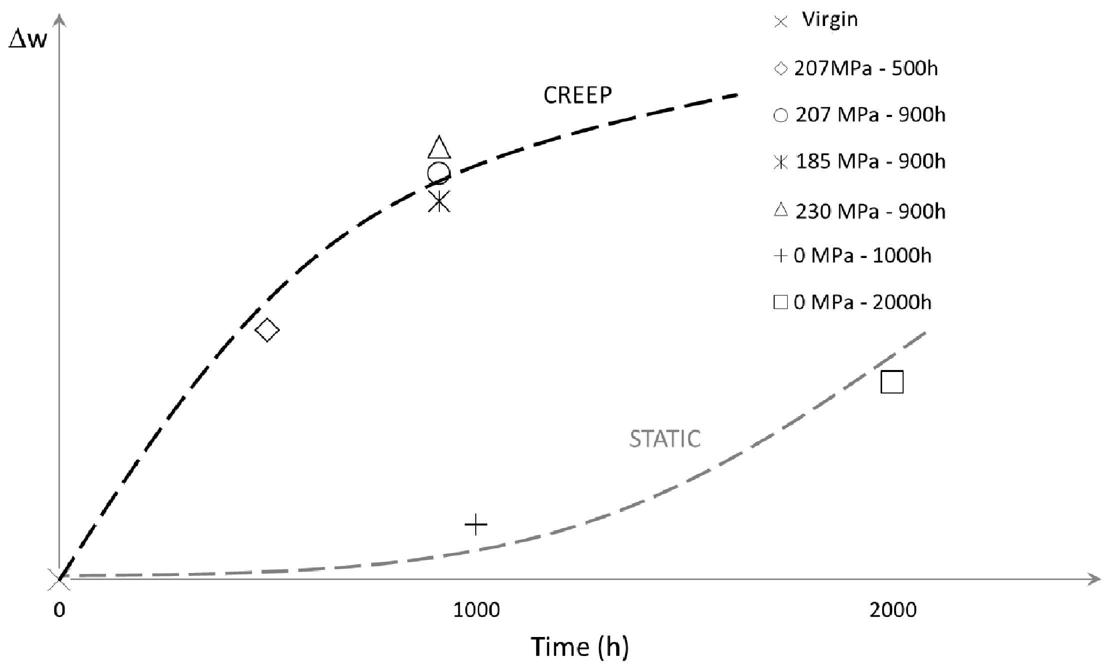

Figure 5 – Results of preliminary test referred to Δw.

Δw trend seems to be strongly affected by the application of stress and its variation in function of time is significantly different between static and creep tests. With these considerations, it is interesting to analyze both parameters trend in function of applied stress. Tests 2, 3 and 4 were used for this scope and were all performed at 925°C for 900 hours, with three different stress levels. Figure 6 shows parameters trend in function of stress for above mentioned tests. In addition to the average values of Δλ and Δw, for each test also the measure uncertainty is reported, which is constant in all the tests shown in Table 2 for both Δλ and Δw and corresponds to about ± 10% of the average values.

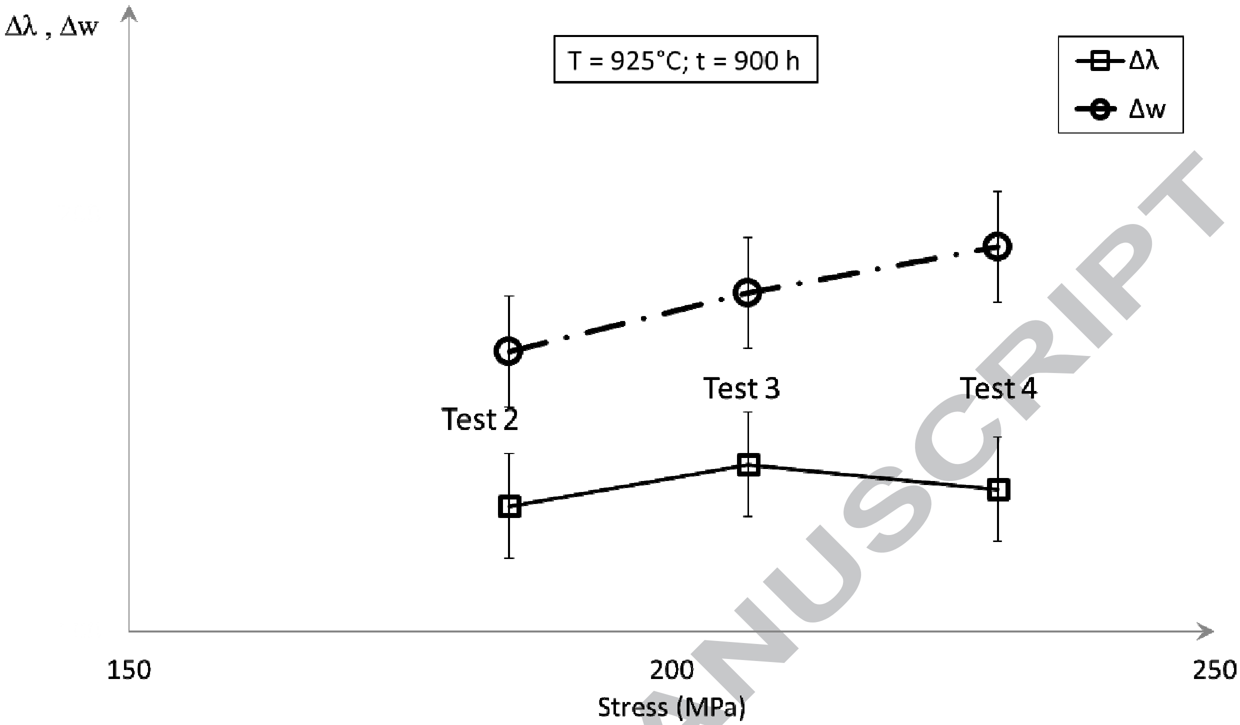

Figure 6 – Δλ and Δw trend referred to tests 2, 3 and 4.

The results shown in Figure 6 indicate that the Δλ parameter really seems to be independent of the applied stress; in fact the difference between the highest (test 4) and the lowest average value (test 2) is about 7% of test 2. Instead Δw shows an apparent increase proportional with stress but also in this case the difference between the highest and the lowest average test value (about 15% of the average value of test 2) is enclosed in the uncertainty range.

Also tests 5, 6 and 7 were performed at same time and temperature with three different stress levels. Figure 7. shows Δλ and Δw trend in function of stress for these additional three tests, both in terms of average values and uncertainty intervals.

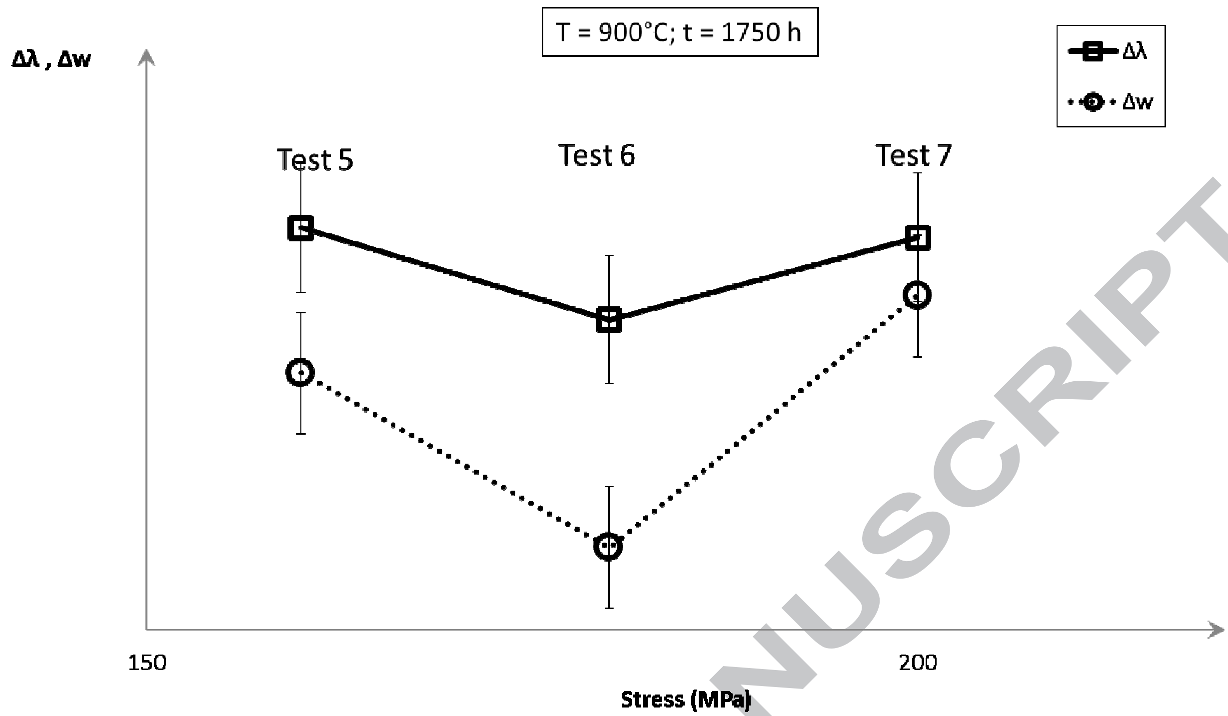

Figure 7 – Δλ and Δw trend referred to tests 5, 6 and 7.

In this case, while Δλ independence on stress seems to be confirmed, Δw does not show any explicit dependence on stress. In particular, it is interesting to observe that Δw value in test 6 is significantly lower than test 5 even considering the measure uncertainty, although the stress level is higher for test 6. The difference between the minimum and the maximum average value of Δλ is about 12%, less than the measure uncertainty. These results obtained by the tests 5, 6 and 7 for Δw are in contrast with what seen in literature for CMSX-4 superalloy, while Δλ independence on stress seems to be confirmed.

Tests 11, 12 and 13 were all performed at 870°C and 1800 hours, also in this case with three different stress levels. The results of these three tests are very interesting, since the difference between the stress levels is much higher than for the previous groups of tests (see Figure 8).

Tests 11, 12 and 13 confirm Δλ trend: this parameter does not show any dependence on stress level even in this case, with a limited variation from one test to another. In this case the maximum variation between the average values is about 15%, indicating that even for these tests the difference between the results fall in the measure uncertainty. The condition is different for Δw as, if from one side there is a clear increase from test 11 to test 12, on the other side it is not possible to appreciate any significant change from tests 12 and 13.

Even the results of these three tests about Δw trend for René N4 superalloy are in contrast with what known from literature for other SX superalloys: Thus this parameter does not show any direct dependence on stress level, though its trend is undoubtedly influenced by the stress itself (Figure 5).

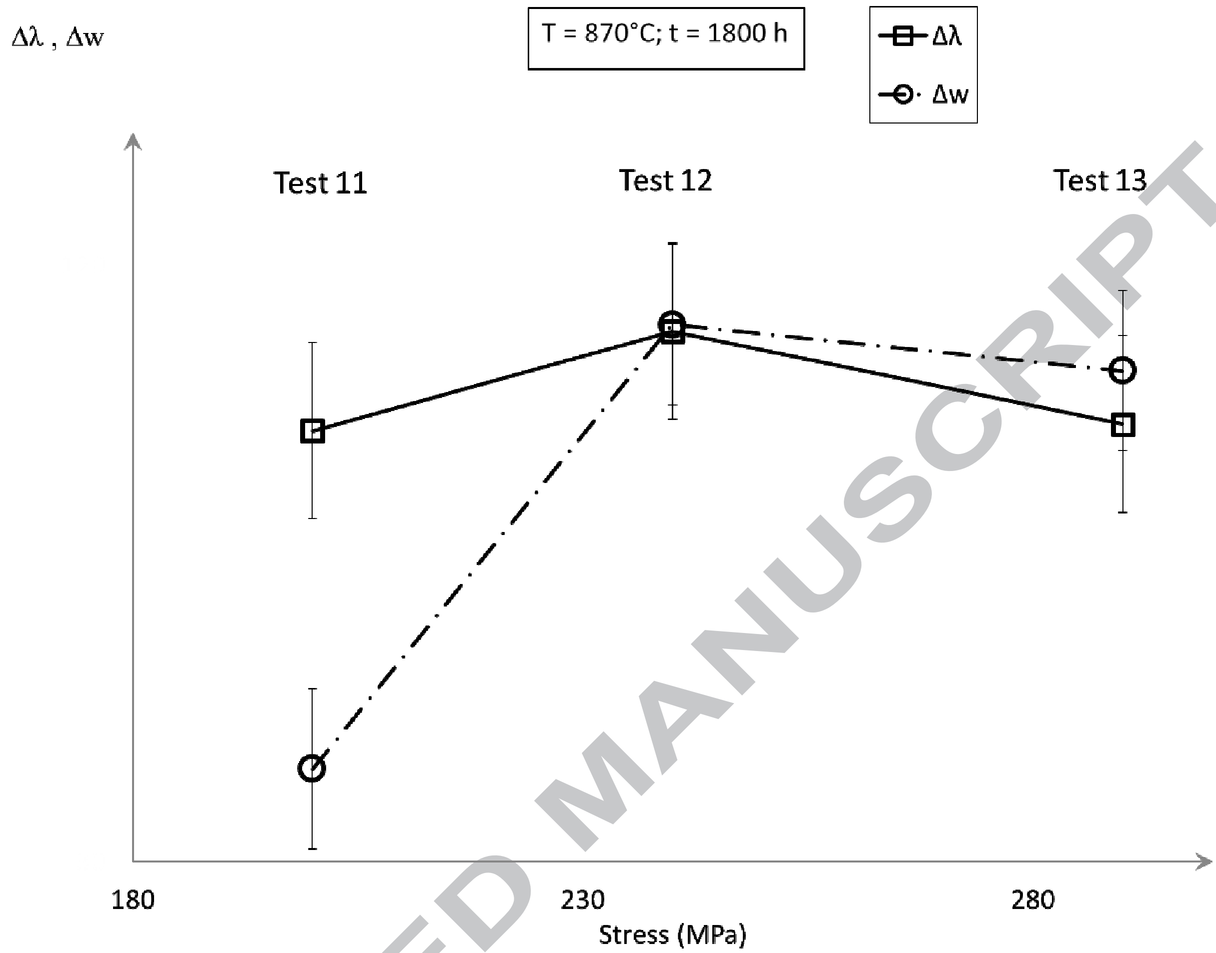

Figure 8 – Δλ and Δw trend referred to tests 11, 12 and 13.

Moreover Δλ and Δw parameters have been analyzed also in terms of repeatability of the measures: for this scope test 6 was repeated twice through tests 6A and 6B (same conditions of time, temperature and stress, as reported in Table 2). The Figure 9 shows Δλ and Δw results related to tests 6, 6A and 6B, with the average value obtained and the total variation from the average.

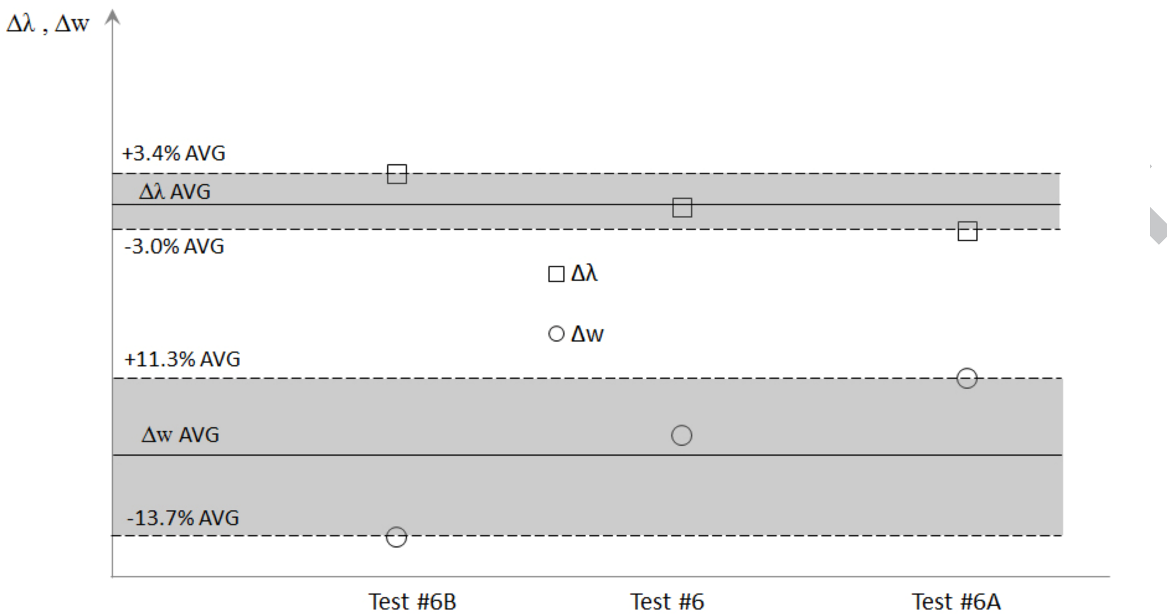

Figure 9 – Δλ and Δw repeatability results relative to tests 6, 6A and 6B (at 900°C, 180 MPa, 1750h).

While Δλ shows a good repeatability, Δw results are characterized by a larger scatter. In fact the maximum variation of the average Δλ values (about 6%) is much less smaller than the measure uncertainty (±10%). Instead the difference between Δw average values of test 6B and 6A results greater.

Some of the previous results on Δλ parameter are summarized in Figure 10 considering the overall trend at 900°C and 925°C obtained with all tests performed at these two temperatures. Δλ parameter, as already suggested by the previous test results (e.g. Figure 4), is not influenced by stress level in all tests performed at 900°C and 925°C. This characteristic, together with the good repeatability shown (Figure 9), suggests that Δλ could be a very suitable parameter to describe microstructural evolution of René N4 in function of service time and temperature.

On the contrary the situation appears very different for Δw parameter: although the analysis of preliminary tests clearly show that its trend is highly influenced by stress application during the subsequent tests the parameter does not show a clear and identifiable dependence on stress itself. Moreover, Δw measures show much worse repeatability compared to Δλ (see Figure 9). These results could confirm that Δw is not very suitable for the description of the microstructural evolution of René N4 superalloy during creep.

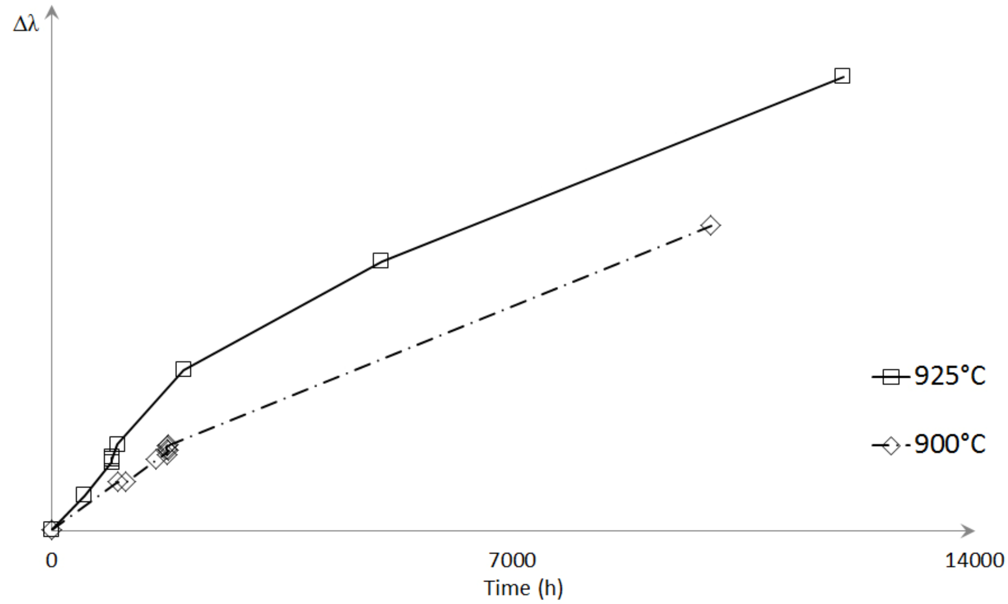

Figure 10 – Trend of Δλ parameter for all tests performed at 900 °C and 925°C.

Conclusions

This work analyzes the microstructure evolution of SX superalloy René N4 during creep and aging at high temperatures, in function of time, stress and temperature. During creep at high temperatures, SX microstructure changes from a dense and ordered distribution of cuboidal γ' particles to a configuration characterized by alternate rafts of γ' phase and γ matrix. In this work, two microstructural parameters were identified and analyzed, to describe the microstructural evolution of the material during high temperature exposure: the matrix channels width w along the [001] lattice direction and the periodicity width λ, given by the sum of w and the width of the γ' precipitates along [001]. Both parameters were measured on some creep-damaged and some statically damaged specimens. The analyses performed show that the parameter Δλ looks independent of both the stress level and the microstructural morphology (i.e. with or without rafting), while the parameter Δw does not show a clear dependence on the applied stress nor a good repeatability, in opposition to what seen in literature for CMSX-4 superalloy. These results suggest that Δw does not seem to be a good parameter to describe microstructural evolution of René N4 SX superalloy. Instead the independence on the stress level combined with a very good repeatability, suggests that Δλ could be the right parameter to describe microstructural evolution of René N4 in function of service time and temperature. The results obtained could be used in future work to develop a microstructural model for this superalloy, able to assess the service conditions of a component through the results of microstructural analysis.

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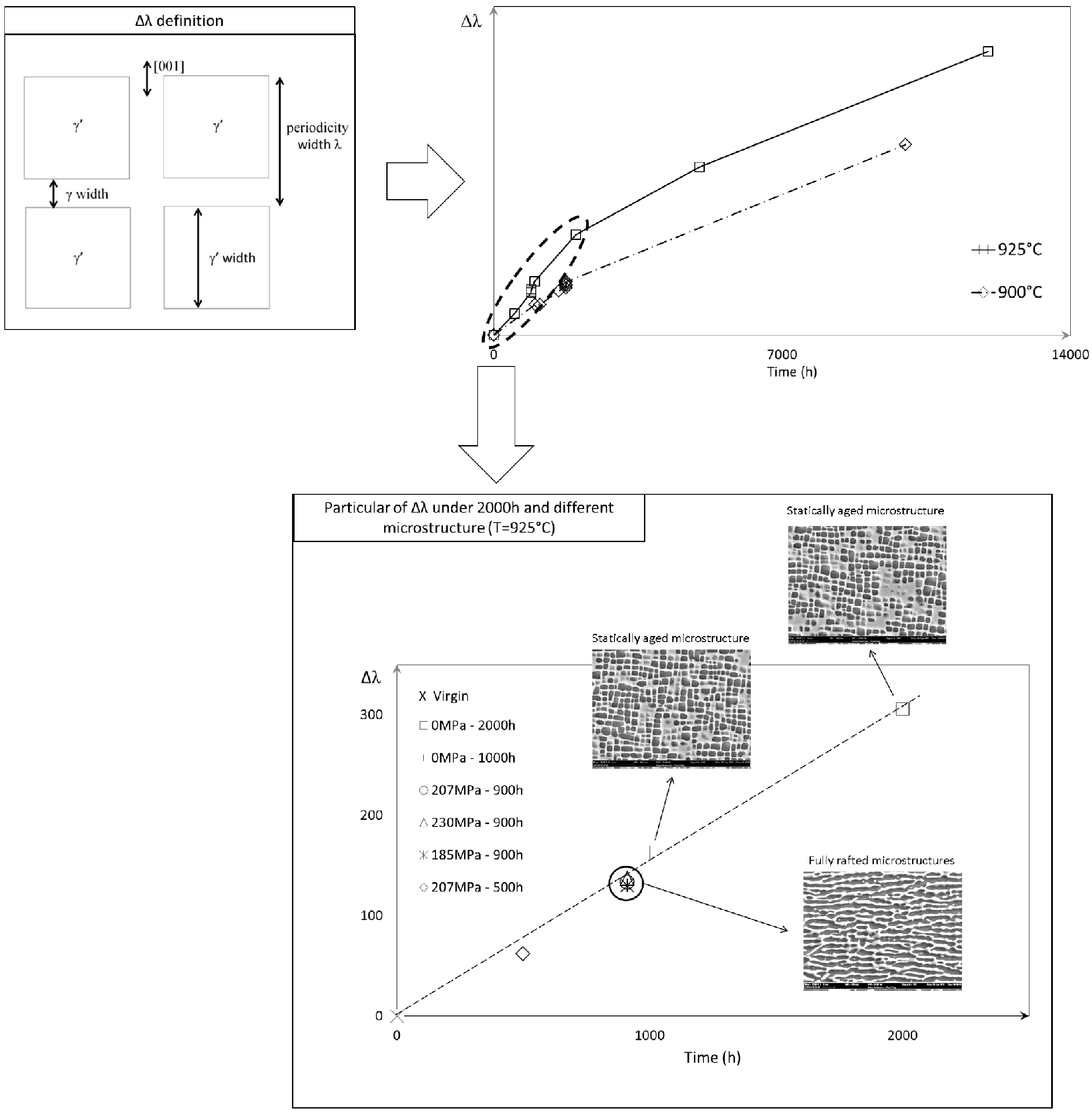
Highlights

- René N4 microstructural evolution is fundamental to derive models able to predict service conditions of components through microstructural analysis.
- Microstructural parameters w and λ and their variation from virgin condition were measured on some creep and statically damaged specimens.
- After all tests performed, $\Delta\lambda$ shows a good repeatability and clear independence on the stress applied.
- Differently from what stated in literature for other superalloys, Δw does not show a direct dependence on the stress applied in René N4.
- $\Delta\lambda$ could be the right parameter to describe microstructural evolution of René N4 in function of service time and temperature.


Δλ definition
[001]
γ'
γ'
γ'
γ'
periodicity width λ
γ width
γ' width
Δλ
925°C
900°C
0
7000
14000
Time (h)
Particular of Δλ under 2000h and different microstructure (T=925°C)
Statically aged microstructure
Statically aged microstructure
Fully rafted microstructures
Δλ
X Virgin
0MPa - 2000h
0MPa - 1000h
207MPa - 900h
230MPa - 900h
185MPa - 900h
207MPa - 500h
300
200
100
0
0
1000
2000
Time (h)