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HTR-M1 Deliverable D4

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CEA RCGMA R4 REPORT

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Abstract

The HTR-M1 project was launched within the 5th Framework Programme to consolidate and advance HTR materials technology in Europe. The HTR-M1 programme puts a special emphasis on the effects of creep and irradiation damage focusing specifically on the high temperature materials for the turbine and irradiation testing of graphites for the core.

This report gives a synthesis of experimental results obtained on turbine blade alloys that were selected as potential candidates for the turbine of future HTRs (High Temperature nuclear Reactors).

Two grades of directionally solidified superalloys (CM 247 LC DS and IN 792 DS) have been subjected to tensile and creep tests. Creep and tensile tests have shown that CM 247 LC DS possesses superior high temperature properties, when compared to IN 792, and therefore appears as the best candidate for HTR turbine blades.

A thermal ageing of nearly one year at 850°C was applied to IN 792. The effect on mechanical properties is a slight decrease of tensile strength and ductility. Creep strength is more severely altered, but creep ductility remains correct. These results would need to be confirmed by longer ageing durations to see if the embrittlement continues for long term service.

It was also shown that the thermal cycle associated with the carburisation performed at JRC (1000h/950°C) resulted in a slight decrease of tensile and creep strength for the two grades. This effect will have to be taken into account to compare the properties of carburised specimens to that of non-carburised ones.

This report mainly presents the results obtained at CEA-Grenoble, except for the chapter presenting the carburisation facility that was built at JRC. The results of mechanical tests after carburisation will be obtained by JRC after the end of HTR-M1 and will be reported in 2006 within RAPHAEL-IP project.

Key Words

HTR
Turbine
Superalloys
CM 247, IN 792

CONTENTS

I. Introduction. HTRM-1 General objectives.

The objective of this document is to present a synthesis of the work performed for turbine blade alloys in the frame of HTR-M1 project.

This report gives a synthesis of experimental results obtained on 2 blade alloys that were selected as potential candidates for the turbine of future HTRs (High Temperature nuclear Reactors). This reports presents the main results obtained at CEA-Grenoble, except for the chapter presenting the carburisation facility built at JRC. The results of mechanical tests after carburisation will be obtained by JRC after the end of HTR M1 and will be reported in 2006 within RAPHAEL-IP project.

Detailed information on the selection of the blade alloys, material procurement conditions and test matrix discussions can be found in previous reports available on SINTER :

- HTR-M1 Deliverable D1 : Report on selection of materials and testing requirements for the turbine, Document number HTRM1-02/09-D-1.1.7.
- HTR-M1 Deliverable D2 : Report on specimen fabrication, procurement and testing requirements for the turbine blade materials, Document number HTRM1-03/03-D-1.0.12.
- HTRM1 Deliverable D3 : Intermediate report on mechanical testing covering results from CEA and JRC, report CEA DTEN/DL/2005-033.

HTRM&M1 general objectives :

The HTR-M1 project was launched within the 5th Framework Programme to consolidate and advance HTR materials technology in Europe. Work was underway within the HTR-M project to highlight the requirements and establish a materials platform for the HTR technological developments focusing on the reactor pressure vessel, high temperature alloys for the internal structures and turbine, and graphite for the reactor core. The HTR-M1 programme extends the materials platform further with a special emphasis on the effects of creep and irradiation damage focusing specifically on intermediate duration creep testing of the high temperature materials for the turbine and irradiation testing of manufacturer recommended graphites for the selection of graphite for the core.

Turbine materials operate at temperatures of 850-900°C (HTR) and up to 1000^{o}C (VHTR). High strength alloys are being considered for the blades that are capable of withstanding such temperatures and conditions in the direct cycle environment for operating periods approaching 60,000h.
Creep strength and the corrosive effects of helium impurities on the properties and strength of the materials are important issues that must be evaluated. Manufacturing considerations are especially important and candidate alloys must have good properties and proven thermal stability. For the blades cast material is not sufficient, and so directionally solidified alloys have been considered.

The HTR-M investigations have highlighted the need for cooling and possibly coatings for both disc and blade alloys. Impurities in the helium at temperature can result in carburisation and/or de-carburisation of the super-alloys and lead to a lessening of material strength. From HTR-M both an Aluminium former and Chromium former were selected as potential options for the 850^{o}C gas temperature case.

The turbine test programme within HTR-M1 covers medium term creep tests under simulated HTR environments. The impact of corrosion is an important selection criterion since the coolant gas used in an HTR contains small levels of impurities (H_2, H_2O, CO, CO_2) at low partial pressures, and these can interact with the core graphite and metallic components and cause degradation of their properties.

Within HTRM&M1, specimens (or blanks) are machined from the as-received materials and then "*pre-treated*" (temperature 950°C) in Ar + 2% methane for carburizing and in Ar + 500 µbar H_2 + 50 µbar H_2O for de-carburizing, to achieve the different conditions to be tested :

- material in the condition "fully heat treated for service" (standard delivery for reference, "non-damaged condition") : denoted **"state I"**

- material entirely de-carburized: denoted **"state II"**

- material heavily carburized: denoted **"state III"**

- material with additional treatment (heat treated at 950°C, time same as pre-treatment, to separate thermal ageing effects from the effects of decarburization or carburization: denoted **"state I_{mod}"**)

CEA was responsible for heat treating the blanks for I_{mod} state and JRC was in charge of carburising and decarburising heat treatments.
The states II and III are tested by JRC and the states I and I_{mod} by CEA. All mechanical tests are performed under air atmosphere.

II. HTR-M1 main achievements.

The Work Package 1 of HTR-M1 project was focused on the high temperature mechanical properties of potential turbine blade alloys with respect to HTR specifications.

The main achievements of this project are the following :

- After a discussion between project partners, the selection and procurement (45k€) of the 2 blade alloys was achieved.

- A carburisation furnace was built at JRC. This facility allows to carburise or decarburise metallic specimens with 1000 hours thermal cycles under specific atmospheres (Figure 1).

- A “high temperature mechanical property” database was built by performing 24 tensile tests (from 20°C to 850°C), 40 000 hours of creep at 850°C (18 specimens) and 1 year of thermal ageing at 850°C.

Figure 1 : Carburisation furnace built at JRC.

III. Blade alloy selection.

Material selection :

The initial review made within HTR-M considered eight potential materials for the turbine disc, thirteen for the blade, including the Russian backup options. Creep resistance and high temperature strength were important requirements and expected to be the main selection criterion (HTR-M 02/7-D-2.1.39).

For the turbine blade failure modes such as creep, high cycle fatigue and low cycle fatigue have to be taken into account. The gas radial temperature variation often peaks typically in the middle third of the blade with steep temperature gradients across the blade section that varies according to the transient and steady state conditions. All regions of the blade profile therefore undergo complex stress-strain cycling with reversed plasticity in some areas.

From the blade materials investigated the best potential long term creep strength was provided by the Molybdenum based alloy “TZM”. This material has been investigated through a significant amount of testing within the German HTR programme but its high ductile-brittle transition temperature appears to make it a less practical option compared with Ni based alloys. The most promising Ni based super alloys in terms of creep strength are the single crystal alloys PWA 1483 (Cr-oxide former) and CMSX4 (an Al-oxide former). For the direction solidified blade suitable materials would be DS IN 792 LC (Cr-oxide former) and CM 247 LC DS (Al-oxide former). In the case of a conventionally cast blade (which could be used if the blade was cooled) IN 792 (Cr-oxide former) and MAR M247 (Al-oxide former) are also possible candidates.

Finally, the materials selected for mechanical testing within HTR-M1 are :

- IN 792 DS and CM 247 LC DS (Directionally Solidified)

IV. Blade material procurement and test matrix

The Directionally Solidified grades were supplied by Doncasters GmbH. Bars with a cylindrical geometry (ϕ 16 mm * 150 mm) were ordered in the “heat treated” condition. The total cost of these materials (45k€) was equally shared by CEA and JRC.
Finally, Doncasters delivered the following bars :

- 48 bars of IN 792 DS (heat RC415) received in march 2003 with a solution heat treatment (vacuum 1180°C/2h, cooling rate 30K/minute).
- 30 bars of CM 247 LC DS (heat X4012) received in may 2003 with a solution heat treatment and aging (vacuum 1220°C/2h + 1240°C/2h + 1246°C/2h cooling rate 80K/minute + vacuum 1080°C/4h/forced gas circulation + 900°C/8h/ forced gas circulation).

All test bars have been subjected to a "Dye Penetrant Inspection Examination" and a "X-Ray" examination to ensure that no cracks or open porosities were present.
The chemical composition of the test bars is given in Table 1.

	C	**Si**	**Mn**	**Cr**	**Mo**	**Ni**	**Ta**	**Ti**	**W**	**Co**	**Fe**	**Al**	**Hf**	**V**	**Zr**
CM 247	0.074	<0.03	<0.03	8.16	0.43	Bal.	3.21	0.68	9.55	9.364	0.03	5.63	1.42	<0.03	0.015
	Cu	**B**	**P**	**S**	**Pb**	**Ag**	**Bi**	**Te**	**Tl**	**Sb**	**Sn**	**Zn**	**Cd**		
	<0.03	0.0155	<0.005	6 ppm	<2ppm	<2ppm	<0.3ppm	<1ppm	<1ppm	<3ppm	<3ppm	<2ppm	<0.5ppm		

	C	**Si**	**Mn**	**Cr**	**Mo**	**Ni**	**Nb**	**Ti**	**Ta**	**Al**	**Fe**	**Co**	**Cu**	**Hf**	**W**
IN 792	0.079	<0.05	<0.05	12.63	1.82	Bal.	<0.1	3.91	4.18	3.52	0.02	9.08	0.03	1.34	4.25
	B	**Zr**	**P**	**S**	**Ag**	**As**	**Bi**	**Mg**	**N**	**O**	**Pb**	**Se**	**Te**	**Tl**	
	0.017	0.02	<0.002	<20ppm	<0.1ppm	<5ppm	<0.1ppm	<6ppm	<11ppm	<12ppm	<0.3ppm	<2ppm	<1ppm	<0.2ppm	

Table 1 **:** Chemical composition of IN 792 DS and CM 247 LC DS blade materials. (weight %).

Heat treatment problem with IN 792 DS :

We discovered in September 2003 that the IN792 material was not properly heat treated by the manufacturer (a second solutioning step (1080°C/4h) and the final aging (845°C/24h) were missing). When this problem was discovered, some IN 792 bars were already machined at CEA and some others had started a 10000 hours thermal aging at 850°C.

It was therefore decided to re-start the whole heat treatment on the aged bars and to simply add the missing HT steps to the other bars. Ageing was then restarted "from zero".

V. Experimental results on blade materials.

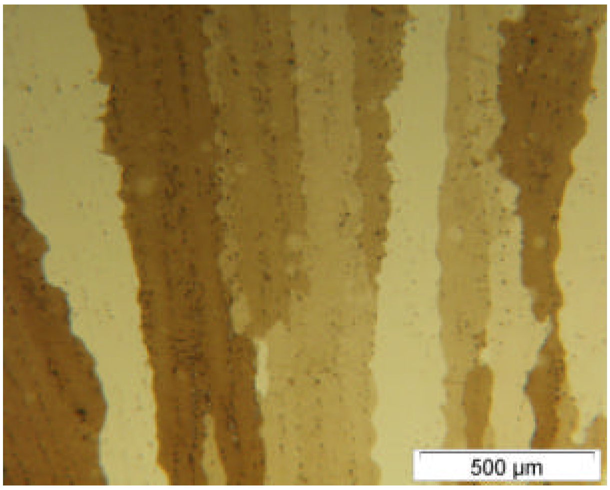
V.1 Metallography

The detailed results of the metallographic and TEM exams performed on IN 792 and CM 247 have been presented in the report "HTR-M deliverable 2.10 : Materials for turbine, final report, RT-DTEN-DL-2005-013" available on SINTER.

In this report, only the main conclusions on the microstructure of blade alloys are given.

The structure of CM 247 LC DS was observed by optical microscopy for sections parallel to the grains (001 direction) and perpendicular to the grains (Figure 2). A piece of the test bar labelled AB0746-5 (ϕ 16 mm, l = 150 mm) has been cut for metallographic examination.
The grain average width is 150 μm. For the structure parallel to the grains, we observe some misorientations and the presence of carbides and areas with primary eutectic γ' phase.
The structure with a normal orientation with respect to the grains shows residues of casting micro segregation and dendrite structure, and a few microporosities.

When compared to usual DS blade alloys, CM247 LC alloy seems to show the usual microstructural aspects.



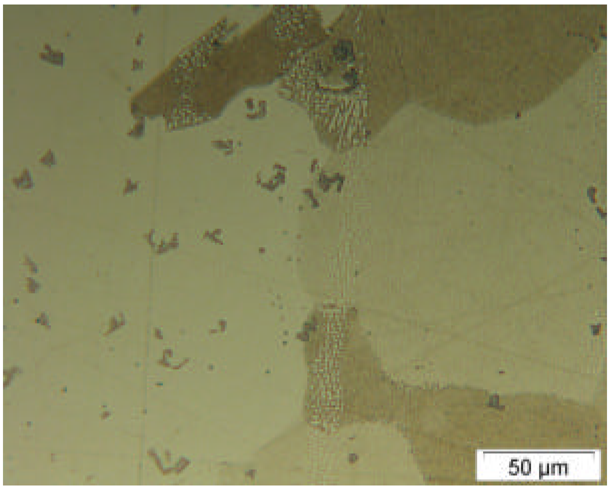


Figure 2 : Microstructure of CM 247, orientation parallel to the grains, at low and high magnifications.

The microstructure of IN 792 DS for sections parallel to the grains (001) and perpendicular to the grains do not exhibit microporosities, but the chemical segregation observed in the transverse direction seem to be more pronounced than for CM 247 (Figure 3 & Figure 4).

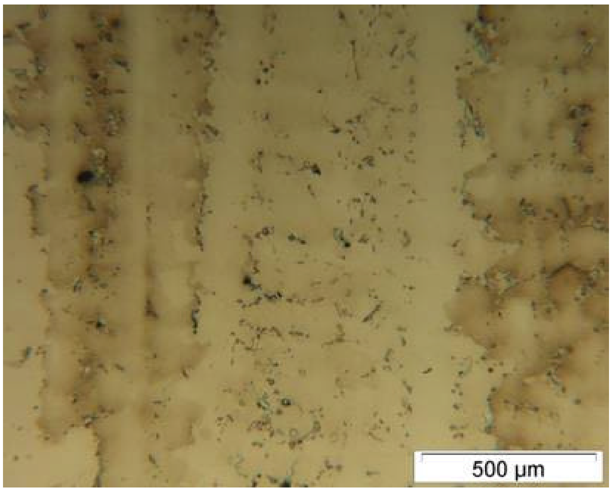


Figure 3 : Microstructure of IN 792, orientation parallel to the grains

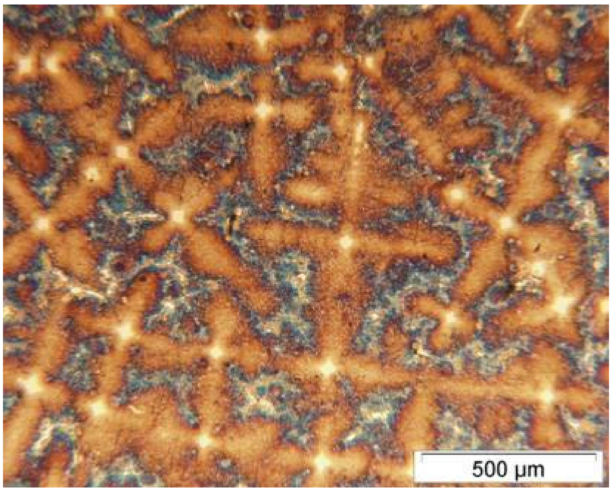


Figure 4 : Microstructure of IN 792, transverse orientation.

TEM observations of the microstructure have been performed for CM 247. We observe the gamma prime precipitates in the gamma matrix. The γ’ size is uniform except in areas close to the large primary γ’ where a very small γ’ distribution can be seen (Figure 5). Low angle grain boundaries are decorated with small precipitates that have been identified as phases enriched in W, Cr and Ni. These precipitates seem to have a chemical composition corresponding to the so-called ‘µ phase”. A few very large precipitates are also dispersed in the matrix. These precipitates were identified as carbides enriched in Hf and Ta.

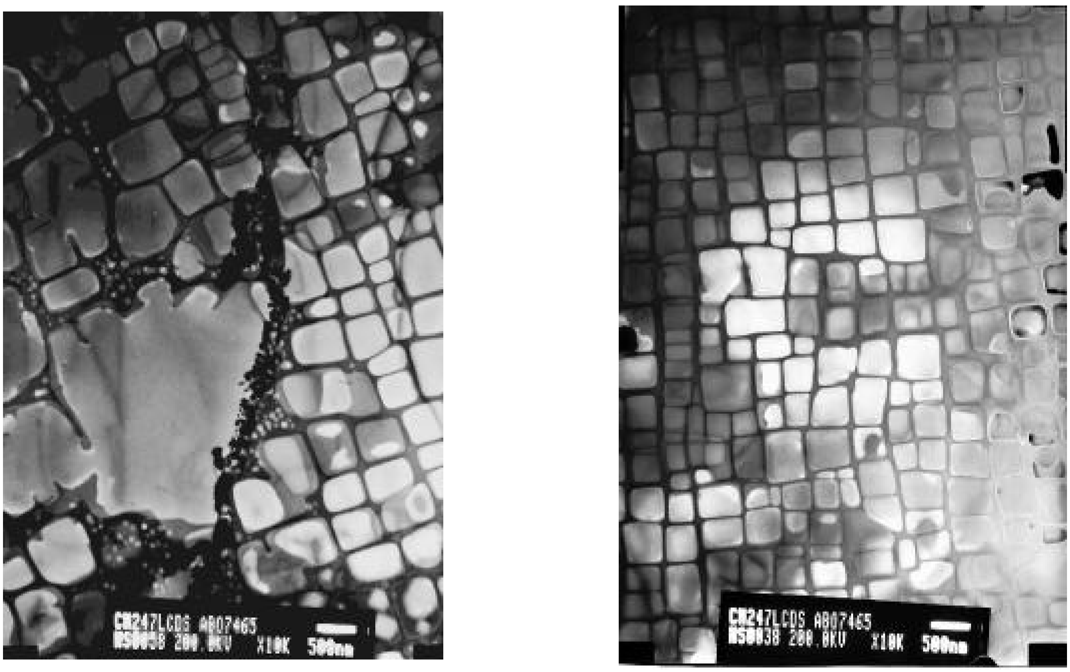


Figure 5 : General TEM view of CM 247 LC DS structure.

V.2 Tensile properties.

Tensile tests have been performed at 20, 750, 800 and 850°C with a strain rate of 5.10^{-4} s^{-1} for both CM 247 and IN 792.

A detailed analysis of the results is given in Appendix 1, together with the tensile specimen geometry.

Figure 6 presents the yield stress ($R_{0.2\%}$) of both grades at various temperatures. CM 247 shows a better yield strength than IN 792, especially at 850°C. The same trend is seen in Figure 7 for Ultimate Tensile Strength (UTS).

For rupture strain, the two grades show equivalent data except at room temperature where the IN 792 ductility is rather low (Figure 8).

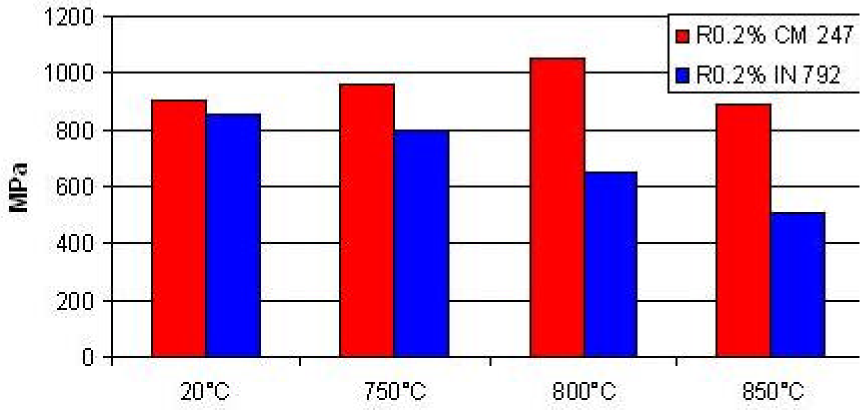


Figure 6 : Comparison of 0.2% Yield Stress for CM 247 and IN 792 from 20°C to 850°C.

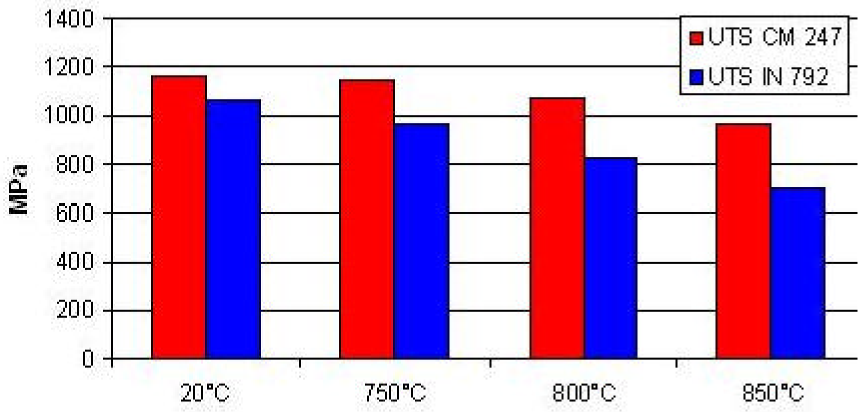


Figure 7 : Comparison of Ultimate Tensile Stress for CM 247 and IN 792 from 20°C to 850°C.

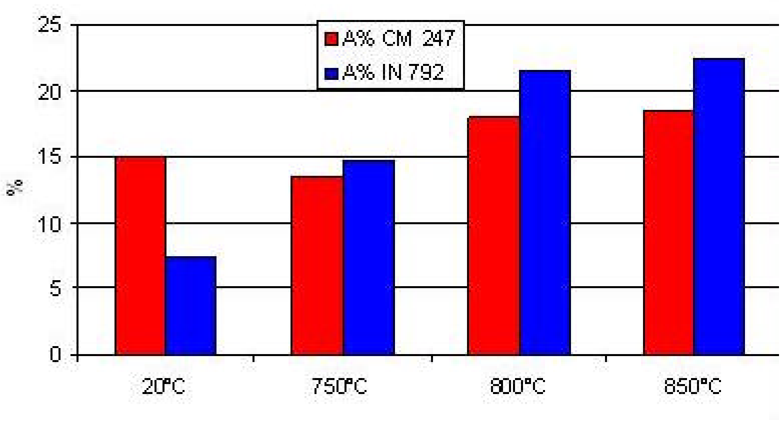


Figure 8 : Comparison of elongation at rupture for CM 247 and IN 792 from 20°C to 850°C.

V.3 Creep properties.

V.3.1 <u>*Creep test results*</u>

Creep tests were performed on both IN 792 and CM 247 grades at 850°C in air. Creep stresses were selected to match the rupture times planned in the test program (~1000h, ~3000h and ~9000h). The details of tests conditions and results are given in appendix 1.

The stresses selected to obtain 1000h, 3000h and ~9000 h to rupture at 850°C are based on bibliographic data found on these materials [ASM 1997] :

- for CM 247 LC DS, 275 MPa (9000h), 325 MPa (3000h) and 375 MPa (1000 h)
- for IN 792 DS, 225 MPa (9000h), 275 MPa (3000h) and 325 MPa (1000 h)

The Figure 9 shows the creep curves obtained for CM 247 at 850°C. It can be seen that the targeted times to failures are reasonably well attained. For all tests, it can be noticed that the elongation at rupture is larger than 20%.

Figure 10 shows the creep curves for IN 792. Like for CM 247, ductility is correct for all tests.

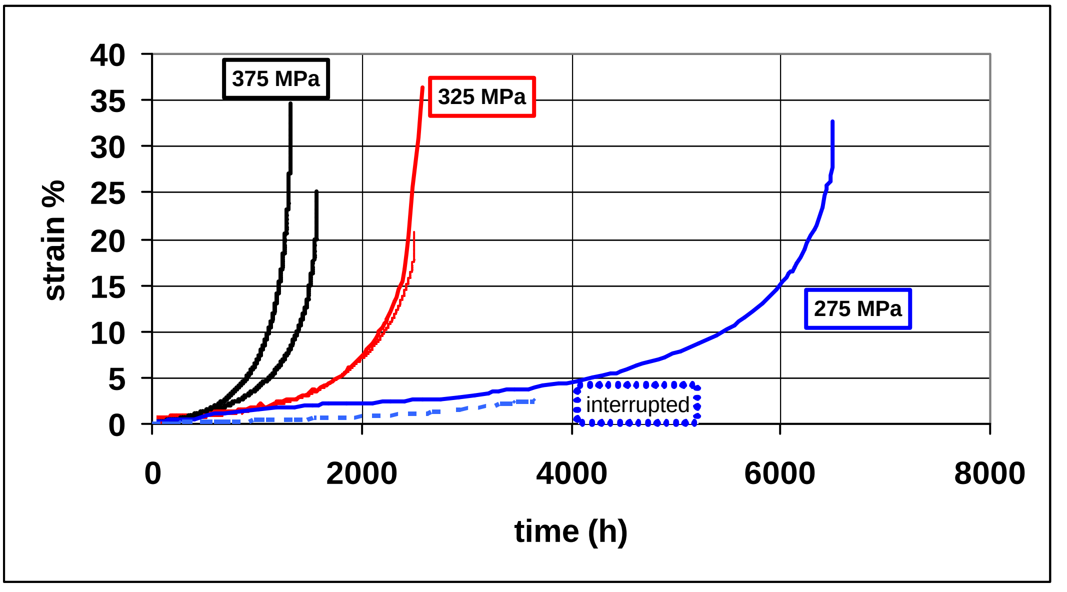


Figure 9 : Creep curves obtained at 850°C for CM 247 grade.

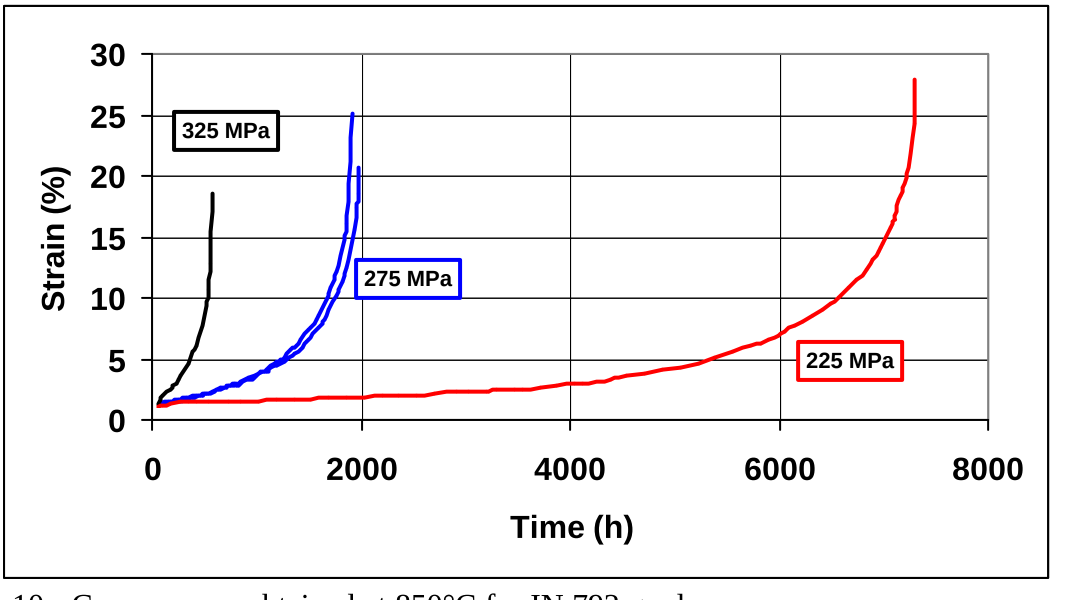


Figure 10 : Creep curves obtained at 850°C for IN 792 grade.

Figure 11 compares the measured creep rupture times with results from the bibliography ([ASM 1997]). It shows that our results are consistent with bibliography for the two blade alloys tested. As for tensile properties, it can also be noticed that the creep strength of CM 247 is superior to that of IN 792.

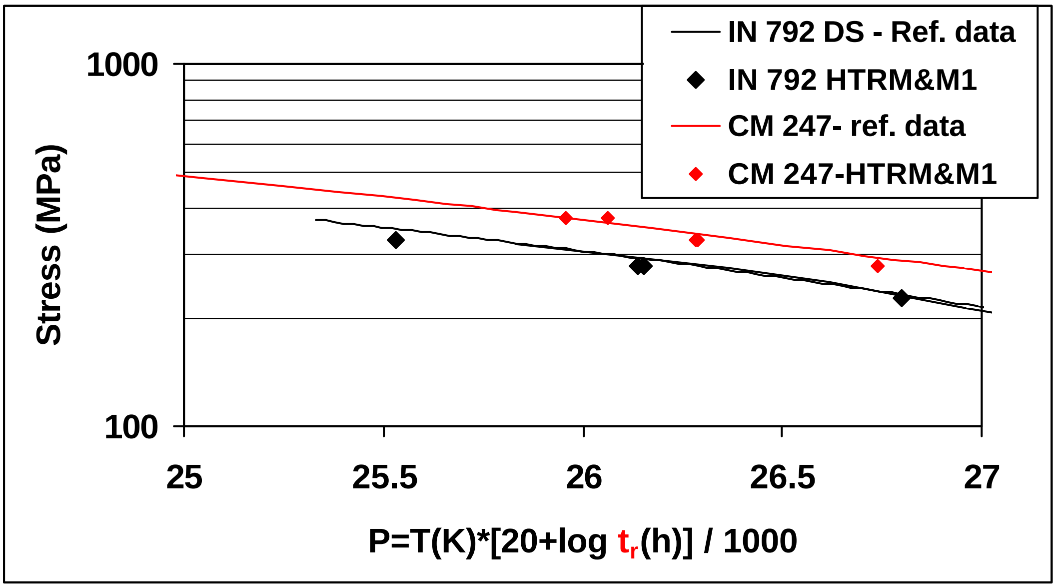


Figure 11 : Comparison of CM 247 and IN 792 creep rupture properties, with the Larson-Miller parameter calculated at rupture.

The Russian team working within ISTC on the design of the GT-MHR turbine has calculated minimum required properties for the blades of the first stage turbine. One of the criteria that can be compared to our results is the required creep stress for a 60000 h lifetime that leads to a 1% creep strain [ITSC 2002]. The ISTC team has proposed a minimum stress of 164 MPa at 830°C for 1% creep strain. This data can be plotted in a Larson Miller graph calculated with the experimental time to reach 1% strain, as shown in Figure 12.

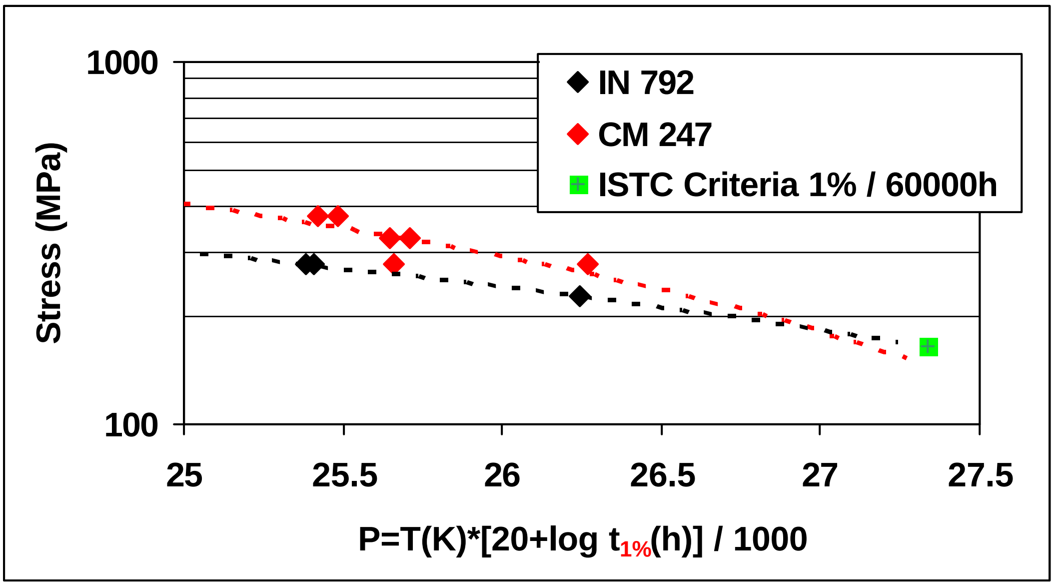


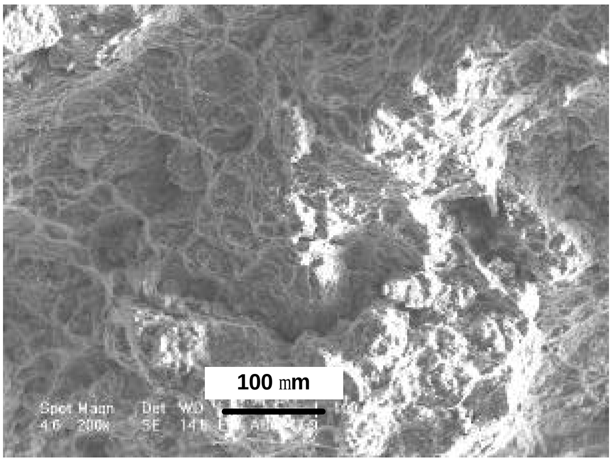
Figure 12 : Larson-Miller plot for 1% creep strain together with ISTC criteria for non cooled turbine blades.

Figure 12 shows that the extrapolation of HTR-M1 results to high P values are close to the data point corresponding to the ISTC criteria. It means that the two selected grades have creep strengths that are close to the required values, but longer term creep tests would be needed to ensure that our grades fulfil the long term ISTC criteria.

V.3.2 *Fracture analysis*

The fracture surface of a CM 247 creep specimen tested at 850°C/275 MPa (tr = 6340 h) has been observed by Scanning Electron Microscopy.
Figure 13 Shows that for this creep test (the longest achieved for CM 247 alloy), the failure mode is ductile with a lot of dimples on the fracture surface. Similar observations were made for IN 792 specimens, and it was also seen that a ductile failure mode is present after long term creep.

Longitudinal sections of ruptured creep specimens were polished and slightly etched to reveal the nature and location of damage. Figure 14 shows two optical views of CM 247 specimen crept at 850°C/275 MPa. Secondary cracks are observed mainly in areas were chemical segregation is present. Cracks close to the surface are oxidised, but the internal penetration of these cracks is rather limited, showing a good resistance of CM 247 to air oxidation. This good resistance is due to the fact that CM 247 is forming an alumina-rich oxide when exposed to air.



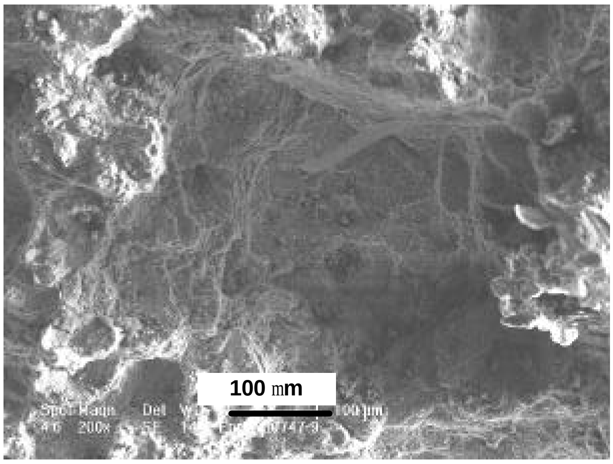
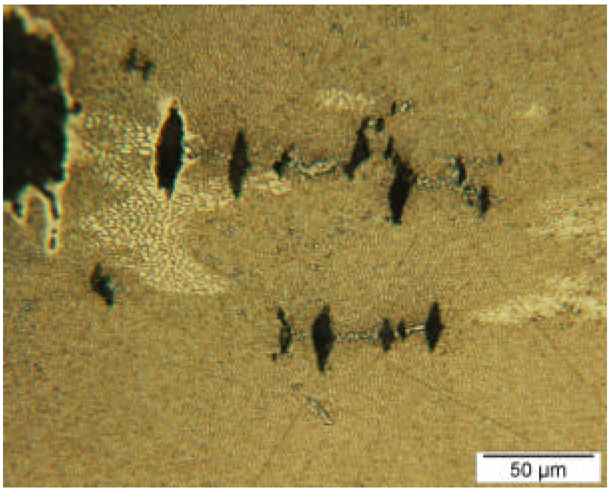


Figure 13 : SEM observation of the fracture surface of CM 247 creep specimen (creep at 850°C/275 MPa, tr = 6340 h).



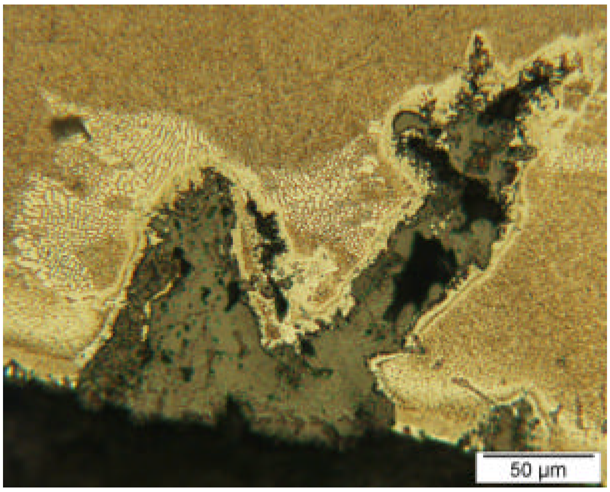
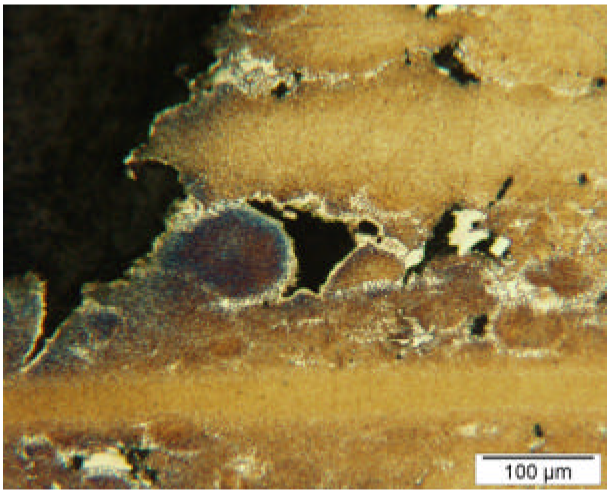


Figure 14 : Longitudinal section (optical metallography) of CM 247 creep specimen (creep at 850°C/275 MPa, tr = 6340 h).

Figure 15 shows a longitudinal cut of IN 792 specimen after a creep tests at 850°C/225 MPa (tr = 7250 h). Like for CM 247, cracks are observed mainly in the interdendritic areas. The surface oxidation of the specimen is more pronounced than for CM 247. A 50 µm thick band without γ' precipitates is observed beneath the surface oxide. IN 792, which is a chromium oxide former, has indeed a lower oxidation resistance.



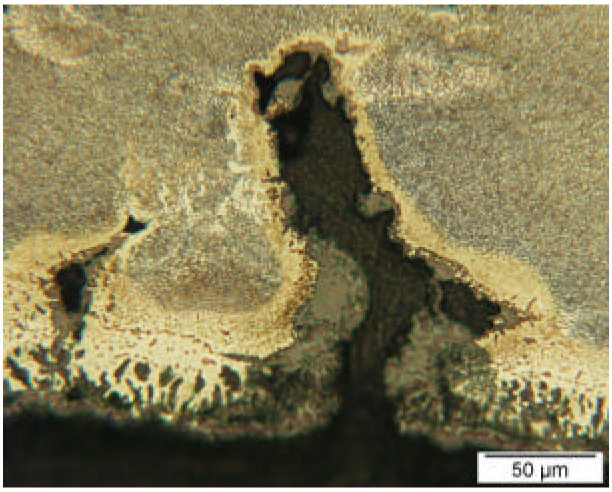


Figure 15 : Longitudinal section (optical metallography) of IN 792 creep specimen (creep at 850°C/225 MPa, tr = 7250 h).

V.4 Effect of the thermal ageing heat treatment (850°C) on mechanical properties.

The HTR-M1 mechanical tests are performed on IN 792 DS and CM 247 LC DS after two types of ageing treatments :

- after a thermal ageing of ~5000 h and ~8000 h at 850°C for IN 792 only.
- after the carburization heat treatment cycle defined by JRC (950°C/1000h) for both IN 792 and CM 247 **(condition denoted I_{mod} in test matrix).** The results will be presented in paragraph V.5.

The ageing heat treatment was performed under air atmosphere (on specimen blanks) for IN792 only, as planned in the test program. Two ageing durations of 5000 h and 8540 h were finally attained before the evaluation of the ageing effects on the mechanical properties.

V.4.1 *Tensile properties*

The effects of the two thermal ageing duration on tensile properties are shown on Figure 16 to Figure 18.

It can be seen that the effect of a long term ageing at the service temperature only results in a slight decrease of strength. Ductility is also decreased, except for the tensile tests performed at 750°C. The room temperature ductility of IN 792 after 850°C/8540 h ageing is rather low, and this would certainly not be acceptable for a use in a turbine within a nuclear plant.
These effects could be explained by a slight coarsening of the γ' precipitates during the thermal ageing, together with the precipitation of few phases (carbides, TCP phases) having an embrittlement effect.

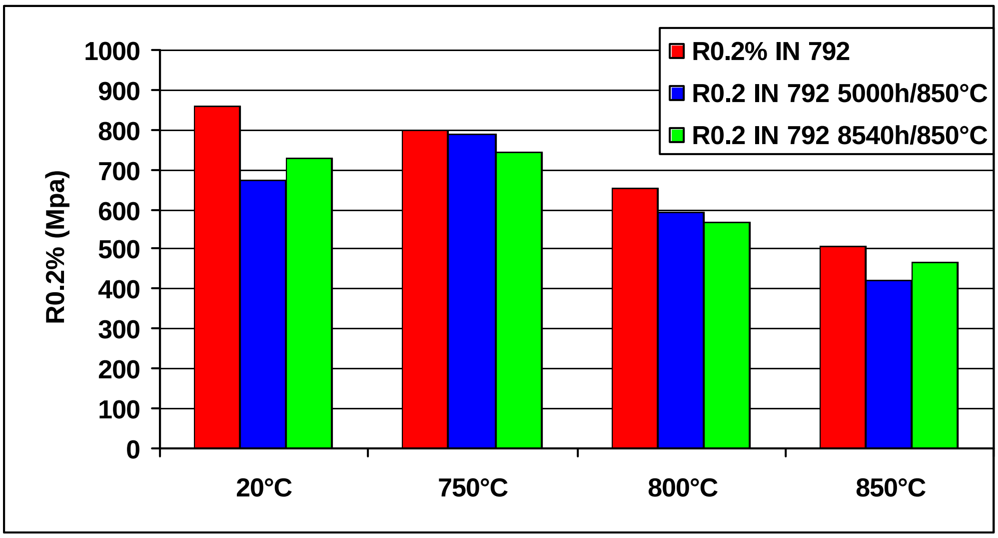


Figure 16 : 0.2% Yield Stress for IN 792 from 20°C to 850°C, in the as received condition and after 5000h and 8540 h of thermal ageing at 850°C.

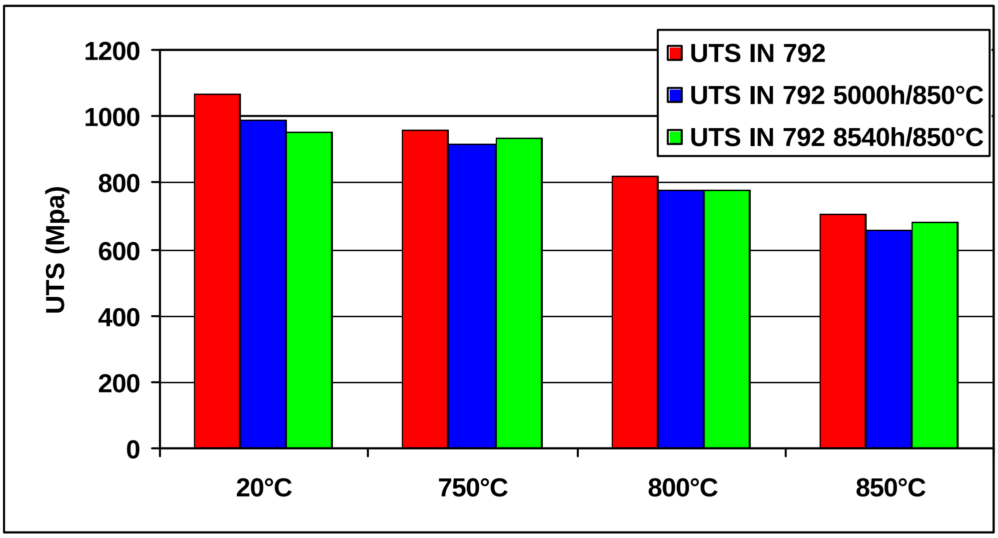


Figure 17 : Ultimate Tensile Strength for IN 792 from 20°C to 850°C, in the as received condition and after 5000h and 8540 h of thermal ageing at 850°C.

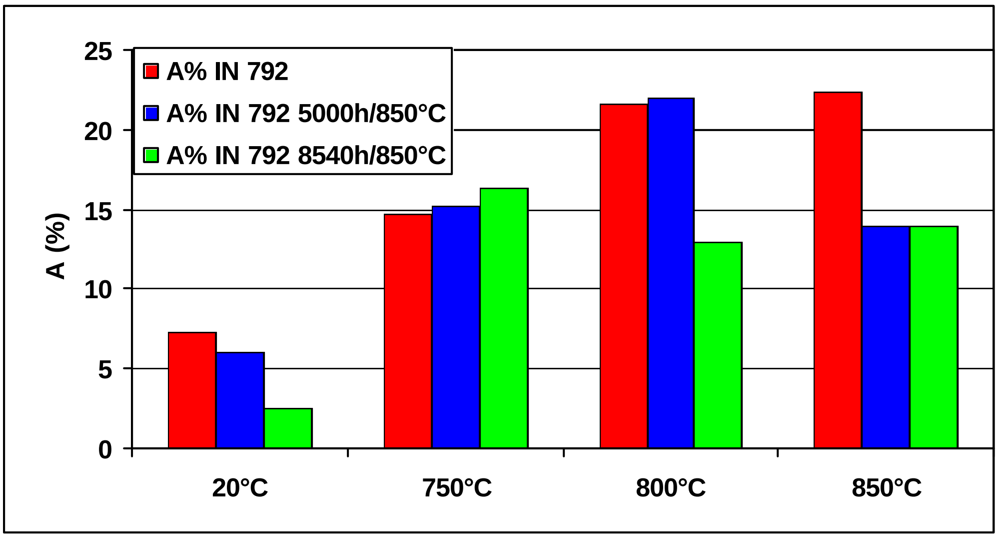


Figure 18 : Elongation at rupture for IN 792 from 20°C to 850°C, in the as received condition and after 5000h and 8540 h of thermal ageing at 850°C.

V.4.2 Creep properties

Figure 19 shows the results of creep tests performed on IN 792 (creep at 850°C/275 MPa) with and without prior thermal ageing of the specimen blanks. It can be seen that the long term ageing performed resulted in a creep lifetime decreased by a factor 2, with a slight decrease of ductility. This results shows that IN 792 does not have a very stable structure at 850°C, and that the properties of this materials are expected to be significantly altered by a long term use as turbine blades.

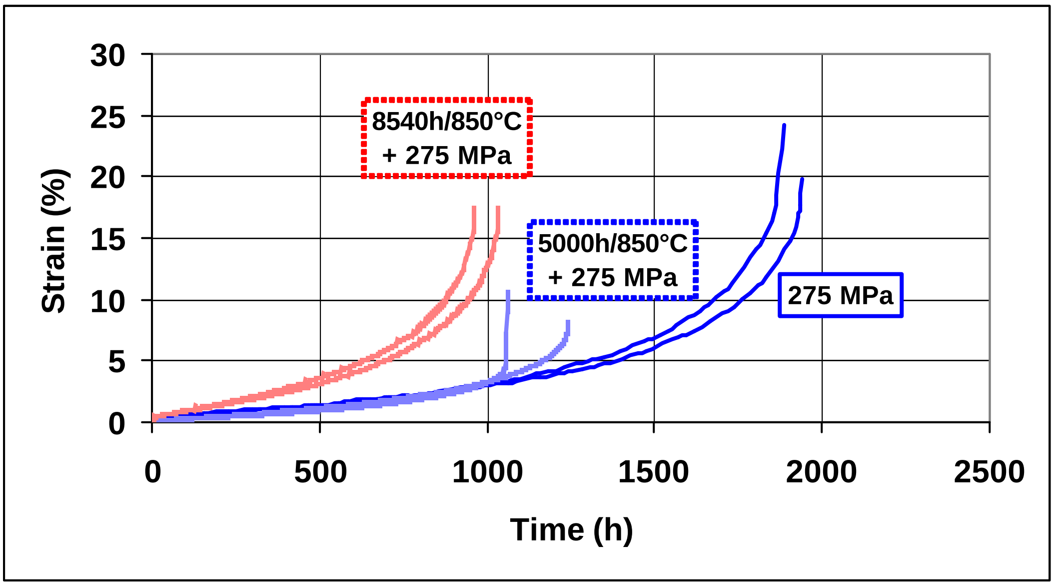


Figure 19 : Creep curves obtained at 850°C/275 MPa for IN 792 specimens with and without prior thermal ageing.

V.4.3 <u>Fracture analysis</u>

Figure 20 shows the fracture surface of a long term aged (850°C/8540 h) IN 792 specimen after creep testing at 850°C/275 MPa. Like for un-aged specimens, it is observed that the rupture mode remains ductile.

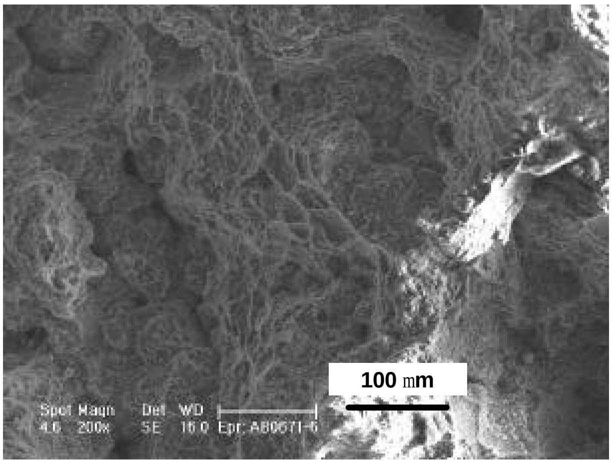


Figure 20 : SEM observation of the fracture surface of IN 792 creep specimen (creep at 850°C/275 MPa, tr = 1035 h after a thermal ageing of 8540h at 850°C).

V.5 Effect of the carburisation heat treatment (950°C) on mechanical properties

The carburization Heat Treatment (HT) I_{mod} was performed under argon atmosphere for CM 247 and IN 792. Due to a problem during the recording of the heat treatment cycle, the duration of the HT was finally 1120 hours instead of 1000 h. This difference should not have a strong impact on the conclusions made after mechanical testing.

V.5.1 <u>*Tensile properties*</u>

Figure 21 presents the 0.2% yield stress of CM 247 and IN 792 after the carburization HT, compared to the 0.2% yield stress of the non heat treated material.

For CM 247 material, the carburisation HT results in a decrease of the yield strength, from -10% to -15% over the test temperature range. Figure 22 also shows that the ultimate tensile strength is slightly decreased (-5% to -10%). We can observe on Figure 23 that the effect on ductility is not pronounced.

For IN 792, the effect of carburisation HT is a slight decrease of yield strength and ultimate tensile strength at room temperature, and a slight increase of strength at high temperature. This effect could be explained by the fact that the carburisation HT resulted in a partial solutioning of the γ' precipitates. The microstructure of IN 792 after the HT is in this case strongly dependant on the cooling rate, and it is possible that the γ' formed during cooling lead to an increase of high temperature strength.

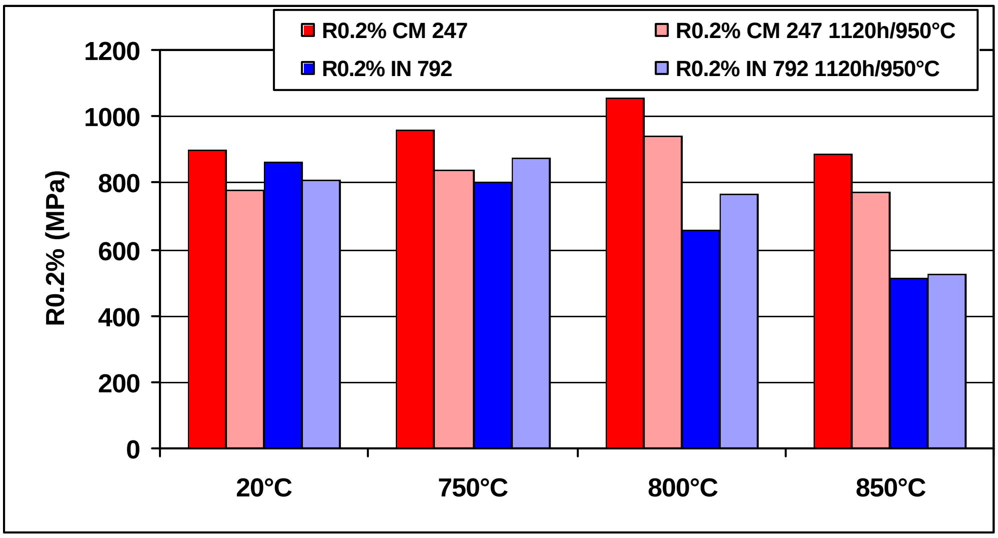


Figure 21 : Comparison of 0.2% Yield Stress for CM 247 and IN 792 from 20°C to 850°C, in the as-received condition and after the carburisation HT.

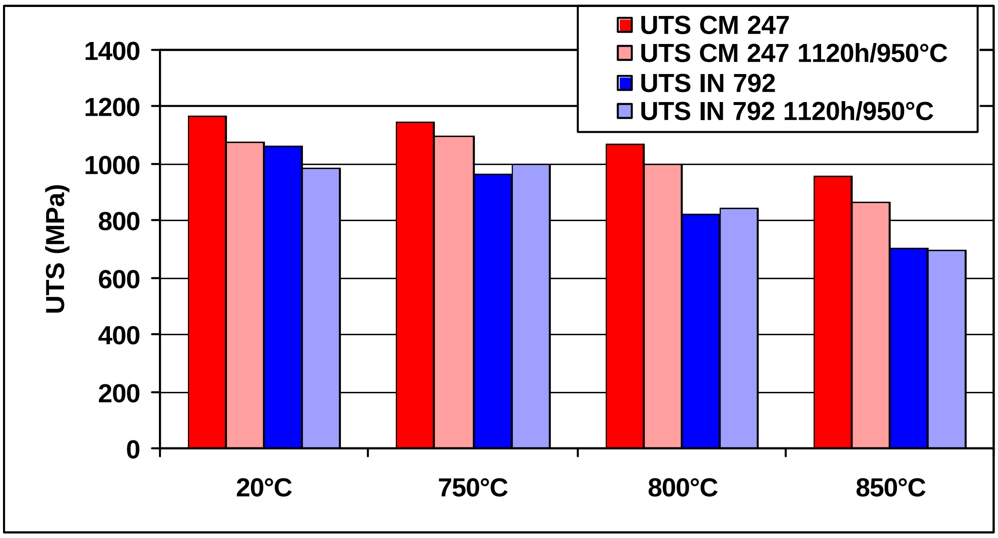


Figure 22 : Comparison of Ultimate Tensile Strength for CM 247 and IN 792 from 20°C to 850°C, in the as-received condition and after the carburisation HT.

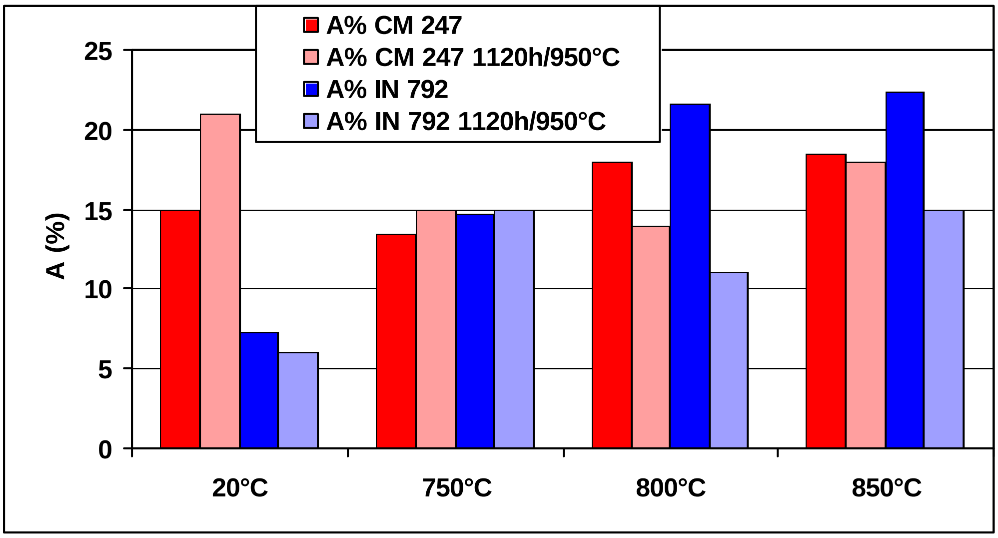


Figure 23 : Comparison of elongation at rupture for CM 247 and IN 792 from 20°C to 850°C, in the as received condition and after the carburisation HT.

V.5.2 *Creep properties*

The creep properties after the carburisation heat treatment have been assessed for the two grades, and the results are presented in Figure 24 and Figure 25. For CM 247 material, the carburisation heat treatment has a strong effect on creep lifetime, with a reduction by a factor 2. For IN 792, only two tests were performed after carburisation HT, and the longest one had to be stopped at the end of HTR-M1 project. The test at 275 MPa also showed an effect of carburisation HT on lifetime, but ductility is unchanged.
To conclude, it can be said that the carburisation HT decreases creep lifetime but has no effect on creep ductility of the 2 blade alloy grades evaluated in HTR-M1.

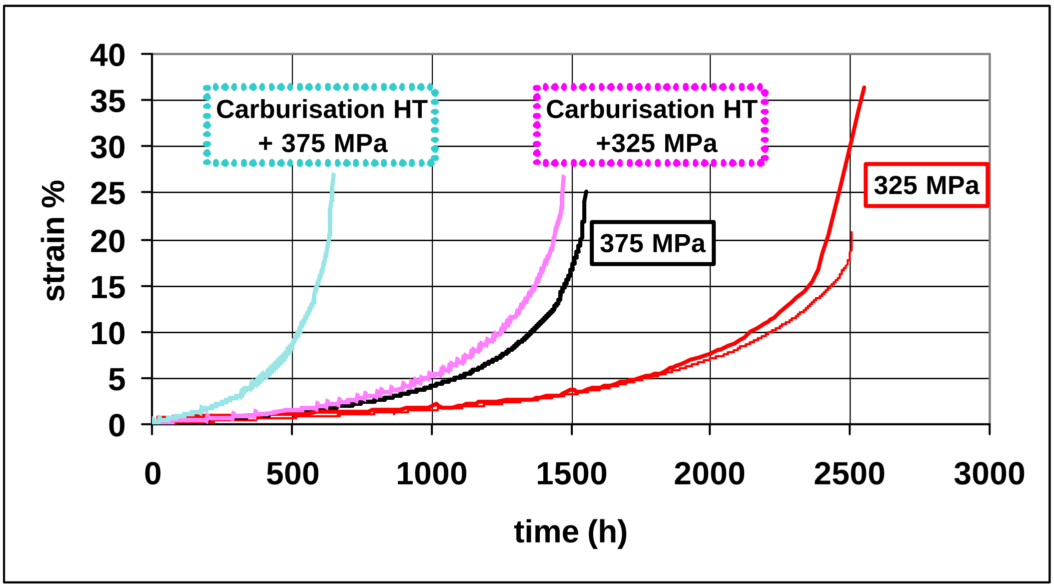


Figure 24 : Creep curves obtained at 850°C MPa for CM 247 specimens with and without prior carburization Heat Treatment.

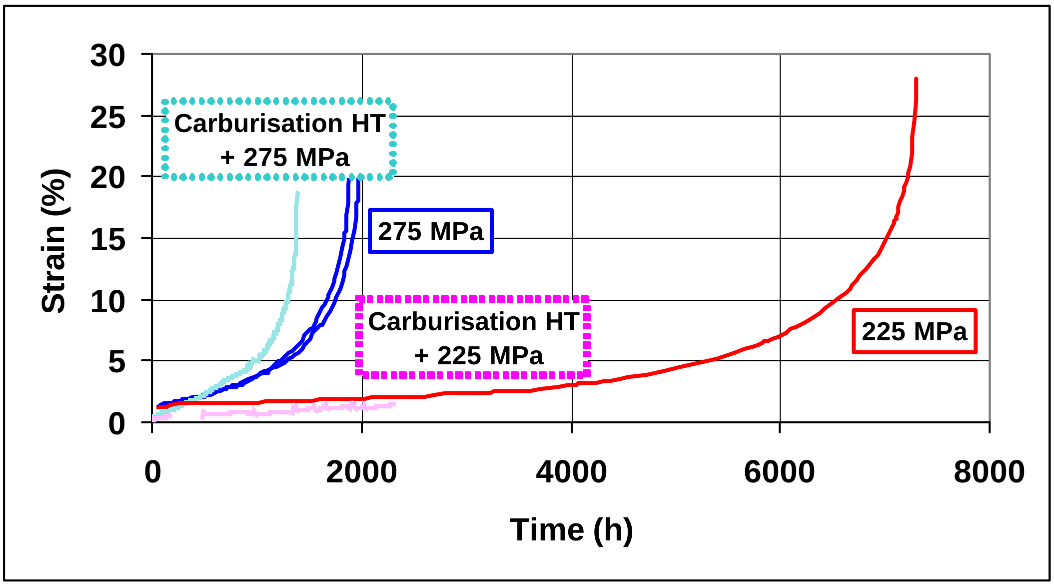


Figure 25 : Creep curves obtained at 850°C MPa for IN 792 specimens with and without prior carburization Heat Treatment.

VI. Experimental results on the carburization facility at JRC : Status at the end of HTR-M1 project.

To test the HTR-M turbine and disc materials at the agreed two extreme conditions – in the carburised and de-carburised conditions – the specimens from the as-received materials had to be "*pre-treated*" at a temperature 950°C in Ar + 2% methane for carburizing and in Ar + 500µbar H_2 + 50µbar H_2O for de-carburizing. This pre-treatment was specified by FZ Juelich based on their previous experience with similar materials.

A special facility had to be built for these treatments, but due to unavailability of technician manpower the fabrication of this facility was seriously delayed and after completion did not function properly until the technical problems were solved. The facility consists of a furnace with a specimen chamber with a 45mm diameter and a homogeneous temperature zone of 200mm. A gas mixture can be fed through the specimen chamber. For the de-carburisation treatment a moisturiser will be used to provide the small moisture content.

In July 2004 the first dummy carburisation test on pieces of IN617 material (from another project) was started. After the treatment the pieces were analysed by FZJ confirming that a carburisation depth of 0.9mm was achieved. Also some shrinkage porosity and internal oxidation of Al was observed in this layer.

The original planning had been to pre-treat specimen blanks and machine the specimens after the treatment, but because carburisation took place only on the surface then machined specimens had to be treated instead. Even then the whole specimen cross-section will not be carburised, but a reasonable proportion of the cross-section will be affected by the carburisation and the effect on creep properties will be observed.

At the date of the official end of HTR-M1 project (12/05), the results are not available for blade alloys.
The results of mechanical tests after carburisation will be obtained by JRC after the end of HTR M1 and will be reported in 2006 within RAPHAEL-IP project.

VII. Conclusion

This report has given a final synthesis of mechanical tests performed within HTR-M1 program.

The tensile and creep tests performed on two grades of blades (CM 247 and IN 792) resulted in the completion of a "property database" that will be used in future programs (RAPHAEL IP) to assess the effect of carburisation on properties.

The experimental results can be used to draw the following conclusions :

- CM 247 shows the best mechanical properties (tensile, creep) at high temperature. It would be necessary to perform longer term creep tests (at least 20000 h) to confirm whether the ISTC mechanical criteria for blades are fulfilled with this grade.

- A thermal ageing of nearly one year at 850°C leads to a slight decrease of tensile strength and high temperature ductility for IN 792, but room temperature ductility is severey altered. The decrease of creep strength observed after ageing is more pronounced. These results would need to be confirmed by longer ageing to see if the property drop continues for longer durations.

- The thermal cycle associated with the carburisation performed at JRC (1000h/950°C) has a moderate effect on tensile strength for the two grades. The decrease of creep strength is again more pronounced. This effect will have to be taken into account when we will compare the properties of carburised specimens to that of non-carburised ones.

This HTR-M1 project has shown that Directionnaly Solidified nickel bas alloys are good candidates for the use as HTR turbine blades, but the long term stability of the alloy microstructure is a key point that would need to be further evaluated to ensure a safe use of the turbine.

VIII. References

[ASM 1997] : "ASM specialty handbook, Heat resistant materials", Ed. By J.R. Davis, ASM International, 1997.

[ISTC2002] : "Testing and investigations of materials for turbocompressor high temperature components", A. Romantsov, ISTC Report 1313-2001, 2002.

IX. Appendix 1 : mechanical tests results

Tensile tests

Tensile tests are done with an electro-mechanical 100 kN machine, with a specimen geometry shown in figure A1-1. The gage length of the specimens is 20 mm, strain is measured by a LVDT connected to metallic arms attached to the ridges. The results of tensile tests are given A1-1 and A1-2 for blade material.

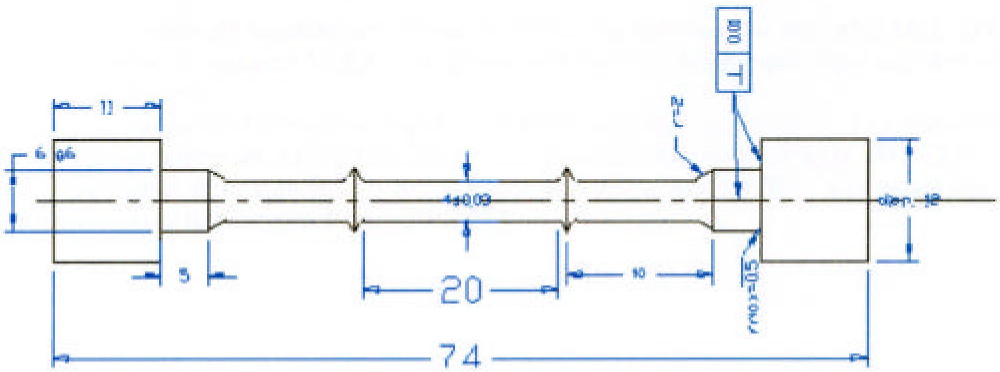


Figure A1-1 : Tensile specimen geometry.

Ageing	Specimen ref.	File n°	T (°C)	Strain rate (s^{-1})	0.2% PS (MPa)	UTS (MPa)	A%	Z%
-	AB0747-1	2003-254	20	5.10^{-4}	901	1165	15	18
-	AB0747-2	2003-255	750	5.10^{-4}	960	1150	13.4	17
-	AB0747-3	2003-256	800	5.10^{-4}	1054	1066	18	22
-	AB0747-4	2003-257	850	5.10^{-4}	889	960	18.5	24
1120h/950°C	AB0746-1	2005-022	20	5.10^{-4}	775	1075	21	16
1120h/950°C	AB0746-2	2005-023	750	5.10^{-4}	840	1095	15	26
1120h/950°C	AB0746-3	2005-024	800	5.10^{-4}	940	1000	14	23
1120h/950°C	AB0746-4	2005-025	850	5.10^{-4}	770	870	18	28

Table A1-1 : Tensile tests results on CM 247 LC DS blade material.

Ageing	Specimen ref.	File n°	T (°C)	Strain rate (s^{-1})	0.2% PS (MPa)	UTS (MPa)	A%	Z%
-	DA9875-1	2004-045	20	5.10^{-4}	860	1065	7.4	11
-	DA9875-2	2004-042	750	5.10^{-4}	800	961	14.7	25
-	DA9875-3	2004-043	800	5.10^{-4}	655	822	21.6	30
-	DA9875-4	2004-044	850	5.10^{-4}	510	703	22.4	32
1120h/950°C	DA9877-1	2005-018	20	5.10^{-4}	805	985	6	10
1120h/950°C	DA9877-2	2005-019	750	5.10^{-4}	875	1000	15	23
1120h/950°C	DA9877-3	2005-020	800	5.10^{-4}	765	845	11	25
1120h/950°C	DA9877-4	2005-021	850	5.10^{-4}	525	695	15	35
5000h/850°C	AB0670-1	2004-179	20	5.10^{-4}	670	990	6	8
5000h/850°C	AB0670-2	2004-180	750	5.10^{-4}	790	920	15	32
5000h/850°C	AB0670-3	2004-181	800	5.10^{-4}	590	775	22	37
5000h/850°C	AB0670-4	2004-182	850	5.10^{-4}	425	660	14	41
8540h/850°C	AB0671-1	2005-005	20	5.10^{-4}	730	950	2.5	7.5
8540h/850°C	AB0671-2	2005-006	750	5.10^{-4}	745	930	16.3	30
8540h/850°C	AB0671-3	2005-007	800	5.10^{-4}	570	775	13	35
8540h/850°C	AB0671-4	2005-008	850	5.10^{-4}	465	685	14	40

Table A1-2 : Tensile tests results on IN 792 DS blade material.

Creep tests

Creep tests are performed on 20kN Mayes machines. Creep stress is applied by dead weights with a 10:1 lever ratio. Creep specimens have the same geometry as tensile specimens. Table A1-3 to A1-7 give the results for CM 247 LC DS and IN 792 LC DS grades.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
AB0747-5	01-001	850	325	5.4	2535	36	48	67	448	780	1250	1740	3.10^{-9}	
AB0747-6	03-115	850	375	3.1	1599	27	48	25	300	490	730	1100	3.10^{-9}	
AB0747-8	04-018	850	375	4.2	1286	35	47	160	300	430	600	875	4.10^{-9}	
AB0747-7	04-027	850	325	2.7	2530	22	49	155	350	686	1175	1775	3.10^{-9}	
AB 0749-9	05-010	850	275	4.6	6340	33	52	300	400	700	2000	4200	$2.\ 10^{-9}$	
AB0747-10	05-004	850	275	4.6	>3668	>2.6	-	650	1350	2400	3300	-	1.10^{-9}	interrupted

Table A1-3 : Creep tests results on CM 247 LC DS alloy.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
DA9875-6	-	850	325	5.4	540	28	40	10	28	75	185	370	3.10^{-8}	
DA9875-5	04-035	850	275	4.6	1929	20	38	-	120	400	810	1390	6.10^{-9}	
DA9875-7	04-034	850	275	4.6	1876	24	42	50	160	420	780	1350	5.10^{-9}	
DA9875-8	05-016	850	225	5	7253	27	41	70	700	2300	4000	5700	1.10^{-9}	

Table A1-4 : Creep tests results on IN 792 DS alloy.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
AB0670-5	05-008	850	275	4.6	1242	18	40	60	200	430	760	1170	7.10^{-9}	
AB0670-6	05-013	850	275	4.6	1060	17	43	90	295	540	840	1060	5.10^{-9}	

Table A1-5 : Creep tests results on IN 792 DS alloy after 5000h/850°C thermal ageing.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
AB0671-5	05-018	850	275	4.6	963	18	33	~0	25	110	290	640	$1\ 10^{-8}$	
AB0671-6	05-019	850	275	4.6	1035	19.4	34	~0	25	127	330	700	$1\ 10^{-8}$	

Table A1-6 : Creep tests results on IN 792 DS alloy after 8540h/850°C thermal ageing.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
DA9877-5	05-027	850	275	4.6	1387	21	43	5	40	190	460	1000	$1\ 10^{-8}$	
DA9877-6	05-046	850	225	4.6	>2319			20	230	1390			$1\ 10^{-9}$	interrupted

Table A1-7 : Creep tests results on IN 792 DS alloy after 1120h/950°C thermal ageing.

Specimen n°	File n°	T (°C)	s (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					De/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
AB0746-5	05-033	850	325	5.4	1473	28	47	15	170	380	620	980	$6\ 10^{-9}$	
AB0746-6	05-021	850	375	402	651	29	42	6	50	120	230	401	$2\ 10^{-8}$	

Table A1-8 : Creep tests results on CM 247 LC DS alloy after 1120h/950°C thermal ageing.