EUROPEAN COMMISSION
5th EURATOM FRAMEWORK PROGRAMME 1998-2002
KEY ACTION : NUCLEAR FISSION

HTR-M

European Project for the development of HTR Technology - Materials for the HTR

CONTRACT N°
FIKI-CT-2000-00032
&
FIKI-CT-2001-00135

Materials for the Turbine: Final Report

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Dissemination level : CO
Document Number: HTR-M-04/10-D-2-3-103
Deliverable 2.10

Direction de la recherche technologique
Département des technologies pour l'énergie et les nanomatériaux
Service Modélisation, Mécanique et Milieux Extrêmes - S3ME
Laboratoire Mécanique et Intégration des Composants - LMIC

Rapport technique DTEN/DL/2005/013

HTR-M Deliverable 2.10

« Materials for Turbine : Final Report »

Livrable RCGMA-R7

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Référence du contrat	769134
Référence PRODEM	00-00181-C
Nature du rapport	Final
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Abstract

The objective of this report is to present the results of mechanical tests and microstructural analyses obtained for both blade and disk alloys investigated in the European projects HTR-M (Materials for High Temperature Reactors).

For turbine disk material, the properties of forged U720 superalloy seem appropriate to the design requirements of a cooled turbine for HTR (High Temperature nuclear Reactor). This alloy, used with a specific supersolvus heat treatment to increase the grain size, has a creep strength that is much higher than that of conventional U720 with a fine grained microstructure. Fatigue and tensile properties are not strongly affected by the duplex grain size.

For the turbine blade material, two grades of directionally solidified superalloy (CM 247 LC DS and IN 792 DS) have been subjected to tensile and creep tests. The microstructure observed by TEM for CM 247 shows the classical distribution of gamma prime phase within the matrix. Low angle grain boundaries are decorated with small precipitates enriched in W and Cr.

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.

Tests on carburised and decarburised blade material will be achieved within HTR-M1 and compared to the data presented in this report, in order to obtain a first assessment of the effect of the specific HTR environment.

Key Words

HTR
Turbine
Superalloys
U720, CM 247, IN 792

CONTENTS

I. Introduction- Selection of materials

The objective of this report is to present the synthesis of the work performed for both turbine blade and turbine disk alloys in the frame of HTR-M project.
Material procurement details and the full test matrix for both HTR-M and HTR-M1 projects are first presented, but only HTR-M experimental results are discussed in this document.

This final report gives a general overview of HTR-M experimental tests results obtained by JRC and CEA. Intermediate results were already presented in deliverable 2.9 (HTRM-03/12-D-2-3-84).

HTRM&M1 general objectives :

The need for reliable material data and properties is a key issue in the development of any innovative reactor technology and is especially important for those areas where safety considerations are of prime interest. The **HTR-M/M1 project** (HTR-M1 project was started one year after HTR-M to complete the test matrix) deals with the selection and development of materials for the vessel, high temperature components and the graphite core. The project includes specific experiments on selected materials, including irradiation tests on vessel weldments and graphite. The aim of the HTR-M/M1 project is to provide a materials platform on which to develop the future HTR technological basis within Europe and an understanding of the behaviour and manufacturing requirements for some of the new materials that will be needed for such developments.

Within HTR-M the second Work Package (WP2) deals with high temperature materials, and the task 2.1 is especially dedicated to the study of turbine materials.

The objectives of this “turbine materials” task are the following :

- To review available materials for turbine discs and blades applications.
- To select reference materials and perform short term (tensile, creep, fatigue) and medium term (creep) tests under HTR relevant conditions representative of the He environment and temperature.
- To set up a materials database on properties (tensile, fatigue and creep properties at high temperature)

In addition to HTR-M project, HTR-M1 has the objective to complete the tests performed on blade alloys by tensile and creep tests after a long term thermal ageing.

Material selection :

The review 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 turbine discs there is a requirement to limit the permanent growth and distortion to within typical design life targets. Critical regions for the disc are the hottest and highly stressed parts (high stress and moderate temperature at the hub, low stress and high temperature at the rim).
Discussions with the turbine materials manufacturer Aubert & Duval identified UDIMET 720 as the best available candidate. This alloy can be processed by powder metallurgy and Hot Isostatic Pressing (HIPping) or by forging for small diameters. Although the literature search showed few published data on this material, the operating temperature for UDIMET 720 is thought to offer the highest at around 700^{o}C (which suggests that a non-cooled disc operating at 850^{o}C may be too ambitious). For large diameter disks, IN706 on the other hand operates successfully at temperatures of 550-600^{o}C (steam turbines) and IN718, a further possible material, is near to commercial production but R& D is still required in order to control the grain size (T_{max}~610°C).

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 (HTR-M 02/4-D-2.2.32).

Finally, the materials selected for mechanical testing within HTR-M & M1 are as follows:

Turbine disc:	**UDIMET 720 (Cast &Wrought)**
Turbine blade:	**IN 792 DS and CM 247 LC DS (Directionally Solidified)**

Information on the test programme and planning for the tests has been reported for HTR-M (HTR-M 02/6-D-2.2.35) and HTR-M1 (HTR-M1 02/9-D-1.1.7). Since the impurity levels in the helium atmosphere are not well known it was decided to test the selected alloys under extreme carburising and decarburising conditions by exposing the specimens before testing to different environments to gain information on the likely strength variations expected. An argon or air atmosphere could then be used for the tests.

Specimens (or blanks) are machined from the as-received materials and then “*pre-treated*” (temperature 950°C) in Ar + 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 :

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

Ø material entirely de-carburized: denoted **“state II”**

Ø material heavily carburized: denoted **“state III”**

Ø material with additional treatment (heat treated at 950°C or 850° depending on *pre-treatment*, time same as pre-treatment, to separate thermal ageing effects from the effects of decarburization or carburization: denoted **“state Imod”**)

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 tests are performed under air atmosphere.

II. Material procurement and test matrix

II.1 Disk material

A fully heat treated U720 forged pancake (Material certificate n° HY540203) was ordered to Aubert&Duval and delivered in october 2002. This pancake has a disk shape with a diameter of 320 mm and a height of 80 mm. The cost of this pancake is 9430 euros, its chemical composition is given in Table 1. Figure 1 shows the as-received disk that has been cut into 2 equal parts (1 for CEA and 1 sent to JRC).

Ni	**Cr**	**Co**	**Ti**	**Mo**	**Al**	**W**	**Fe**	**Mn**	**Si**
Balance	16.02	14.81	5.1	2.98	2.63	1.19	0.1	0.03	0.02
B	**Cu**	**C**	**P**	**S**	**N_2**	**O_2**			
0.014	0.01	0.009	<0.005	<0.002	<0.002	<0.002			

Table 1 : Chemical composition of U720 test material.(weight %).

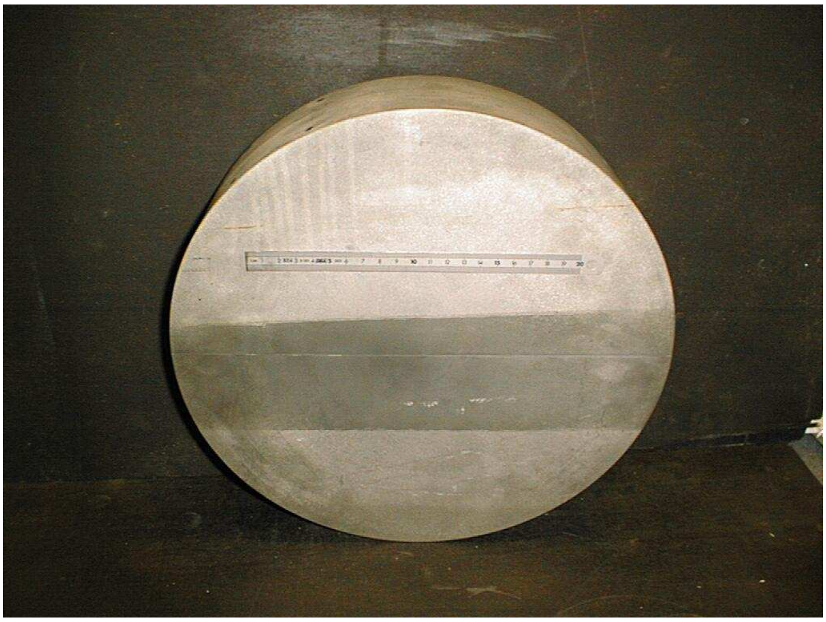


Figure 1 : U720 forged disk used in HTRM project (ϕ=32 cm).

The heat treatment of the disk has been adapted to the HTR specifications, where creep and thermal stability are of major concern. This heat treatment includes a two steps (supersolvus + subsolvus) solutioning for grain growth and γ' precipitation and a typical ageing sequence for creep resistance :

- Solutioning at 1157°C for 4 hours, air quenched
- Partial solutioning at 1090°C for 4 hours, oil quenched
- Ageing at 845°C for 24 hours, air cooled
- Ageing at 760°C for 16 hours, air cooled

We can notice that the first solutioning temperature is very close to the γ' solvus temperature (~1153°C) to closely control the γ grain size.
The test matrix, considered as a "minimum required test matrix" is presented in Table 2 for each material condition. Apart from these mechanical tests, an analysis of the microstructure is also performed. Tests for conditions Imod, II and III have not been performed since the JRC carburization facility was not yet available at the end of HTR-M project.

Test / n° of specimens	**T (°C)**	**I**	**I_{mod}**	**II**	**III**
Tensile	20 650 700 750	2 2 2 2	2 2 2 2		
Creep ~400 MPa (3000h) ~500 MPa (1000h)	750	4	4	2	2
LCF (3Δε)	650	4	6	10	

Table 2 : HTR-M Test matrix for U720 disk material.

II.2 Blade material

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 3.

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

<u>*Heat treatment problem with IN 792 DS :*</u>

Unfortunately, we only 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 (HTR-M1 program).

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”.

The HTR-M mechanical test matrix is presented in Table 4 for each material condition. The HTR-M1 test matrix presented in Table 5 is defined for IN 792 DS material aged 10000 hours at 850°C.

Test / n° of specimens	**T (°C)**	**I**	**I_{mod}**	**II**	**III**
Tensile	20 750 800 850	1 1 1 1	1 1 1 1		
Creep x MPa (3000h) y MPa (1000h) z MPa (5-10 kHr)	850	2 2 2	2* 2* 2	2 2 2	2 2 2

Table 4 : HTR-M test matrix for the blade alloys. (* : only 1 specimen for CM 247)

Test / n° of specimens	**T (°C)**	**I**	**I_{mod}**	**II**	**III**
Tensile	20 800 850	1 1 1			
Creep x MPa (3000h) y MPa (1000h)	850	3		4	4

Table 5 : HTR-M1 test matrix for IN 792 DS aged 10 000 hours at 850°C.

III. Experimental results on forged U720 disk material

III.1 Microstructural examinations

A detailed analysis of the material structure was performed by optical metallography. Figure 2 shows the different phases that can be seen with conventional metallography : primary and secondary γ' precipitates and carbides at the γ grain boundaries.

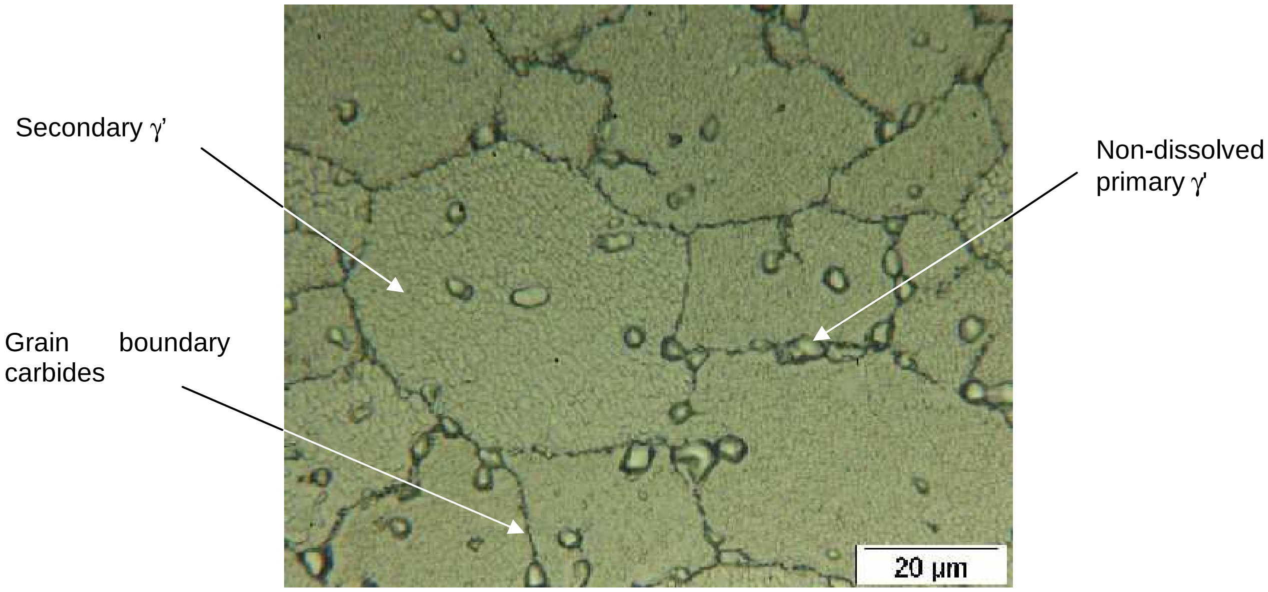


Figure 2 : General microstructure of forged U720.

The first examinations have shown a large variability in the grain size with the location of the observed area in the disk. Therefore, it was decided to perform a full mapping of the grain size within the disk. For this purpose, a thin slice (2 mm thick) was cut in the middle of the disk. This slice was then cut in 10 parts for an easier surface preparation (polishing, etching).
Figure 3 shows the results of this macrographic examination. We observe a typical "texture" of forged materials. The four parts presented in Figure 3 were then analysed with optical micrography, and the results are presented in Figure 4 (a) to 4 (d) .

The grain size is heterogeneous, with bands of coarse grains (500 µm) separated by areas where the grain size does not exceed 50 µm. This duplex grain size is the combined result of forging and heat treatment. During forging, primary gamma prime precipitates are distributed within the material with a non uniform localization. Moreover, all the primary γ' have not the same precise dissolution temperature, as a result of local chemical segregations during the ingot remelting and conversion. When the supersolvus heat treatment is applied (T°=1157°C, just above $T\gamma'_{solvus}$), some γ' will dissolve, allowing the γ grains to grow, and some will not dissolve and pin the grains.

These microstructural observations show the difficulty to achieve a uniform grain size with forged U720 during the supersolvus heat treatment, as local variations in the γ' chemistry can lead to grain sizes varying from 50 to 500 µm.
Measurements of hardness have also been performed in the disk , near the surface and in the bulk. Because of the heterogeneous grain size, the hardness scatterband is large and no surface/bulk effect was detected. The "average" hardness of the material has been measured at 410 ±15 HV30.

As a consequence of microstructural heterogeneity, each specimen has a label that can be related to its location within the disk. The precise location of each specimen can be found in Appendix 1..

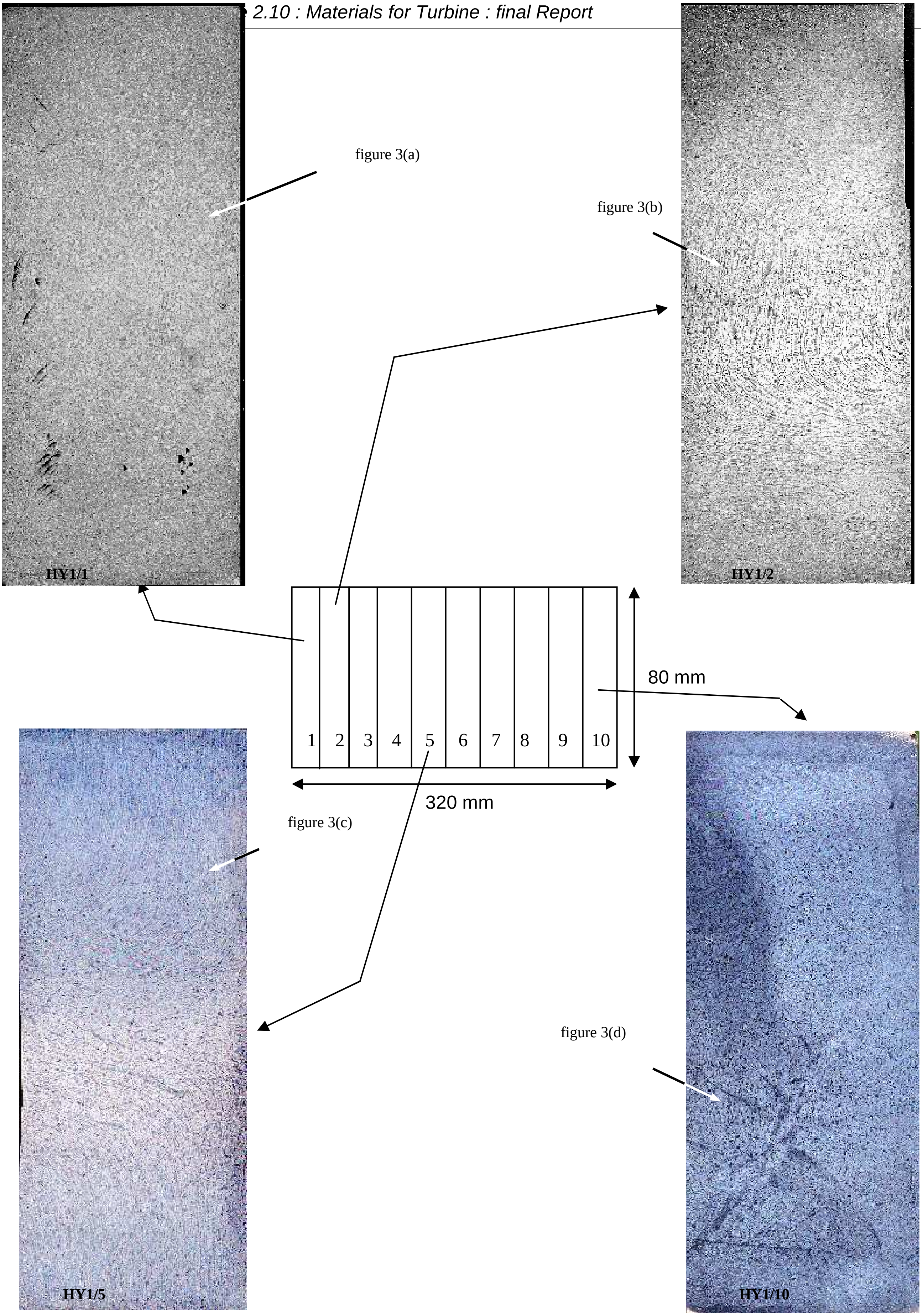


Figure 3 **:** Macrostructure of a 2 mm thick diametral cut of U720 forged disk.

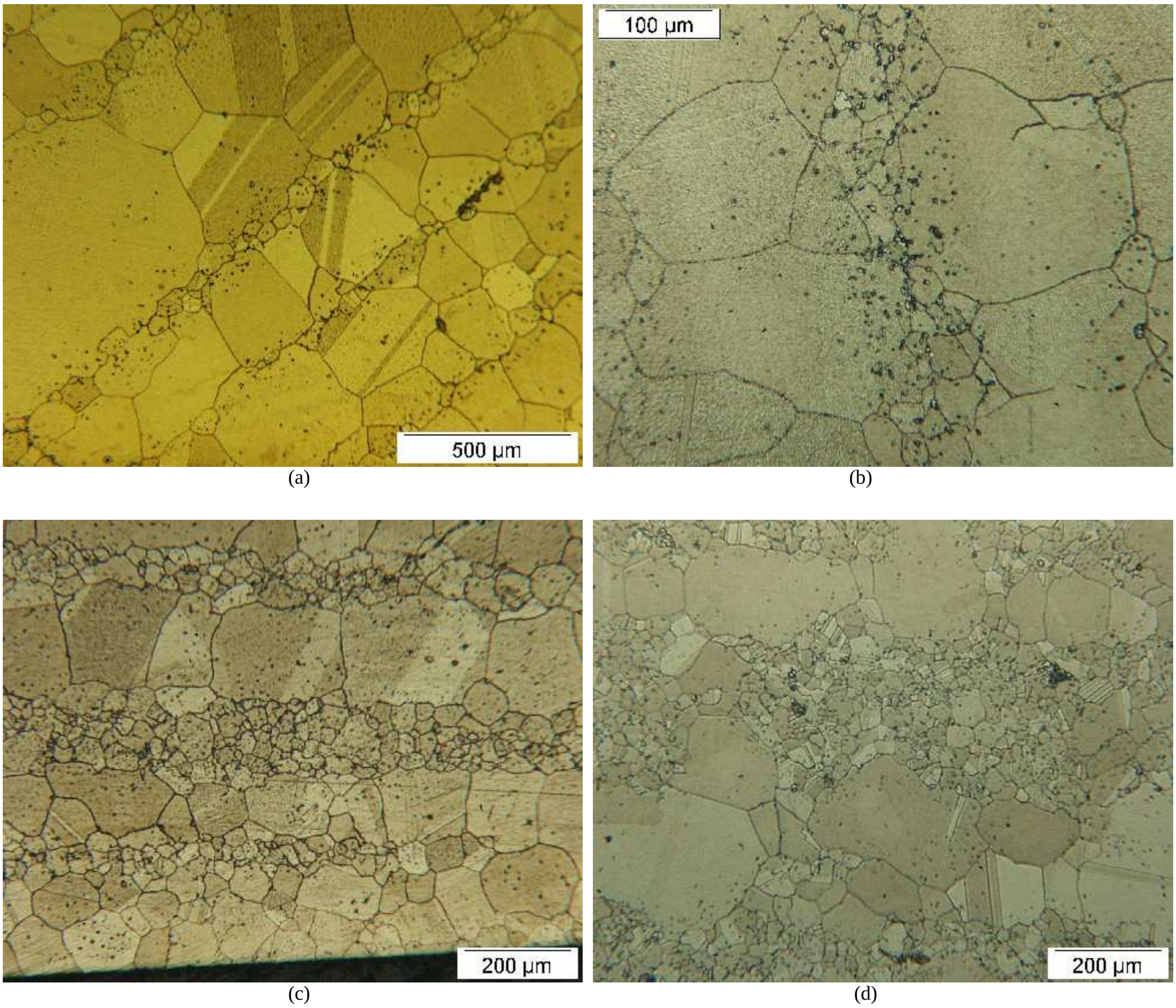


Figure 4 **:** Microstructural observations of U720 disk. (a) HY1/1 (b) HY1/2 (c) HY1/5 (d) HY1/10.

TEM examinations

TEM examinations have been made on the part HY1/1, with thin foils machined near the surface and in the bulk of the disk. No difference in the precipitate size, nature or morphology was detected, and therefore only the “surface” observations are shown in

Figure 5 and Figure 6.

Figure 5 shows the secondary and tertiary gamma-prime precipitates in the matrix and Figure 6 shows primary γ' within grains (b) and at the grain boundaries (a). Primary γ' at the grain boundaries are often surrounded by $M_{23}C_6$ carbides. Some primary γ' within the grains are also surrounded by $M_{23}C_6$ carbides.

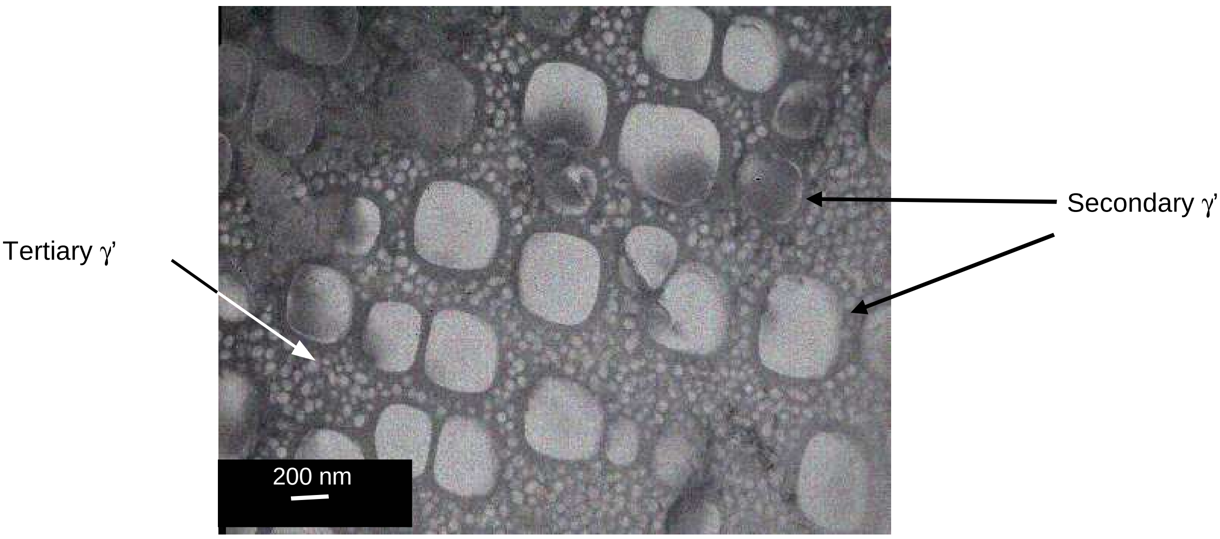
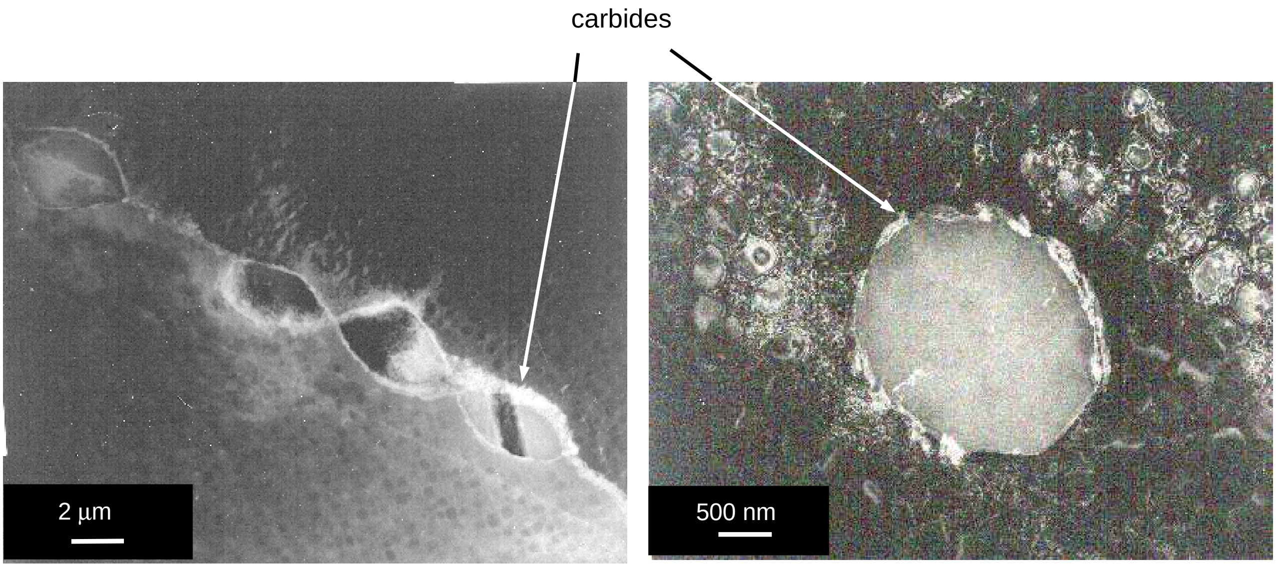


Figure 5**:** TEM observation of the smallest γ' precipitates.



(a) (b)

Figure 6: TEM observation of the primary γ' precipitates and carbides.

To conclude on the microstructural study, we can say that the U720 forged disk delivered by Aubert&Duval has a microstructure with the following parameters :

- The grain size ranges from 10 µm to 500 µm (duplex grain size).
- Three populations of γ' precipitates are present : primary γ' (ϕ = 2-3 µm), secondary γ' (ϕ = 200-650 nm) and tertiary γ' (ϕ = 50 nm).

III.2 Tensile properties

Tensile tests have been performed at 20, 650, 700 and 750°C with a strain rate of 5.10^{-4} s^{-1}. A detailed analysis of the results is given in Appendix 2, together with the tensile specimen geometry. In the following analysis, the results are compared to the usual properties of forged U720 with a supersolvus heat treatment. The corresponding data have been given by A&D and are presented in Appendix 3.
The Figure 7 shows the tensile curves that were recorded.

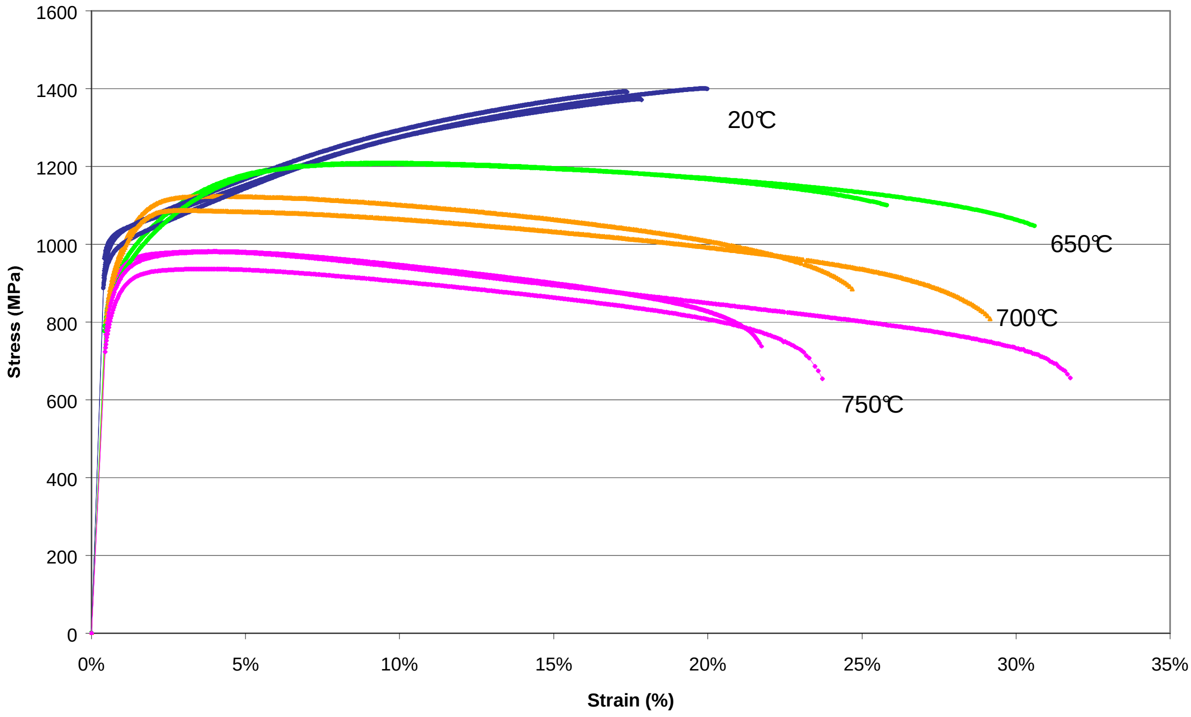


Figure 7 : Tensile stress/strain curves obtained on U720 forged material.

Figure 7 shows that ductility is slightly increasing with temperature but remains correct for all temperatures. At room temperature, rupture occurs without necking. It also shows that the hardening capability of U720 decreases with temperature. The results are not really scattered despite of the microstructural heterogeneity. Moreover, the observed scatter could not be attributed to the location of the specimens within the disk.

Figure 8 compares the 0.2% yield stress (0.2% PS for Proof Stress) and maximum stress (UTS for Ultimate Tensile Stress) with the data given by Aubert&Duval. We see a fairly good coherence of our results with the results of the disk manufacturer.

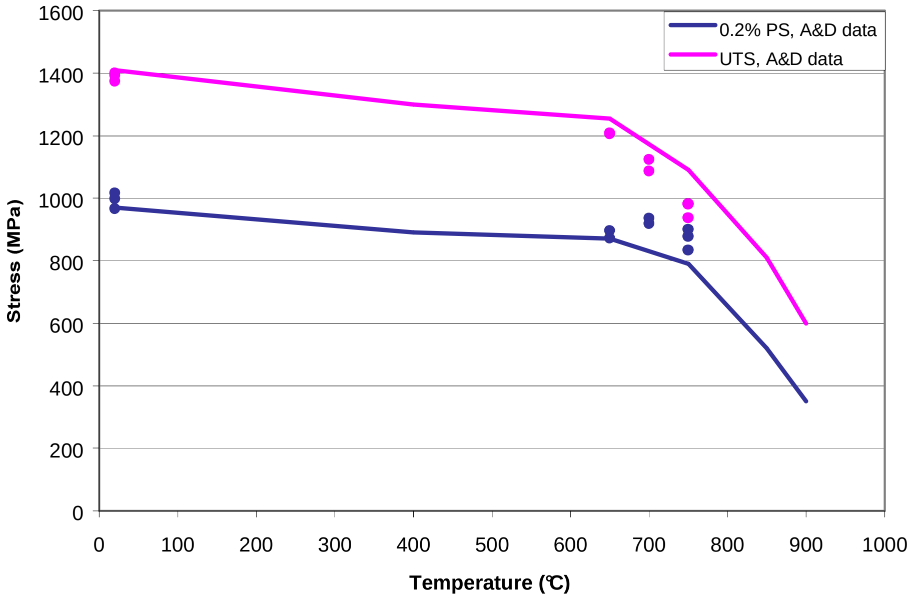


Figure 8 : HTR-M Tensile tests results on forged U720 compared with A&D data.

III.3 Impact toughness

Five specimens (KCV) have been machined in the disk and tested at 20°C on a Charpy pendulum.
The average measured value is KCV = 33 ± 5 J/cm^2. This value is relatively low compared to usual values measured on austenitic steels, but seems correct for a forged superalloy. Failure under this mechanical solicitation (impact) is brittle and inter-granular, as shown in Figure 9.

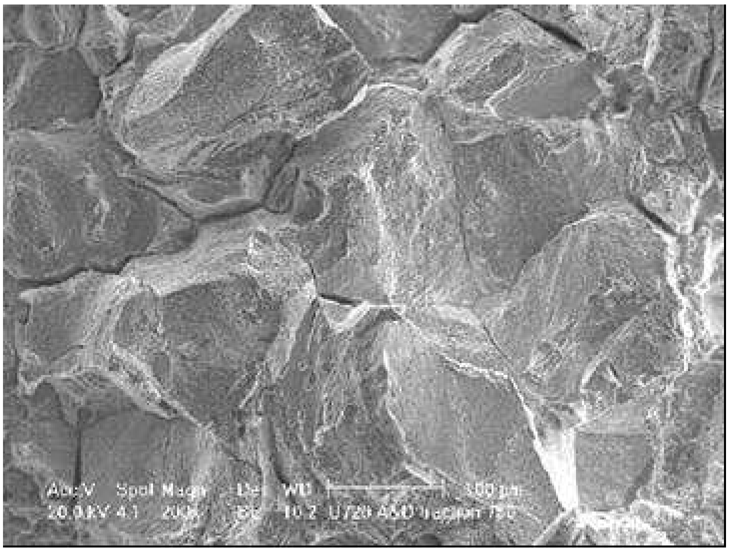


Figure 9 : SEM fracture surface of a KCV impact toughness specimen tested at room temperature.

III.4 Creep properties

Creep tests were conducted at 750°C with stresses ranging from 400 to 700 MPa. Creep specimens have the same geometry than tensile specimens. The creep stress is applied at a rate of 4 MPa/sec, and the creep deformation is set to zero just after loading. Details of the creep results can be found in Appendix 2.

Figure 10 gives the creep strain versus creep time recorded during the tests. Creep times to failure varied from a few hours to around 3000 hours for the lowest stress. We can see that the results are not much scattered, and no relation was found between the location of the specimen in the disk and the creep strength.

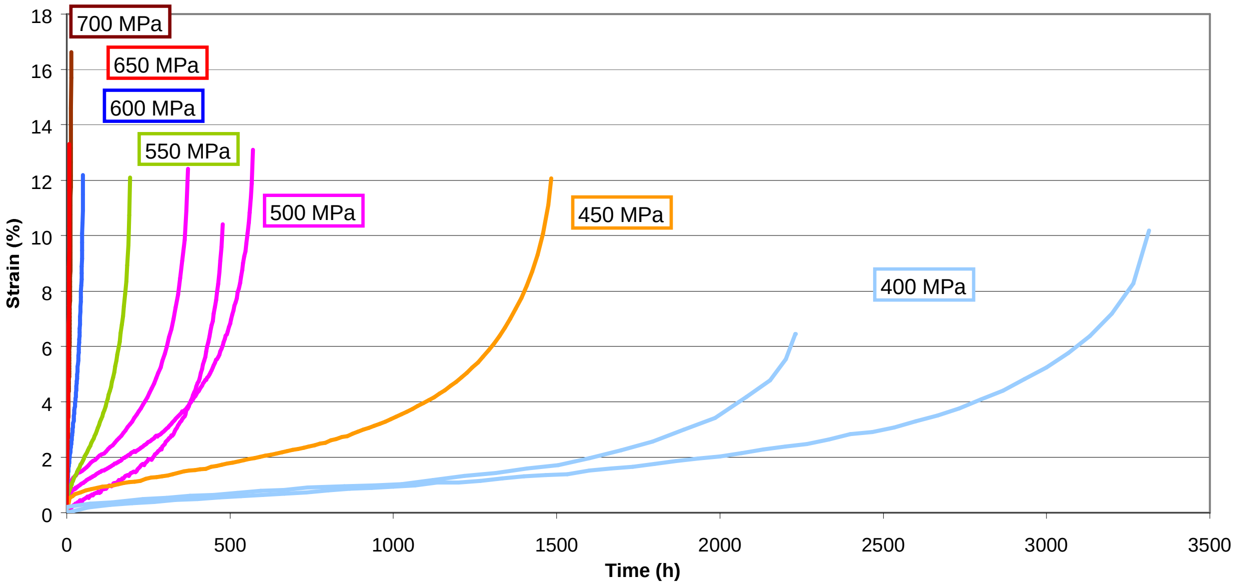


Figure 10: Creep strain versus time for creep tests at 750°C on forged U720.

Figure 11, which shows the creep strain-rate versus time, demonstrates that the creep behaviour is classical with 3 stages : primary creep where creep rate decreases , secondary creep with a steady creep rate and finally tertiary creep with a creep rate acceleration.
By plotting the minimum creep rate versus the creep stress in a log scale, we can measure the Norton coefficient n ($d\varepsilon/dt = A\sigma^n$). We find a value of n=14 over the stress range. This high value is typical of high strength superalloys when the creep deformation mechanism is based on gamma-prime/dislocations interactions.

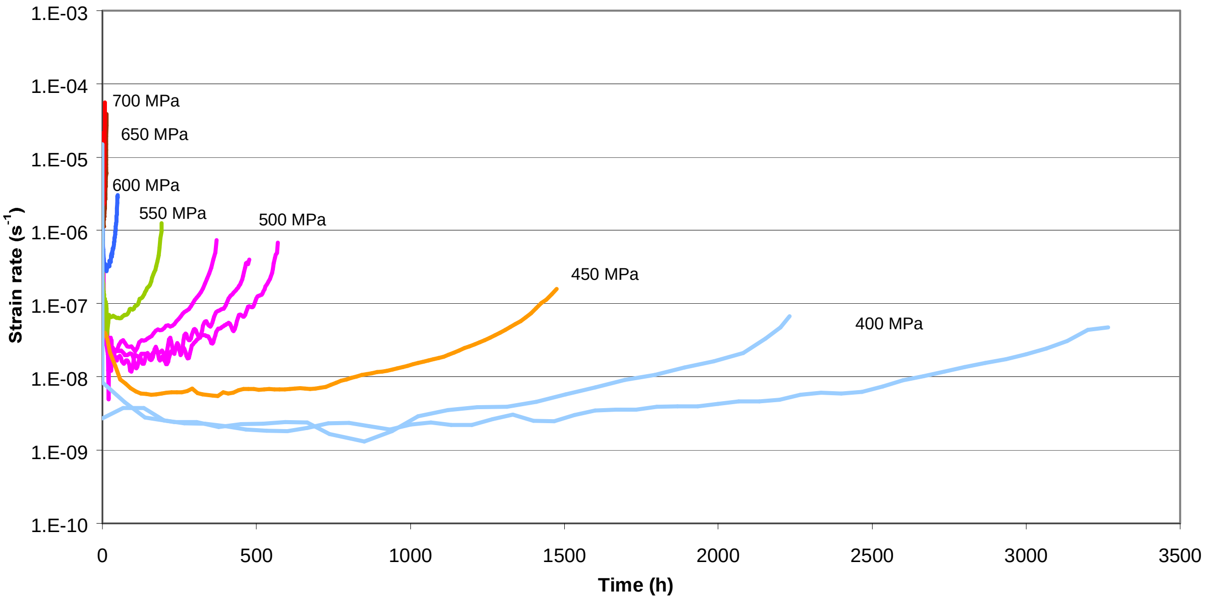


Figure 11: Creep strain-rate versus time for creep tests at 750°C on forged U720.

Based on the creep results, it is possible to use the "Larson Miller" relationship to plot the creep stress versus the paramater P based on temperature and time to failure. This approach allow to extrapolate our results to longer times or to other test temperatures. A plot of creep stress versus the Larson Miller parameter P is presented in Figure 12, together with data from A&D on coarse grain forged U 720 (U720CG) and fine grain forged U720 (U720FG).

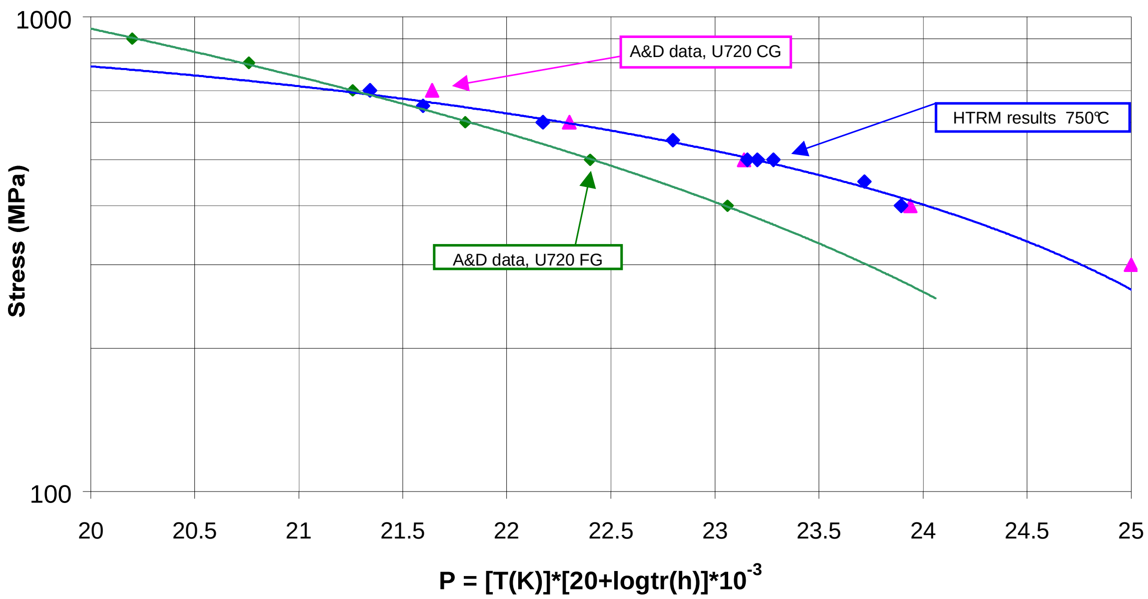


Figure 12 : Creep stress versus Larson Miller parameter for forged U720, compared with A&D data for fine grained (U720FG) or coarse grained (U720CG) alloy.

Figure 12 demonstrates that the effect of the grain size is more pronounced at low stresses (high temperature), where the intergranular mechanisms dominate. For creep at high stresses (low temperature), the U720 with a subsolvus heat treatment (U720FG) has the highest creep resistance.

Larson Miller plot for 1% creep strain.

The Russian team working on ISTC project linked to the GTMHR design has set materials miminum required properties for the turbine disks, in the case of cooled and non-cooled designs [ISTC2002]. These criteria consist of mimimum required stresses for a lifetime of 60000 hours with a creep strain of 0.2% and 1%. The Table 6 gives a summary of the ISTC criteria. These data can be plotted in a Larson Miller plot corresponding to the time for 1% creep strain, as shown in Figure 13. Figure 13 only represents the “cooled option” from ISTC data as the extrapolation needed to compare HTRM results to data for the non-cooled design is far too large (P=27.1 in this case).

	Max. T° in the disk	$\sigma_{0,2\%}^{60000h}$ (MPa)	$\sigma_{1\%}^{60000h}$ (MPa)
Cooled disks	650°C	270	295
	700°C	174	200
Non cooled disks	820°C	74	100

Table 6 **:** ISTC creep design criteria for GTMHR turbine disks.

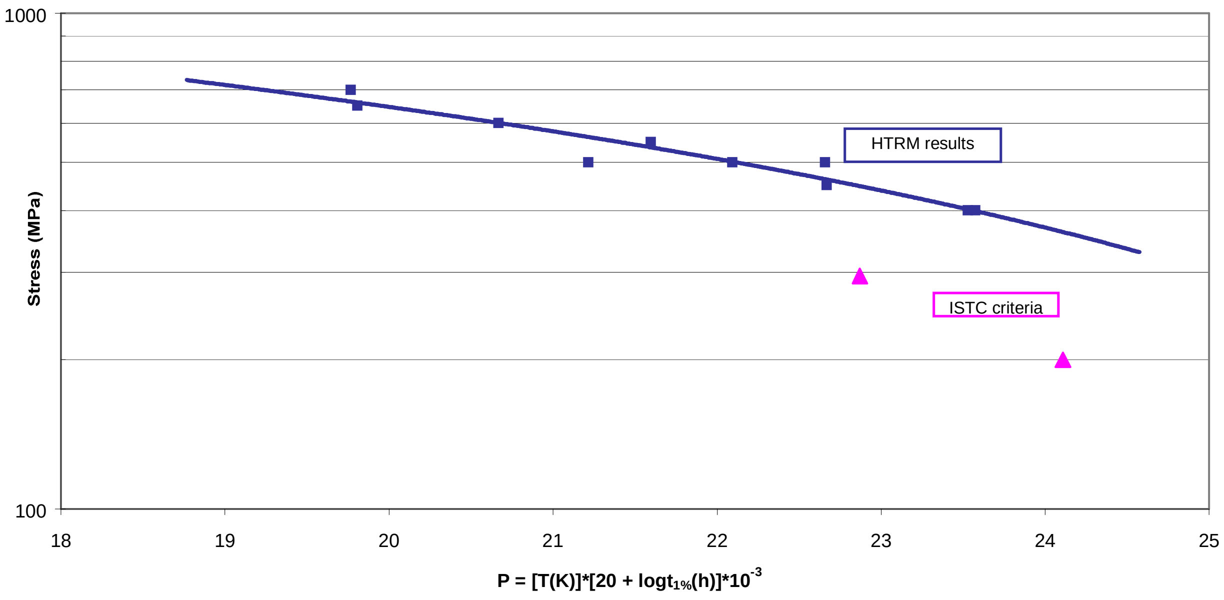


Figure 13: Larson Miller plot for 1% creep strain (HTR-M results) together with ISTC 1% creep design stresses for cooled disks.

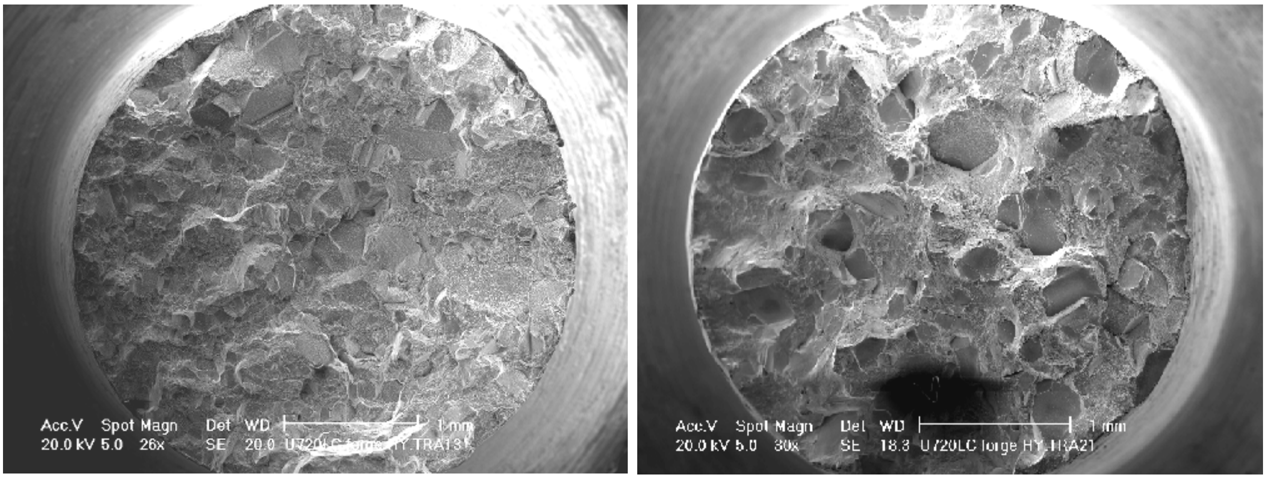
Figure 13 shows that the forged U720 material produced by A&D does fulfil the ISTC criteria for cooled disks. This emphasizes the need for cooling the turbine structures to ensure the safe operation for long term and to allow the use of industrial superalloys like U720.

<u>*Creep damage*</u>

The fracture surface of crept specimens at 750°C under 400 MPa and 700 MPa has been observed by SEM. Figure 14 shows that the failure mode (intergranular or intragranular) is difficult to determine. Therefore, longitudinal sections of broken specimens were polished and slightly etched to reveal the nature and location of secondary cracks.

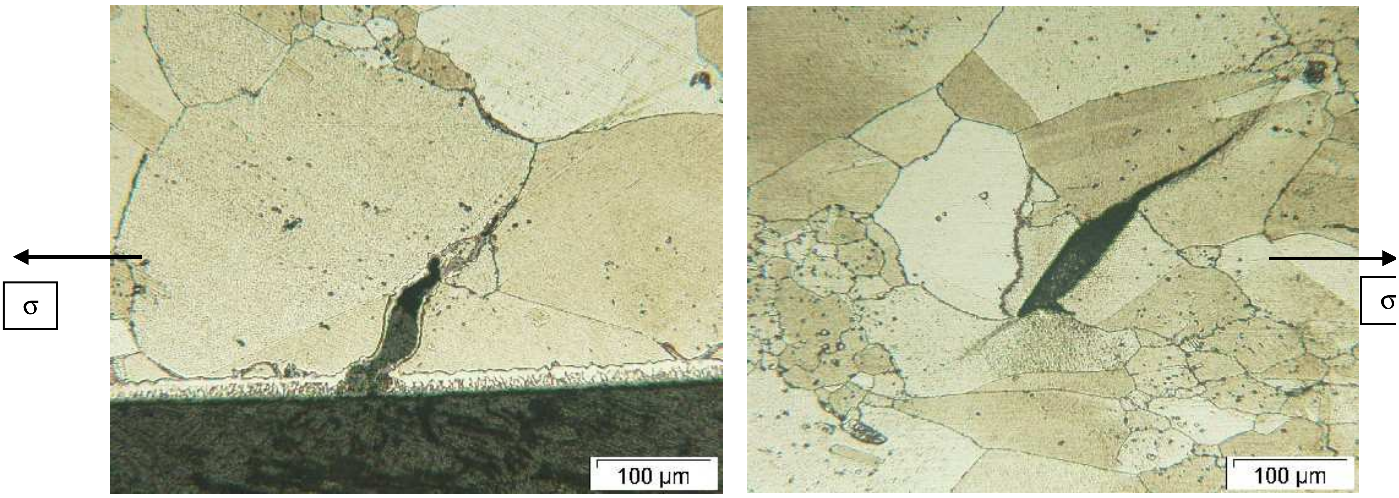
Figure 15 shows two optical micrographies of specimens tested at 400MPa and 700 MPa :

- At low stress (400 MPa), the damage (secondary cracks) is essentially located near the surface and at the grain boundaries.
- At high stress (700MPa), only a few secondary cracks are observed in the bulk of the specimen, and most of them are intragranular cracks.



(a) (b)

Figure 14 **:** SEM observations of creep fracture surfaces at 400 MPa (a) and 700 MPa (b).



(a) (b)

Figure 15 **:** Longitudinal cross-sections of specimens crept at 400 MPa (a) and 700 MPa (b).

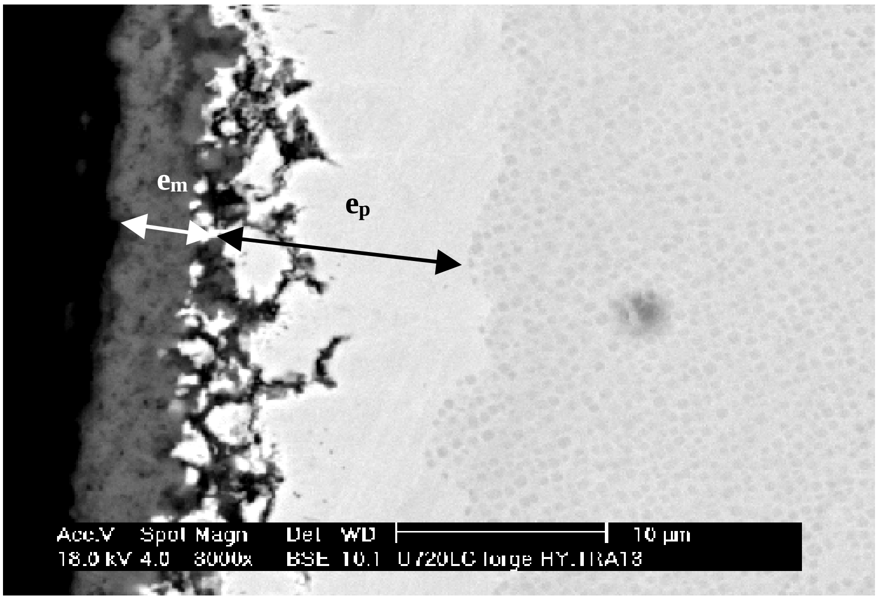


Figure 16 **:** SEM observations of a longitudinal cross section of crept specimen (400 MPa) with external (e_m) and internal (e_p) oxidation.

The damage observed for low creep stress at 750°C is related to a SAGBO mechanism (Stress Accelerated Grain Boundary Oxidation). This mechanism is due to a transport of oxygen atoms at the grain boundaries from the surface leading to a local oxidation. This local oxidation can lead to a dissolution of the γ' phase in the vicinity of the cracks, as observed on Figure 16.

These observations of creep damage prove that there is an "environment" effect for creep tests in air at 750°C at low stress. As the targeted application is in impure helium, the effect of environment on the creep properties will need to be analysed in more details within future studies.

III.5 Fatigue properties

The Low Cycle Fatigue (LCF) tests have been conducted on U720 forged material in the state '**I**" of the test matrix (see table 2). As it was the case for creep, the objectives of these tests are to establish a database that will be used to compare the results obtained on carburised or decarburised disk material.

The results of these tests are compared with bibliographic data obtained by CEA during a recent EC funded project on forged U720 [Burlet 03]. This comparison allows to determine whether the fatigue properties of U720 are modified or not by the specific forging and HT sequences applied to the HTR-M disk material.

The complete results of the five tests performed are summarized in appendix 2.

Figure 17 presents the hysteresis loops (stress versus strain) for the 4 LCF test conditions at Nr/2 (mid-life). Figure 18 shows the mimimum and maximum stresses versus the cycle numbers. Both minimum and maximum stresses are very stable within the fatigue life.

Figure 19 represents the fatigue strength of HTRM disk material in comparison to the bibliographic data. It can be seen that HTRM material exhibits a slightly lower fatigue strength. This could be attributed to the grain size heterogeneity and the presence of few very large grains.

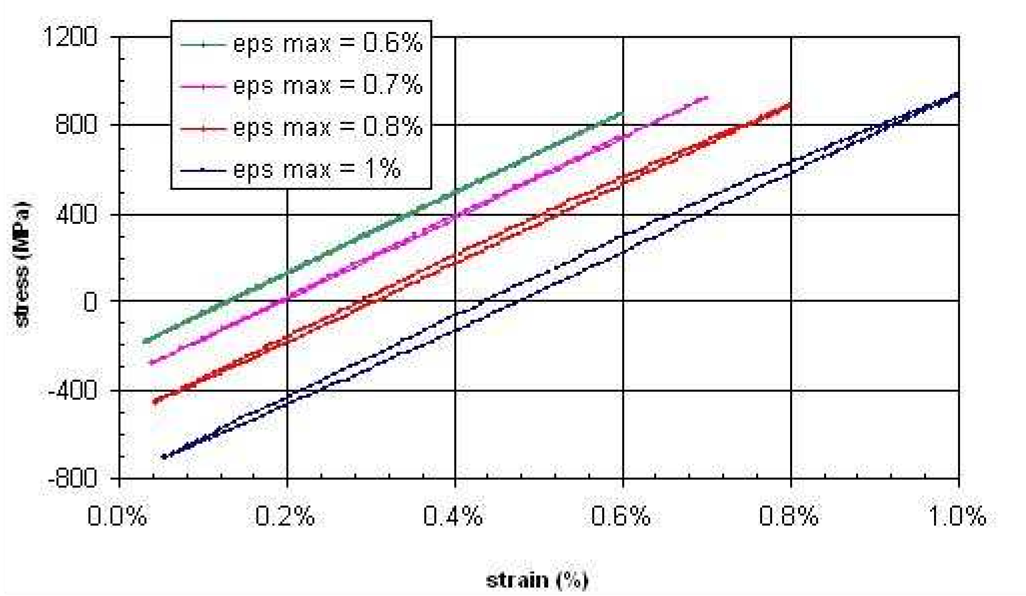


Figure 17 : Stress-Strain curves of U720 LC tested in LCF at 650°C (plotted at cycle $N_r/2$).

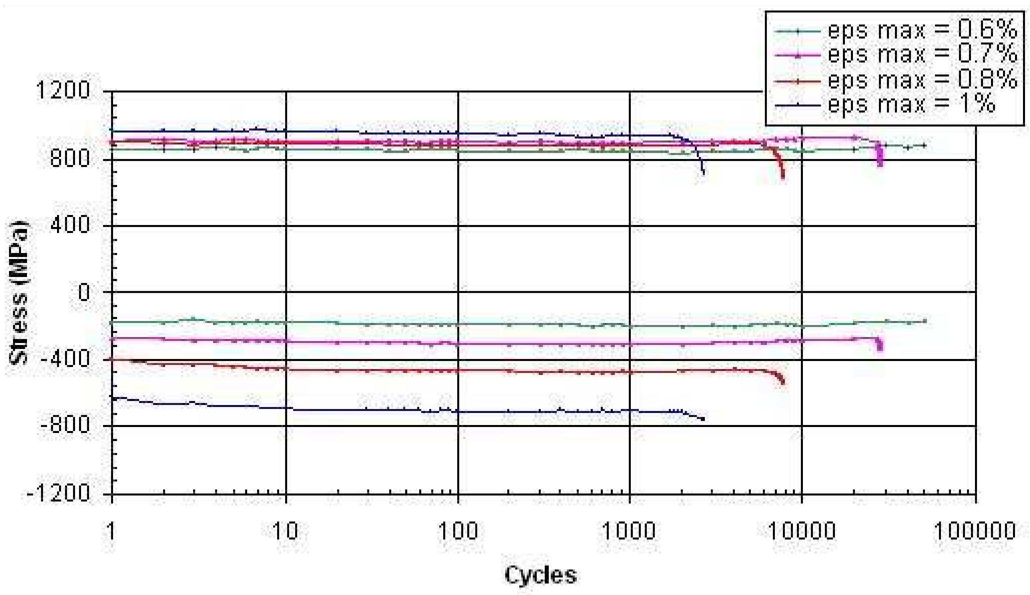


Figure 18 : Stress versus cycle number for U720 LC tested in LCF at 650°C.

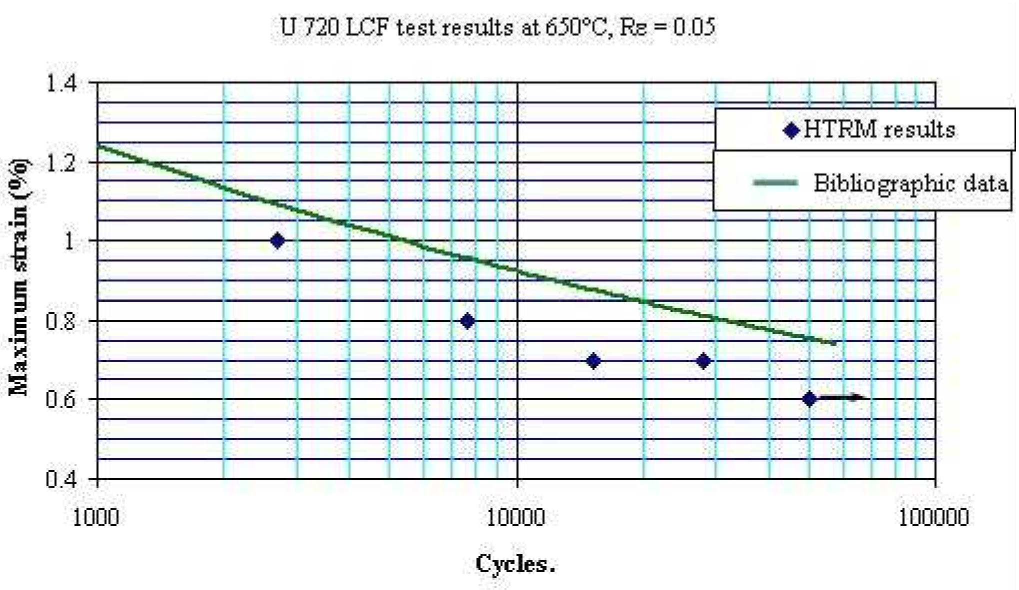


Figure 19 : Comparison of the LCF strength for HTRM U720 forged material with bibliographic data found in [Burlet03].

An analysis of LCF damage was performed by examination of longitudinal cuts of specimen at the end of the tests. After polishing and etching, Figure 20 shows that the damage is transgranular, with cracks starting from the surface.

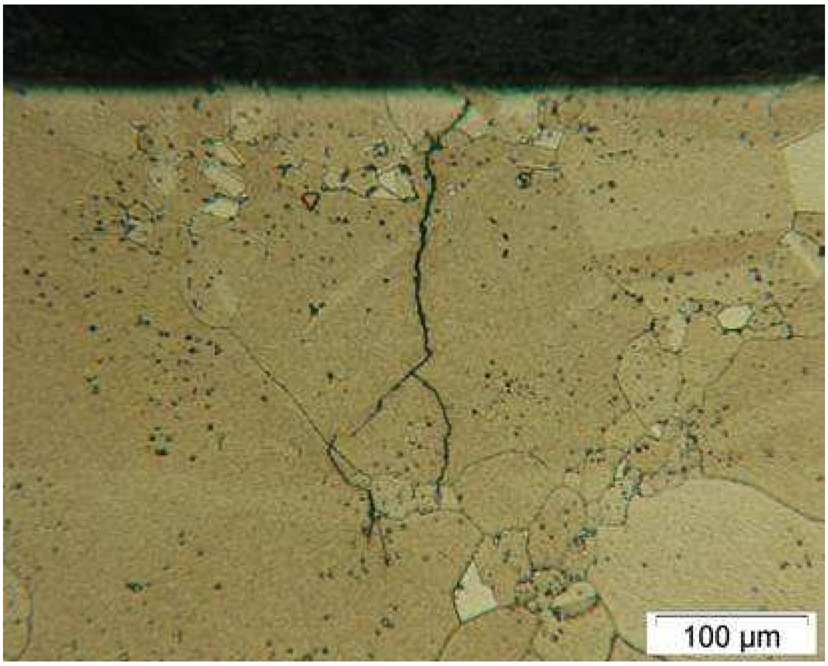


Figure 20 : SEM observation of a longitudinal cut of U720 LCF specimen tested with ε_{max} = 0.7%.

IV. Experimental results on blade materials.

IV.1 Metallography

The structure of CM 247 LC DS is observed by optical microscopy for sections parallel to the grains (001 direction) and perpendicular to the grains. A piece of the test bar labelled AB0746-5 (ϕ 16 mm, l = 150 mm) has been cut for metallographic examination.
Figure 21 shows a general view of the grains. The grain average width is 150 μm. Figure 22 shows a view of the structure parallel to the grains at higher magnification. We observe some misorientations and the presence of carbides and areas with primary eutectic γ' phase.
Figure 23 represents a view of the structure with a normal orientation with respect to the grains. Residues of casting micro segregation and dendrite structure are still visible, and a few microporosities are also revealed

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

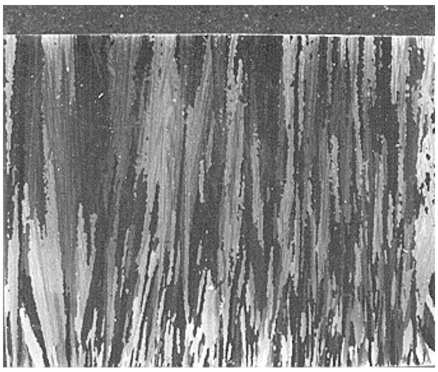


Figure 21 : General view of CM 247 test bar microstructure (specimen width=16 mm)

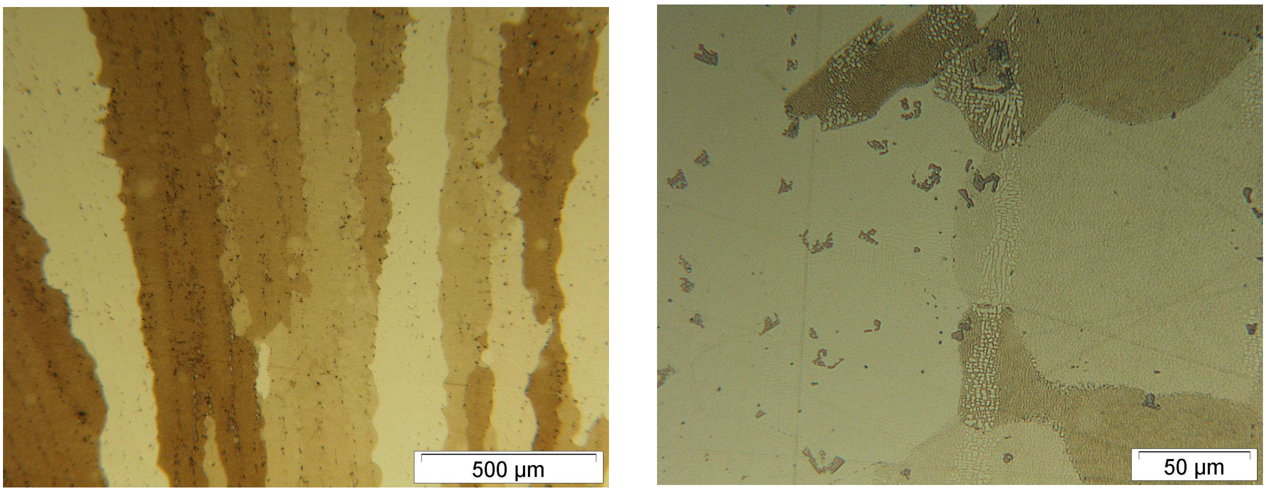


Figure 22 : Microstructure of CM 247, orientation parallel to the grains, at low and high magnifications.

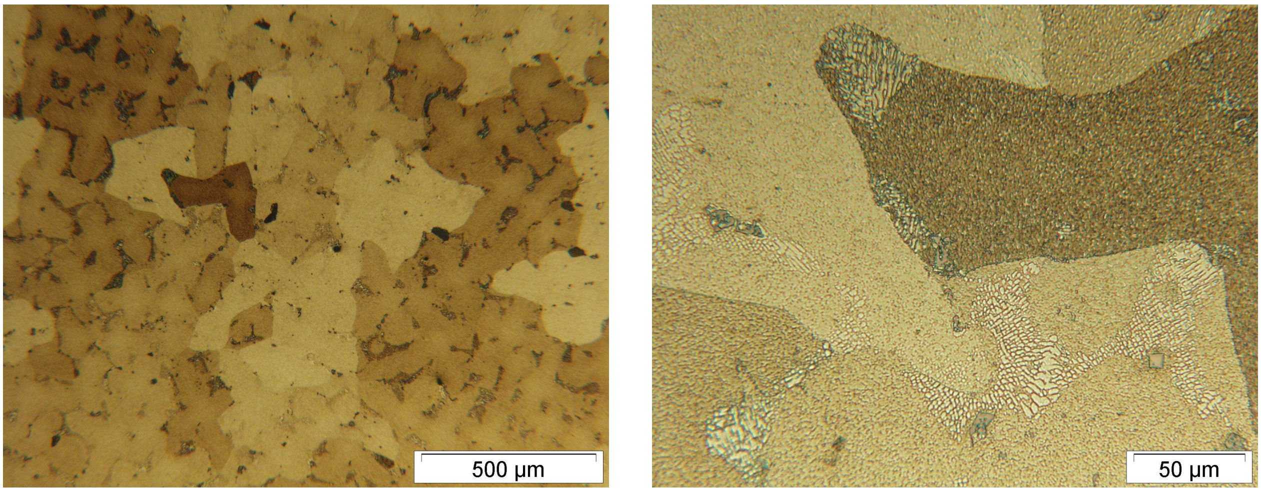


Figure 23 : Microstructure of CM 247, transverse orientation, at low and high magnifications.

The microstructure of IN 792 DS for sections parallel to the grains (001) and perpendicular to the grains are presented in Figure 24 and Figure 25. No microporosities were observed, but the chemical segregation observed in the transverse direction seem to be more pronounced than for CM 247.

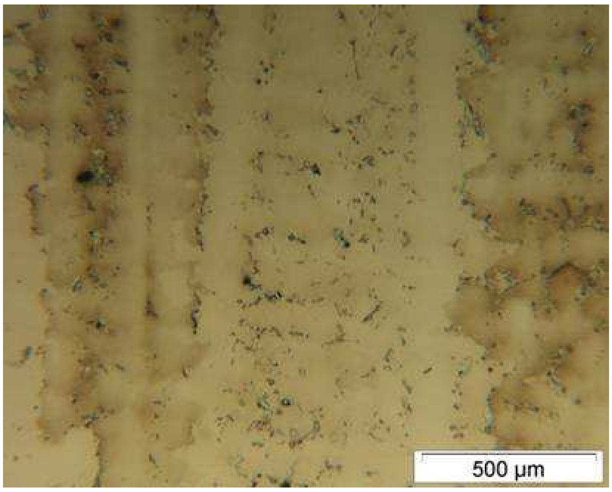


Figure 24 : Microstructure of IN 792, orientation parallel to the grains

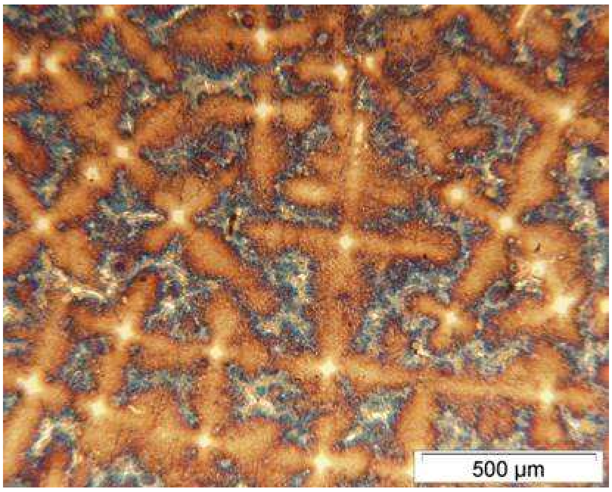


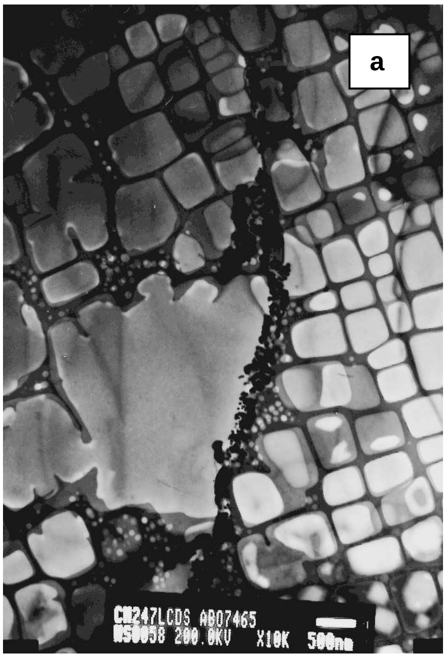
Figure 25 : Microstructure of IN 792, transverse orientation.

IV.2 TEM

TEM observations of the microstructure have been only performed for CM 247.

TEM examination of the microstructure has been made on a piece of test bar n° AB0746-5 (same as for metallography). Thin foils are cut in a plane normal to the (001) growth direction.

Figure 26 (a) and Figure 26 (b) shows a general view of the microstructure. 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. Low angle grain boundaries are decorated with small precipitates (Figure 27(a)) 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 (Figure 27(b)). These precipitates were identified as carbides enriched in Hf and Ta.



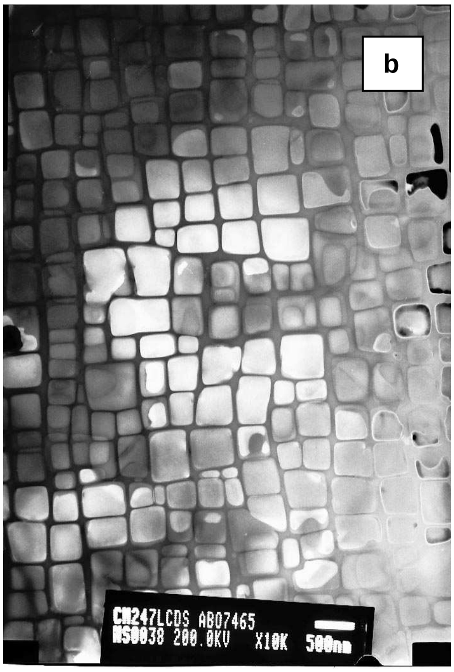


Figure 26 : General TEM view of CM 247 LC DS structure.

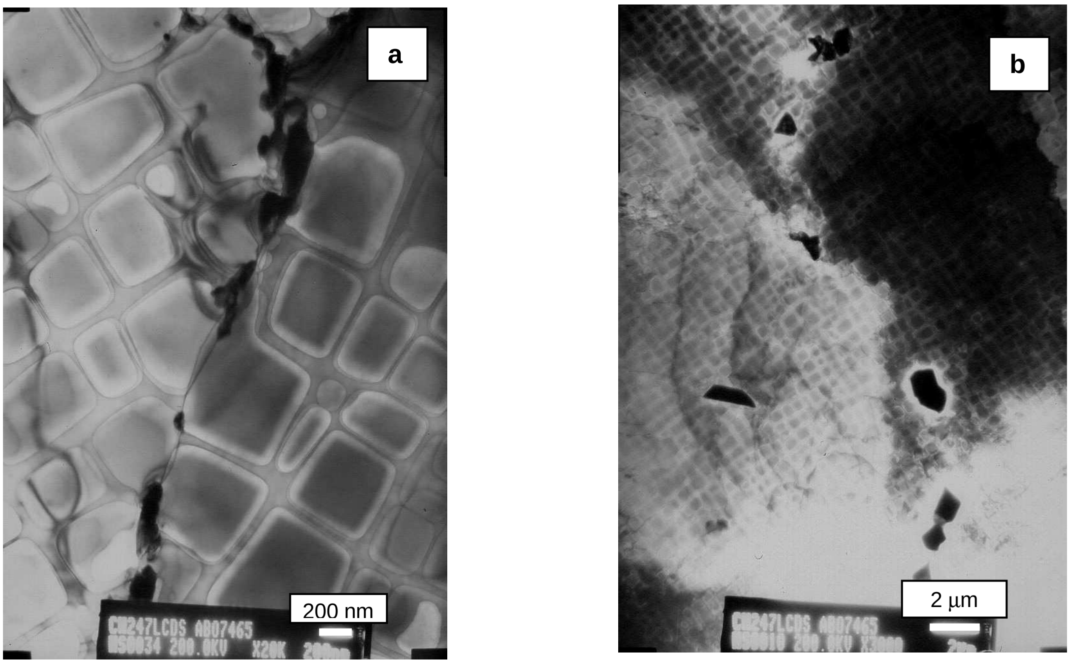


Figure 27 : TEM images of precipitates at low angle grain boundaries (a) and large carbides (b).

IV.3 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 2, together with the tensile specimen geometry. These results will serve as a basis for comparison with the tests in the metallurgical states II, III and I_{mod}.

Figure 28 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 29 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 30).

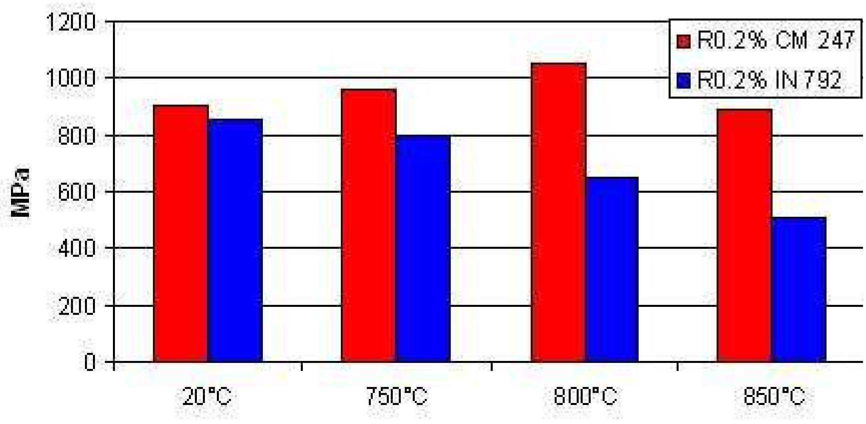


Figure 28 : Comparison of 0.2% Yield Stress for CM 247 and IN 792 from 20°C to 850°C.

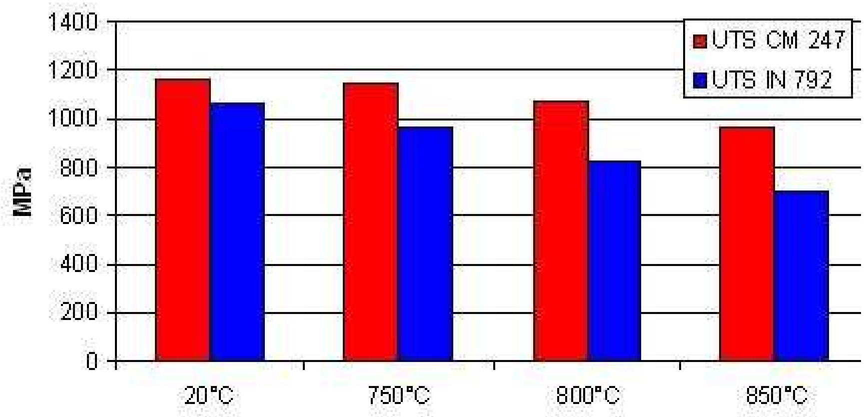


Figure 29 : Comparison of Ultimate Tensile Stress for CM 247 and IN 792 from 20°C to 850°C.

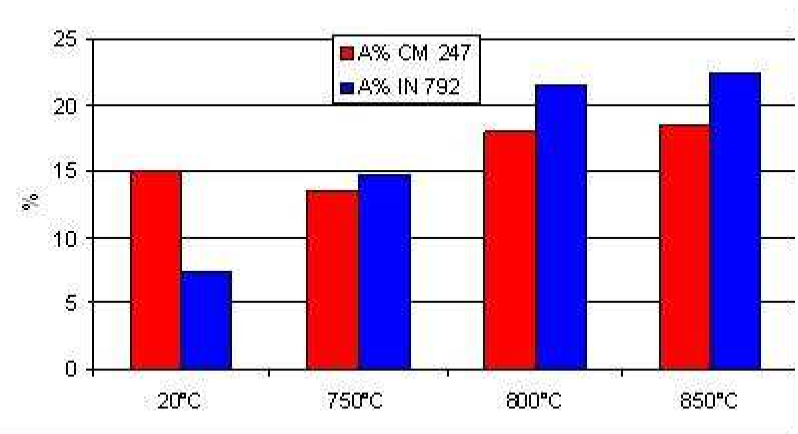


Figure 30 : Comparison of elongation at rupture for CM 247 and IN 792 from 20°C to 850°C.

IV.4 Creep tests

As for other mechanical properties, these results will serve as a basis for comparison with the tests in the metallurgical states II, III and I_{mod}. The comparison with these states will be done within HTR-M1 project.

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 matrix (~1000h, ~3000h and ~9000h). The details of tests conditions and results are given in appendix 2.

The stresses selected to obtain 1000h, 3000h and ~9000 hr 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 31 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 the lowest stress, the test is still in progress and the result will be analysed within HTR-M1 project.

Figure 32 shows the creep curves for IN 792. Like for CM 247, it seem that the times to failure will match the required values. The test at lowest stress will also be continued within HTR-M1.

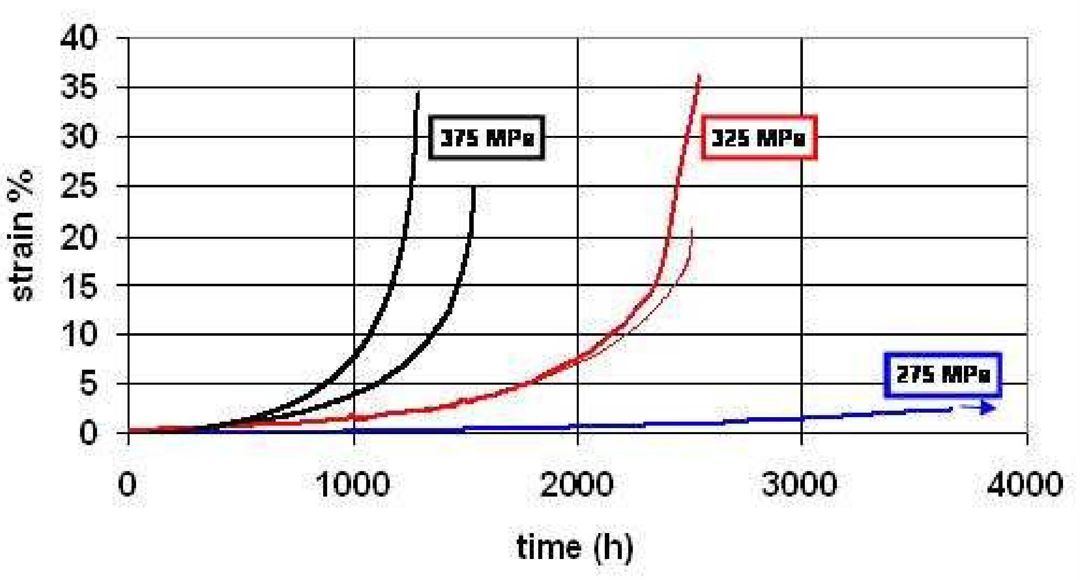


Figure 31 : Creep curves obtained at 850°C for CM 247 grade.

Figure 33 compares the measured creep rupture times with results from the bibliography ([ASM 1997]. It shows that our results are consistent with bibliography. As for tensile properties, it can also be noticed that the creep strength of CM 247 is superior to that of IN 792.

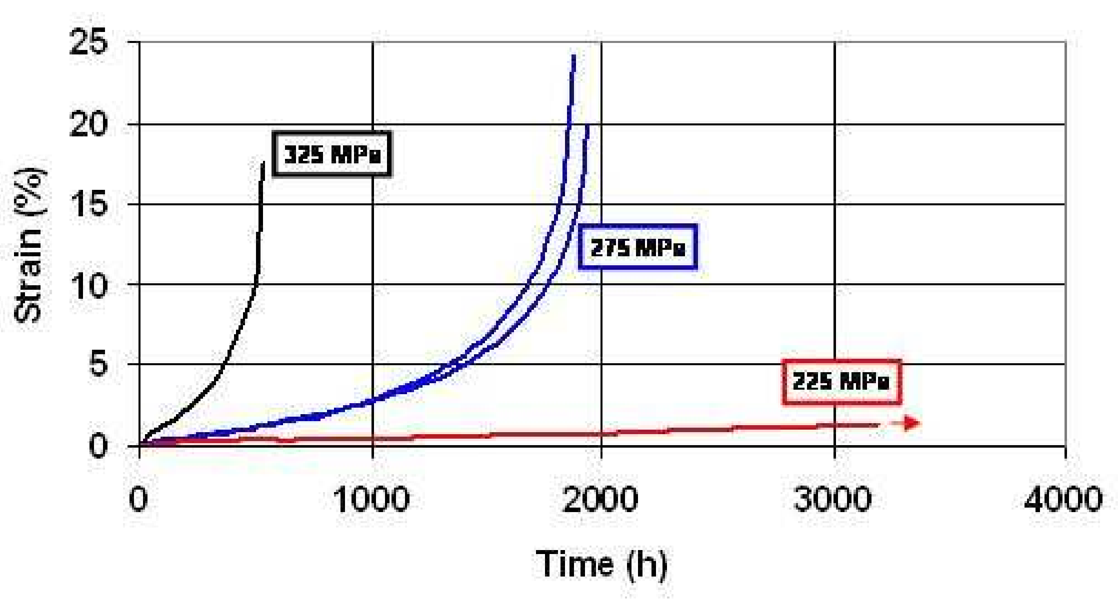


Figure 32 : Creep curves obtained at 850°C for IN 792 grade.

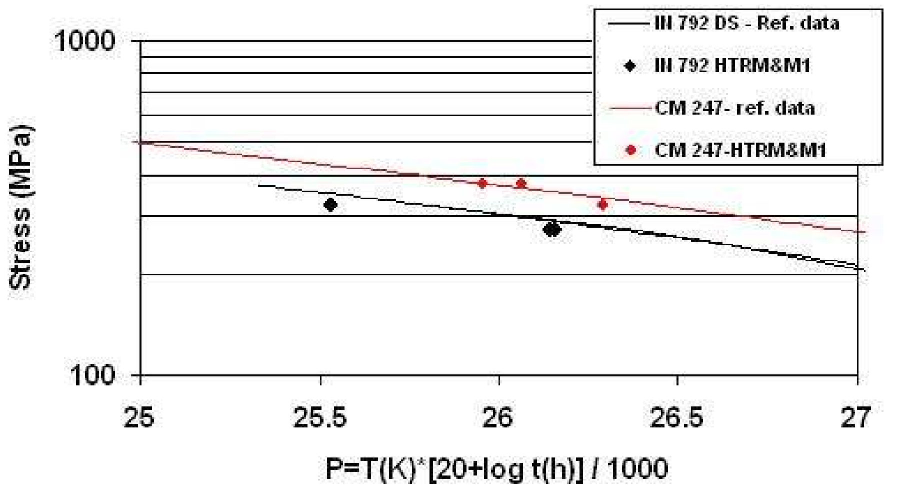


Figure 33 : Comparison of CM 247 and IN 792 creep rupture properties, with the classical Larson-Miller representation.

V. Experimental results on the carburization facility at JRC

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. 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.

When the construction of the equipment was completed in the beginning of 2004 it did not function properly as an automatic safety shutdown was triggered at random intervals. The reason for the shutdown remained unknown as the control system gave an erroneous error code, which did not help to locate the problem. Finally problem was solved and the system was in running order, and 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, see Figure 34.

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.

The depth of de-carburisation is not yet known, because at the time of writing this report only the first batch of carburisation is being completed and after this the second carburisation treatment will take place, and only after these the two de-carburisation treatments will begin. All the treatments and associated mechanical testing will be completed within the extension period of the HTR-M1 project.

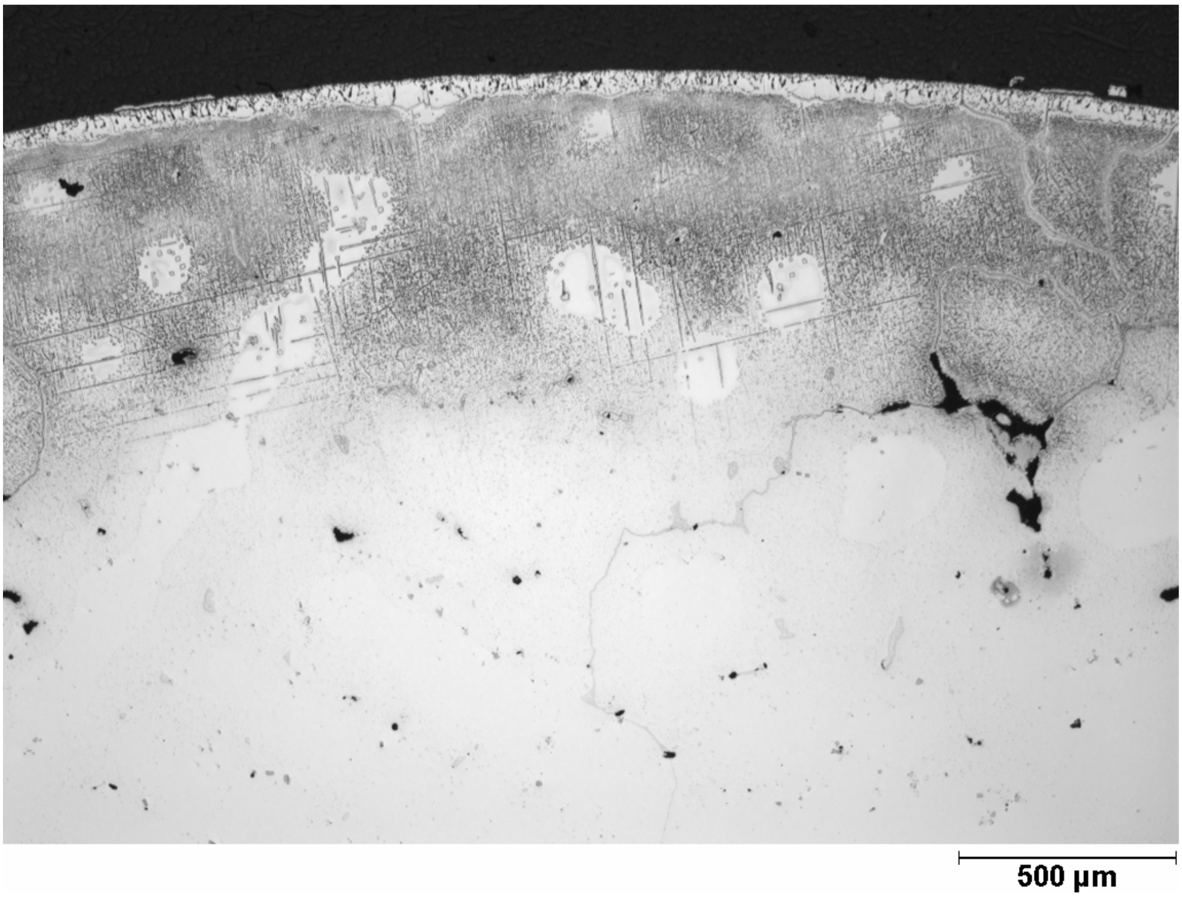


Figure 34 . Carburisation and internal oxidation of Al in a 0.9mm surface layer in a dummy test specimen of IN617 after the carburisation treatment in $Ar+2\%CH_4$ at 950^oC, 1000h.

VI. Conclusion

This report gives the final results of mechanical tests obtained on disk and blade materials evaluated in the HTR-M Project.

Forged Udimet 720 was selected for the disk. Despite of the heterogeneous grain size, the tensile, creep and fatigue properties measured are correct, and the creep mechanical criteria for the design of cooled disks is fulfilled.

For the blade grades (CM 247 and IN 792 with directional solidification), it was shown that the microstructure and properties are coherent with usual properties of this type of materials.
For tensile and creep tests, CM 247 shows better properties (higher strength or lifetime) than IN 792.
Before the end of the HTR-M1 project, additional work will be done on blade materials with the final results of medium term creep tests and an evaluation of the effect of a 10000 hours ageing at 850°C.

Tests on carburised and decarburised blade material will also be achieved within HTR-M1 and compared to the data presented in this report, in order to obtain a first assessment of the effect of the specific HTR environment.

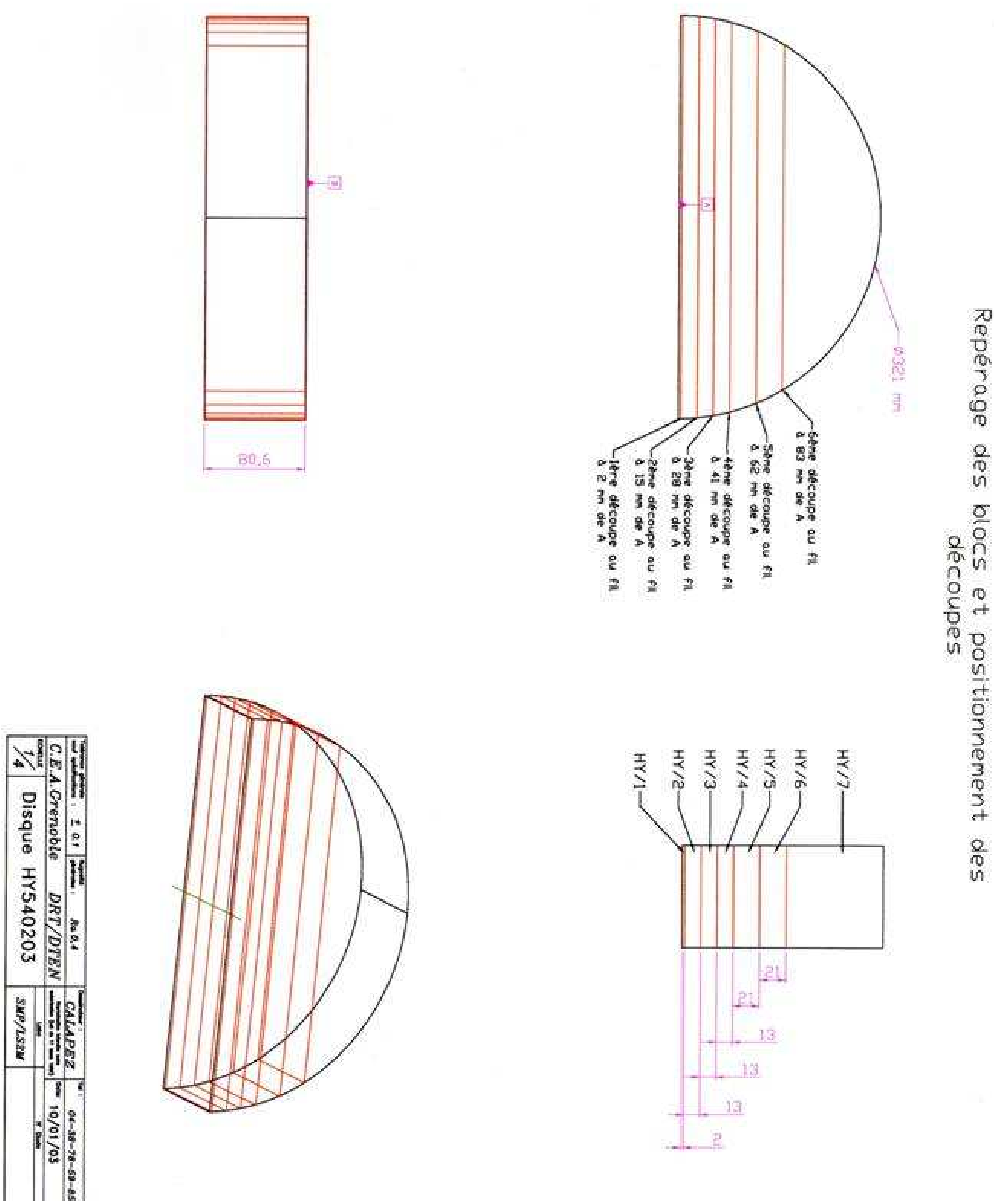
VII. References

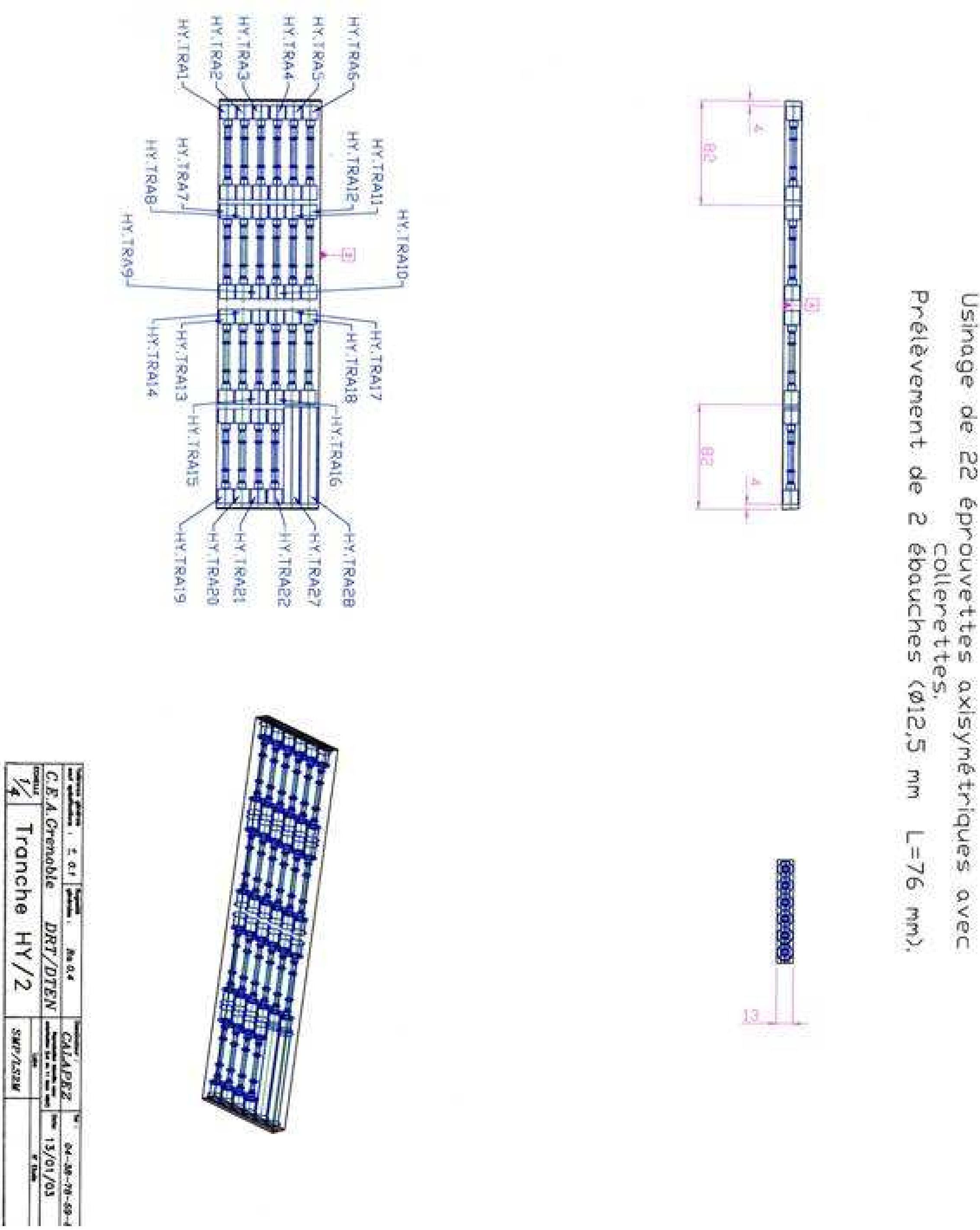
[ASM 1997] : "ASM specialty handbook, Heat resistant materials", Ed. By J.R. Davis, ASM International, 1997.

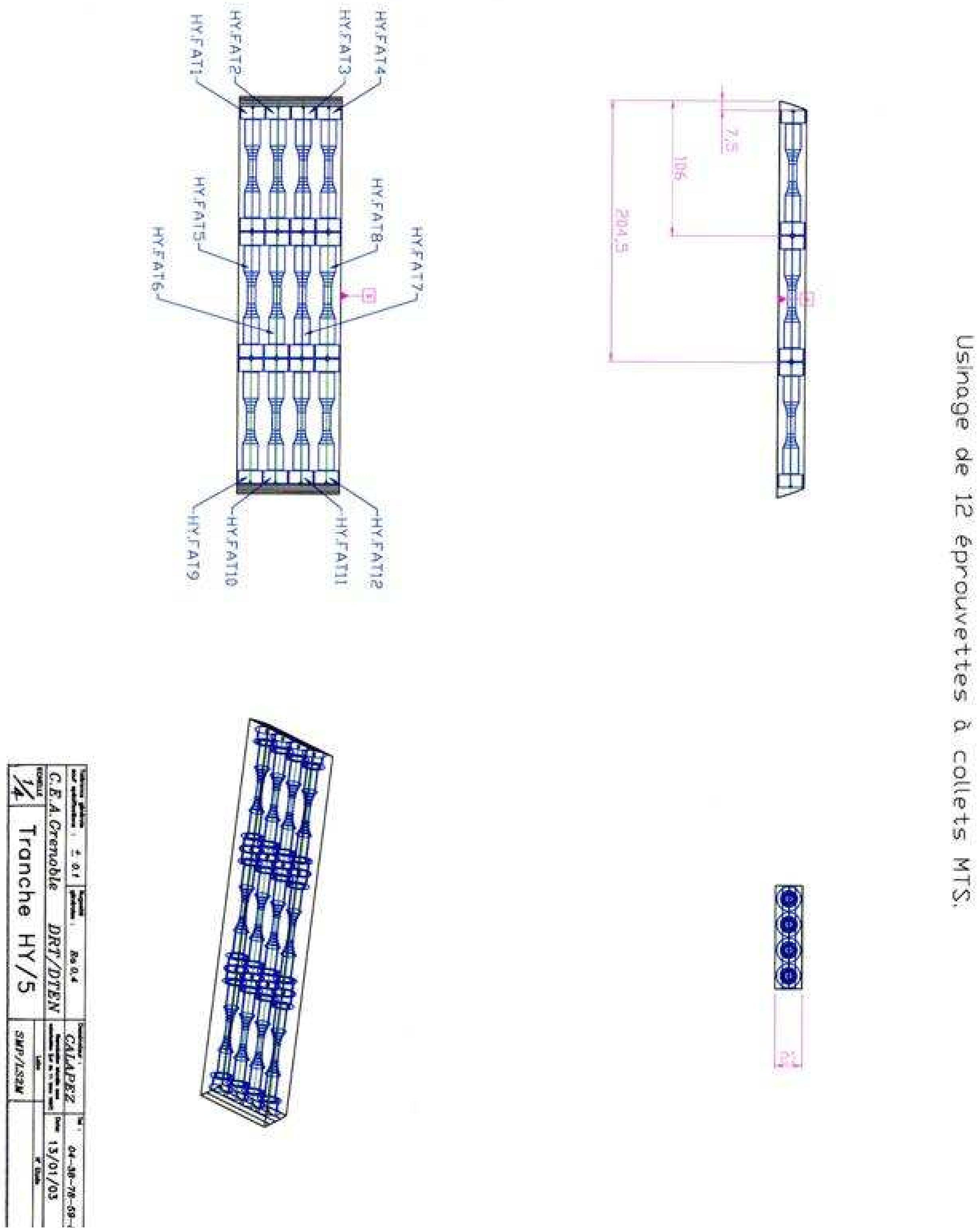
[ISTC2002] : "Testing and investigations of materials for turbocompressor high temperature components", A. Romantsov, ISTC Report 1313-2001, 2002.

[Burlet 2003] : "Microstructural and mechanical performance assessement of diffusion bonded bimetallic model discs", H Burlet et al., Proceedings of the 6th international Charles Parsons conference, ed. By A Strang et. al, Maney, 2003.

VIII. Appendix 1 : location of specimens in the forged U720 disk




Usinage de 22 éprouvettes axisymétriques avec collerettes.
Prélèvement de 2 ébauches (ø12,5 mm L=76 mm).
HY.TRA1
HY.TRA2
HY.TRA3
HY.TRA4
HY.TRA5
HY.TRA6
HY.TRA7
HY.TRA8
HY.TRA9
HY.TRA10
HY.TRA11
HY.TRA12
HY.TRA13
HY.TRA14
HY.TRA15
HY.TRA16
HY.TRA17
HY.TRA18
HY.TRA19
HY.TRA20
HY.TRA21
HY.TRA22
HY.TRA27
HY.TRA28
82
4
13
C.E.A.Grenoble
DRT/DTEN
CALAPEZ
13/01/03
1/4
Tranche HY/2



Usinage de 12 éprouvettes à collets MTS.
HY.FAT1
HY.FAT2
HY.FAT3
HY.FAT4
HY.FAT5
HY.FAT6
HY.FAT7
HY.FAT8
HY.FAT9
HY.FAT10
HY.FAT11
HY.FAT12
7,5
106
204,5
C.E.A.Grenoble
DRT/DTEN
CALAPEZ
13/01/03
1/4
Tranche HY/5
SMP/LS2M

IX. Appendix 2 : mechanical tests results

Tensile tests

Tensile tests are done with an electro-mechanical 100 kN machine, with a specimen geometry shown in figure A2-1. 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 in table A2-1 for disk material and A2-2 and A2-3 for blade material.

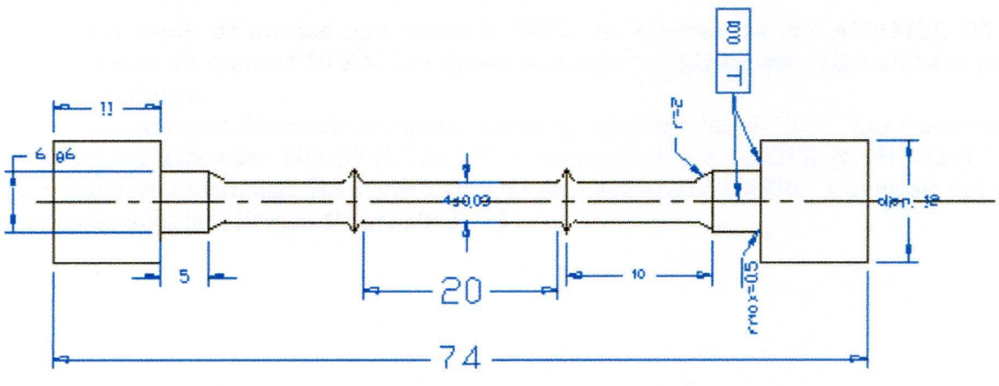


Figure A2-1 : Tensile specimen geometry.

Specimen ref.	File n°	T (°C)	Strain rate (s^{-1})	σ_y (Mpa)	0.2% PS (MPa)	UTS (MPa)	A%	Z%
HY.TRA4	2003-082	20	5.10^{-4}	964	1017	1374	17.5	17,1
HY.TRA6	2003-083	20	5.10^{-4}	914	998	1393	17	17,5
HY.TRA23	2003-084	20	5.10^{-4}	888	966	1400	19.3	18,2
HY.TRA1	2003-085	650	5.10^{-4}	791	872	1206	29.6	37,8
HY.TRA3	2003-086	650	5.10^{-4}	777	896	1208	24.2	32,7
HY.TRA2	2003-087	700	5.10^{-4}	802	919	1087	27.9	41,1
HY.TRA12	2003-088	700	5.10^{-4}	810	936	1123	23.5	38,3
HY.TRA5	2003-089	750	5.10^{-4}	768	877	981	30.8	38,7
HY.TRA7	2003-090	750	5.10^{-4}	808	900	982	21.2	32
HY.TRA24	2003-091	750	5.10^{-4}	724	834	937	22.4	35,8

Tableau A2-1 : Tensile tests results on forged U720 disk material

Specimen ref.	File n°	T (°C)	Strain rate (s^{-1})	σ_y *(Mpa)*	0.2% PS (MPa)	UTS (MPa)	A%	Z%
AB0747-1	2003-254	20	5.10^{-4}	838	901	1165	15	18
AB0747-2	2003-255	750	5.10^{-4}	939	960	1150	13.4	17
AB0747-3	2003-256	800	5.10^{-4}	979	1054	1066	18	22
AB0747-4	2003-257	850	5.10^{-4}	778	889	960	18.5	24

Tableau A2-2 : Tensile tests results on CM 247 LC DS blade material.

Specimen ref.	File n°	T (°C)	Strain rate (s^{-1})	σ_y *(Mpa)*	0.2% PS (MPa)	UTS (MPa)	A%	Z%
DA9875-1	2004-045	20	5.10^{-4}	760	860	1065	7.4	11
DA9875-2	2004-042	750	5.10^{-4}	630	800	961	14.7	25
DA9875-3	2004-043	800	5.10^{-4}	510	655	822	21.6	30
DA9875-4	2004-044	850	5.10^{-4}	400	5140	703	22.4	32

Tableau A2-3 : 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 weigths with a 10:1 lever ratio. Creep specimens have the same geometry as tensile specimens. Table A2-4 gives the results of creep tests on U720 forged alloy. Table A2-5 and A2-6 give the results for CM 247 LC DS and IN 792 LC DS grades.

Specimen n°	File n°	T (°C)	σ (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					Dε/dt mini (s^{-1})	Remarks
								0.2	0.5	1	2	5		
HY.TRA13	-	750	400	3.3	2280	-	18.4	0.03	257	1001	1625	2177	$1.8.10^{-9}$	No recording
HY.TRA10	03-098	750	400	3.3	3395	11.5	13.3	70	400	1111	1974	2965	$1.8.10^{-9}$	
HY.TRA14	03-079	750	450	3.75	1532	11.2	12.2	4	6	144	584	1221	$5.5.10^{-9}$	
HY.TRA20	03-075	750	450	3.75	-	-	-	19	97	280	-	-	6.10^{-9}	Stopped at 1% creep, for TEM
HY.TRA11	03-046	750	500	4.2	431	13.1	18.6	0.5	0.7	5.5	95	279	$2.5.10^{-8}$	
HY.TRA8	03-055	750	500	4.2	571	12.8	24.4	0.2	1.4	39	179	438	2.10^{-8}	
HY.TRA19	03-065	750	500	4.2	482	9.6	18.5	11	59	141	263	410	$1.4.10^{-8}$	
HY.TRA9	03-045	750	550	4.6	193	12.2	15.2	0.03	1.2	13	54	143	$6.5.10^{-8}$	
HY.TRA15	03-059	750	600	3.3	47	13.7	21.1	0.3	0.6	2	9	32	$2.8.10^{-7}$	
HY.TRA16	03-068	750	650	3.6	13	17.2	35.6	0.02	0.05	0.2	1.5	7	$1.4.10^{-6}$	
HY.TRA21	03-083	750	700	3.9	7	13.8	30.4	0.03	0.06	0.2	1.1	4.5	$2.2.10^{-6}$	
HY.TRA17	03-077	750	700	3.8	13	13.7	26.4	0.05	0.1	0.2	1.4	7	$1.4.10^{-6}$	Wrong load applied

Table A2-4 : Creep tests results on U720 forged material.

Specimen n°	File n°	T (°C)	σ (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					Dε/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	
AB0747-10	05-004	850	275	4.6	>3668	>2.6	-	650	1350	2400	3300	-	1.10-9	interrupted

Table A2-5 : Creep tests results on CM 247 LC DS alloy.

Specimen n°	File n°	T (°C)	σ (MPa)	Loading rate (MPa/s)	Tr (h)	A %	Z (%)	Time to x % creep (hours)					Dε/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	-	850	225											In progress

Table A2-6 : Creep tests results on IN 792 DS alloy.

Fatigue tests

Tests are conducted in air at 650°C on a 100 kN hydraulic MTS machine. Tests are "strain controlled" with a triangular strain signal. Strain is measured by a high precision extensometer placed on the specimen. The extensometer is cooled by water to ensure a good thermal stability. The specimen geometry is given in figure A2-2.

Tests are conducted with a standard procedure for LCF tests on turbine disks superalloys. The strain rate is maintained at 1.10^{-3} s^{-1} and the test frequency is therefore adapted with the maximum strain. The Rε ratio ($\varepsilon_{min}/\varepsilon_{max}$) is equal to 0.05. Tests are stopped when a load drop of 15% is detected, corresponding to the presence of fatigue cracks.

The LCF results on U720 are presented in table A2-7

Name	ε_{max}	ε_{min}	N_r	σ_{max} (N=1/4) (Mpa)	$N_r/2$				
					σ_{max} (MPa)	σ_{min} (MPa)	$\Delta\varepsilon_t$ (%)	$\Delta\varepsilon_p$ (%)	$\Delta\varepsilon_e$ (%)
HYFAT-8	0.7	0.035	27944	907	924	-283	0.665	0.0077	0.6573
HYFAT-7	0.8	0.04	7640	906	892	-454	0.76	0.021	0.739
HYFAT-9	1	0.05	2691	966	942	-705	0.95	0.041	0.909
HYFAT-11	0.6	0.03	>50000	856	877	-164.5	0.57	0.0023	0.568
HYFAT-12	0.7	0.035	15211	892	895	-322	0.665	0.0071	0.658

Table A2-7 : LCF results on U720 at 650°C.

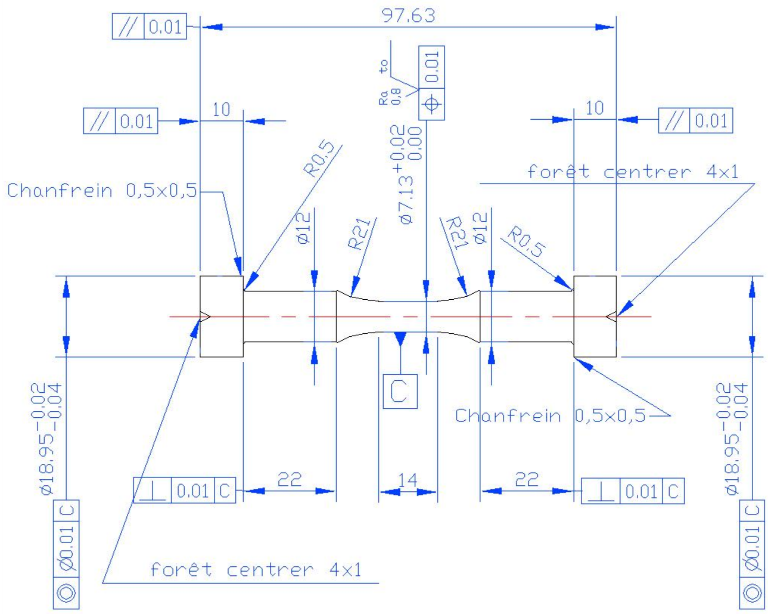


Figure A2-2 : LCF specimen geometry

X. Appendix 3 : U720 properties from A&D data

PER 72 T (APPLICATION HAUTE TEMPERATURE)

1/2 PRODUIT : Grain fin avec bonne inspectabilité US (0.6 mm)

FORGEAGE PIECES : Dia. 500 mm - PDS ~ 150 KG
Matriçage "sub solvus"

TRAITEMENT THERMIQUE STANDARD : 1160°C/4 H /AIR + 1080°C/4 H/HUILE
\+ 845°C/24 H/AIR + 760°C/16 H/AIR
Grain : MOYEN 0 à 3 ASTM + QQ 5/6 ASTM
Détectabilité US : TFP 0.8 mm

PROPRIETES MECANIQUES (TYPIQUES) :

		Rm (N/mm2)	Rp 0.2 (N/mm2)	A 5d (%)	Z (%)
Traction :	20°C	1410	970	18	18
	400°C	1300	890	15	15
	650°C	1255	870	20	25
	750°C	1090	790	25	30
	850°C	810	520	25	35
	900°C	600	350	20	30

Fluage : 800°C/400 MPa - 170 h
850°C/300 MPa - 75 h
900°C/200 MPa - 65 h

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