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# Effect of Environment on the material of GT-MHR Vessel

by

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# Note Technique

**TITRE :** EFFECT OF ENVIRONMENT ON THE MATERIAL OF GT-MHR VESSEL

**AUTEUR(S) :** M.T. CABRILLAT

**RESUME :** The development of advanced HTR concepts requires material data information and an understanding of material behaviour under reactor operating conditions.
In the framework of HTR-M, WP1 is devoted to the study of the RPV (Reactor Pressure Vessel) material. Improvements are necessary in the areas of design analysis, structural integrity analysis and material properties data. Complementary information is also needed on manufacturing of products, influence of environment (neutron-irradiation fluence, temperature, helium purity) and welding. The aim of this WP is to confirm the choice of a material for the RPV of different HTR concepts.
Several tasks have been identified. The present document is a contribution from CEA to the task 1.7 : environmental effects. Two different effects are studied : susceptibility to corrosion in HTR atmosphere and irradiation effect in HTR conditions.
For these 2 points, some results are available at CEA within different research programs on materials which are close to materials candidate for HTR RPV. These results can be used for the future studies. A presentation of the main conclusions is made in this document.
Concerning corrosion in He environment, it is pointed out that atmosphere composition has to be characterized carefully. The knowledge on ferritic and martensitic steels allows to state that 21/4Cr and 9-12Cr steels seem to be appropriate for RPV in HTR operating conditions (T<450°C). Some complementary points have to be investigated.
Concerning irradiation effect, martensitic steels with 9%Cr appear to present a good behaviour with respect to swelling, ductile-to-brittle transition temperature value and activation. Nevertheless it is necessary to get data on the specific cast in representative operating conditions.

**MOTS CLES :** GT-MHR – VESSEL MATERIAL – ENVIRONMENT – CORROSION - IRRADIATION

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# Contents

# 1. INTRODUCTION

The development of advanced HTR concepts requires material data information and an understanding of material behaviour under reactor operating conditions. Specific studies have been undertaken to identify suitable materials for the different components of the reactor and mainly for the RPV (Reactor Pressure Vessel), the internal structures and the turbine.

In the framework of HTR-M, WP1 is devoted to the study of the RPV material. Validations and improvements are necessary in the areas of design analysis, structural integrity analysis and material properties data. Complementary information is also needed on manufacturing of products, influence of environment (neutron-irradiation fluence, temperature, helium purity) and welding.

The purpose of WP1 is to investigate all these points. The final objective is to confirm the choice of a material for the RPV of different HTR concepts, accounting for normal and accidental conditions but also environmental conditions, and to compile the design properties, including the presence of weld joints.

To reach this objective, different tasks have been defined [1]. Different points are investigated such as: identification of sensitive zones to analyse and damage mechanisms to consider, evaluation of the validity of the existing design rules, influence of welds, influence of environment, and review of the existing data base. These different studies allow to identify the missing data and the developments which are necessary to perform the design of RPV.

The present document is a contribution from CEA to the task 1.7 : environmental effects.
Two different effects are studied : susceptibility to corrosion in HTR atmosphere for the different materials candidate for RPV and irradiation effect in HTR conditions.

# 2. CORROSION IN HTR ATMOSPHERE

Considering the corrosion in HTR environment implies to study the interactions between a material and the surrounding atmosphere. Thus, it is of primary importance to characterize the HTR atmosphere.

## 2.1 HTR ATMOSPHERE

The composition of the HTR primary coolant varies according to the design of the reactor, its parameters of operation and the characteristics of the purification system. The past experiment on HTR indicates that the system of purification and the sealing of the circuits ensure a particularly low level of contamination of helium. The pollutants, brought in by the leakages and the degassing from structures and fuel, react with the components of the reactor working at high temperature. The nature and the relative proportions in impurities strongly depend on the materials that constitute the core.

In the case of a conventional HTR, the gas chemistry is dominated by the impurities $H_2$, $H_2O$, $CO$, $CO_2$, $N_2$, $O_2$, $CH_4$. These species stem from different effects: air ingresses during opening of the circuits, in-leakage, degassing, reaction with the metallic structures, reaction with the hot core graphite, purification efficiency...
The table 1 indicates the atmosphere composition estimated for previous HTR and gives indications on the upper level imposed for the GT-MHR.

| Impurities (μbar) | Previous HTR | GT-MHR<br>Upper limit |
|---|---|---|
| $H_2$ | 20-500 | <350 (5 ppm) * |
| $CO$ (+ $CO_2$) | 10-400 | <420 (6 ppm) |
| $CH_4$ | 3-50 | <140 (2 ppm) |
| $N_2$ | 3-15 | <140 (2 ppm) |
| $H_2O$ | 1-30 | <140 (2 ppm) |

* Levels are given as part per millions (ppm) in GT-MHR design report

Table 1: range of composition of HTR He atmosphere

Partial pressures of $H_2$, $CO/(CO_2)$, $CH_4$, $N_2$ and $H_2O$ are in a range of a few microbar to a few hundreds of microbar for a helium pressure of several tens of bar. The partial pressures of the impurities are very low. Thus the probability of gas/gas reaction is low and the gas phase is not at thermodynamic equilibrium. The atmosphere is characterized by a low potential of oxidation and the existence of species that contain carbon (setting a certain carbon activity $a_c$).

Regarding the material environment, it will be of prime importance to specify the coolant composition: nature and relative concentration of impurities and possibly the influence of gaseous fission products.

## 2.2 SPECIFIC CONDITIONS FOR THE RPV

The conditions of loadings of the components are important to study the interaction between the coolant and the structure. In addition to the coolant composition, different factors must be taken into account such as temperature, gas flow characteristics (renewal or not of the corrosive agents), stress level (possibility of stress corrosion). The past experience leads to conclude that results obtained in a specific atmosphere are valid only in similar conditions. Extrapolations outside this domain are doubtful and must be supported by detailed investigations. In particular, one must check that the main corrosion mechanism remains the same.

For the GT-MHR the operating conditions for the RPV are:

- in nominal conditions the temperature is about 400 – 460°C,
- in incidental or accidental situations the temperature will reach 490 to 570°C, but the duration of these situations as well as the accidental atmospheres are not precisely known for the moment,
- the nominal pressure in the gas is 71 bar,
- the flow rate along the vessel is very low, because, due to the design, the helium which is in contact with the vessel is static. This characteristic is important and would have to be considered to estimate the real atmosphere in the vicinity of the RPV,
- the mechanical stresses on the RPV are about 120 – 130 MPa,
- the vessel must be designed for 60 years.

## 2.3 CORROSION PROCESSES

Several reactions between metals and He impurities are possible, depending on the gas relative partial pressures, the material characteristics, and the temperature.
Let us take Cr as an example. The different reactions can be summarized as follows:

- oxydation

  $3 H_2O + 2 Cr = Cr_2O_3 + 3 H_2$ (1)

  steam oxidises metals

- carburisation

  $CH_4 = [C] + 2 H_2$ (2)

  [C] states for C dissolved in the metal.
  If the solubility limit is locally overreached, [C] can react with the metal leading to metallic carbide precipitation ($Cr_xC_y$ in our case).

- carburisation/oxidation

  $3 CO + 2 Cr = Cr_2O_3 + [C]$ (3)

  metallic carbide precipitation

- decarburisation

  $H_2O + [C] = CO + H_2$ (4)

  Decarburisation is the consequence of oxidation of carbon in solution by steam. It can lead to dissolution of carbides.

If reaction (1) predominates, it can promote the formation of a protective oxide film (passive behaviour). If reaction (1) is associated with (2), (3) or (4), the oxide can't provide a good protection.

## 2.4 EXPERIENCE IN HTR ATMOSPHERE

HTR atmosphere is characterized by a low potential of oxidation and a positive carbon activity. In such situations, there will be a competition between oxidation and carburisation,

depending on operating conditions. Consequences on mechanical properties can be noticeable.
Extensive carburisation will lead to embrittlement of the material with a decrease of ductility and toughness.
Oxidation may allow the formation of an oxide film that is a protection against corrosion. Internal oxidation or incomplete oxide layer coverage is not protective.
Mainly active at high temperature, decarburisation induces a loss of strength of the material.

Different experiments have been performed on steels in HTR atmosphere. The main results are presented below.

- Corrosion depends on Cr content in the alloy. In [2], results of tests performed at T=550°C, for a time t=5000h and a pressure P=2 bar on different steels are reported. The atmosphere was polluted by:
  $H_2$ : 1500 µbar, CO : 450 µbar, $CH_4$ : 50 µbar, $H_2O$ : 50 µbar.
  A complete decarburisation was observed for Carbon steels, whereas an internal oxidation up to 6 µm occured for 21/4Cr-1Mo steel. A thin oxide film (<1 µm) formed on the surface of a steel with 11%Cr.

- Corrosion depends on the atmosphere properties. In “oxidizing” atmosphere, mild steels do not corrode. But they decarburise at T> 425°C in “balanced” He atmosphere. For these steels, the corrosion rate increases when the temperature increases. Their use is restricted to temperatures lower than 400°C.

- Corrosion depends on temperature. Under the very same atmosphere, for instance, the 21/4Cr-1Mo steel experiences opposite corrosion effects [3].
  For T ≤ 480°C there is a slight increase of C content, for T ≈ 540°C there is no change in C content, and for T ≥ 593°C decarburisation proceeds.
  Experiments have shown that in the conditions where decarburisation was obtained, a decrease of creep properties was also observed [4].

## 2.5 CONCLUSIONS

About RPV material durability in HTR atmosphere, we can give the following conclusions.
HTR He can be considered as a corrosive medium. Its effect depends on several factors: temperature, atmosphere (oxidising, carburising), alloy composition (mainly Cr content).
Based on middle duration tests (~one year), the knowledge on ferritic and martensitic steels allows to state that 21/4Cr and 9-12Cr steels seem to be appropriate for RPV material with the following upper temperature limits to avoid corrosion:
Mild steels : ~ 400°C,
21/4Cr steels: ~ 550°C,
9-12Cr steels : ~ 650°C.
These upper values should be validated by long-term experiments under realistic He atmosphere.
Some other environmental effects have to be checked:

- accidental conditions: characterisation of the atmosphere (in case of air ingress for instance), influence of the temperature reached, of the time spent at higher temperatures,
- interaction between corrosion and fatigue,
- possibility of stress corrosion,
- evaluation of crack growth in He,
- behaviour of welding in He atmosphere…

# 3. IRRADIATION EFFECTS

Influence of irradiation on vessel material has to be studied in the operating conditions planned for this component.
Many results concerning irradiation effect are available for 9-12%Cr steels, for other applications (Fusion Thermonuclear Reactors, Fast Breeder Reactors, Accelerator Driven Systems…), but generally for conditions which are not fully representative of HTR. Nevertheless, these results are interesting and can be very useful to define the future studies.

## 3.1 IRRADIATION CONDITIONS FOR HTR VESSELS

For HTR RPV, due to the presence of graphite structures, it is thought that the cumulated fluence during the whole life will be limited. For the GT-MHR vessel, an estimation of the expected cumulated fluence for 60 years is 8.E18 n/cm$^2$ for neutrons with an energy E>0.1 Mev and 4.E18 n/cm$^2$ for E>0.5 Mev [5].
So the fluence is very low. It corresponds to about $8.10^{-3}$ dpa [6].
An important point to consider to evaluate the irradiation effect is the operating temperature. For the “hot vessel” conditions, the mean temperature is 400-440°C for nominal conditions, and it can reach 450-550°C in accidental conditions.

## 3.2 IRRADIATION EFFECTS

Due to the irradiation, the material microstructure is modified by complex mechanisms.
From a macroscopic view point, three phenomena are important and must be considered: swelling, irradiation-induced creep and modification of material properties: mainly loss of ductility, loss of strain hardening capability and reduction of fracture toughness.
Irradiation level also has an influence on the ductile-to-brittle transition temperature. This later increases with fluence level.
Creep and fatigue behaviour may be modified by irradiation.
Weld joints are sensitive to neutron irradiation.

All these points must be taken into account.

## 3.3 AVAILABLE RESULTS

Some results concerning irradiation effects are available within different R&D programs. At CEA, in France, 9-12%Cr steels have been studied in the years 1985-2000 for different applications, for different loading situations. The main results are summarized below.

These steels were studied in the following conditions:

- FBR applications for fuel tubes and fuel clads [7]. Tests were performed for a irradiation temperature range 380-550°C, and up to dose of about 100-150 dpa (Fast neutron spectra - PHENIX reactor).
- Fusion applications [8]. In this case, specific reduced activation steels were tested: 7-12Cr steels, with W, V and Ta (mixed neutron spectra).
- Future PWR applications [9]. In this program, conventional 9-12Cr and reduced activation steels were tested. Irradiation temperature: 325°C, dose: 1 to 9 dpa (mixed neutron spectra).
- ADS applications. Some studies are performed to evaluate the behaviour of the target window in 9Cr steel, for high He concentration (to simulate spallation products influence) and high neutron irradiation dose.

The different irradiation programs were conducted :

- at Phenix: 400–550°C, up to 155 dpa,
- at Petten: 250-450°C, 2 dpa,
- at Osiris: 325°C, 1-9 dpa.

In addition, some irradiations are running at BOR 60 and PSI.

Some interesting results obtained in these programs are listed below:

➔ swelling is very low for martensitic steels [10] (see figure 1). Even for dose around 160 dpa, the maximum deformation induced by irradiation is lower than 1%. So, for low dose, swelling will be negligible. These results were obtained for irradiation temperature 400-550°C in Phenix reactor.

➔ In the same irradiation conditions, i.e. irradiation temperature 380-550°C, and for irradiation dose varying between 68 and 110 dpa, the DBTT (Ductile to Brittle Temperature Transition) value is better for 9Cr martensitic steels [8] (see figure 2). We observe on this figure, that the best results are obtained for the steels with 9%Cr. It appears to be an optimum for the % Cr content. F17 is a 17%Cr steel, EM12 and HT9 are 12%Cr steel and EM10 and T91 are 9%Cr steels.
In the temperature range 380-440°C, which is the interesting temperature range for the HTR vessel, EM10 and T91 have a very satisfactory behaviour, with respect to the neutron irradiation dose.

➔ In reference [9], results in PWR conditions are presented for dose varying between 1 and 3.4 dpa. These results can be exploited for HTR RPV, even if the conditions will be slightly different: the coolant is different, the irradiation temperature was lower (325°C), and the dose higher in PWR studies.
In the framework of this research program, 4 austenitic steels, 7 conventional martensitic steels, 4 reduced activation martensitic steels and 2 ODS ferritic-martensitic steels were studied, in different metallurgical conditions.

- It was found that there is a strong influence of the initial metallurgical conditions on post-irradiation behaviour of conventional martensitic steels (see figure 3). For EM10 steels at 3.5 dpa, a loss of ductility is observed, but the best behavior in term of ductility is obtained for the tempered metallurgical conditions.
- Tensile properties at 325°C were evaluated before and after irradiation at 3.5 dpa: yield stress, ultimate tensile stress, rupture striction (reduction of area). Hence again, EM10 (which corresponds to 9%Cr) has the best behaviour (see figure 4). In particular, the reduction of area after irradiation remains very good. During these studies, it was pointed out that it is very important to have a measurement of the area reduction in complement of the tensile properties measurements in order to obtain good informations on the post-irradiation plasticity of the materials.
- Post-irradiation dose rate measurements have been performed on the samples irradiated at 0.9 dpa, after different cooling times. Martensitic steels display a lower level of residual activity than austenitic steels. So they are also attractive for that point of view, especially their “low activation” variants.

## 3.4 CONCLUSIONS ON IRRADIATION EFFECTS

Studies on martensitic 9-12Cr steels have been performed in other research activities fields. They allow to obtain some results on irradiation effects.
Martensitic steels present the following characteristics:

- low swelling
- low shift of DBTT for martensitic steels with Cr content ≤ 9%
- strong effect of initial metallurgical conditions on post-irradiation behaviour
- low activation when compared to austenitic steels

However, complementary studies are necessary to characterize more precisely the candidate steel for the RPV. Of particular interest is the behaviour of the welds which could differ from base metal.

It is also necessary to investigate the irradiation influence in the “real” HTR conditions: temperature, time, influence of neutronic spectra…

# 4. CONCLUSION

In order to select the reference material for the HTR RPV, different criteria have to be considered, such as: workability, weldability, creep resistance, mechanical behaviour, influence of environment…
Influence of environment covers several aspects. The main ones are: the susceptibility to corrosion in HTR atmosphere and the influence of irradiation in operating conditions.

For these 2 points, some results are available at CEA within different research programs. A presentation of the main conclusions is made in this document.

Concerning corrosion in He environment, we pointed out that it is of prime importance to identify the atmosphere composition and the characteristics of the coolant. We also indicated that the knowledge on ferritic and martensitic steels allows to state that 21/4Cr and 9-12Cr steels seem to be appropriate for RPV material with the following upper temperature limits to avoid corrosion: 21/4Cr steels: ~ 550°C, 9-12Cr steels : ~ 650°C.
Finally we identified some points that must be investigated to complete these studies: long-term corrosion behaviour, behaviour in accidental conditions, interaction between corrosion and fatigue, possibility of stress corrosion, evaluation of crack growth in He, behaviour of welding in He atmosphere…

Concerning irradiation effects, some results are available on ferritic-martensitic steels, which present a composition close to mod 9Cr-1Mo steel, which is the candidate for HTR RPV, but no data are available on this specific cast in the operating conditions planned for HTR RPV.
Interesting results were obtained on 9-12%Cr steels for FBR, Fusion and PWR applications. It was shown that swelling is negligible for these steels, DBTT appears to remain satisfactory for steels with a Cr content $\leq$ 9%, there is a strong effect of initial metallurgical conditions on post-irradiation behaviour, and they present a low activation when compared to austenitic steels.
Nevertheless, it is necessary to get data on the specific cast which will be used for the HTR RPV and in representative operating conditions: irradiation temperature, neutronic flux, cumulated dose. Investigations have also to be made to characterize the behaviour of welds in such conditions.

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## 6. FIGURES

**Figure 1 : influence of irradiation on swelling for different materials - Ref. [10]**

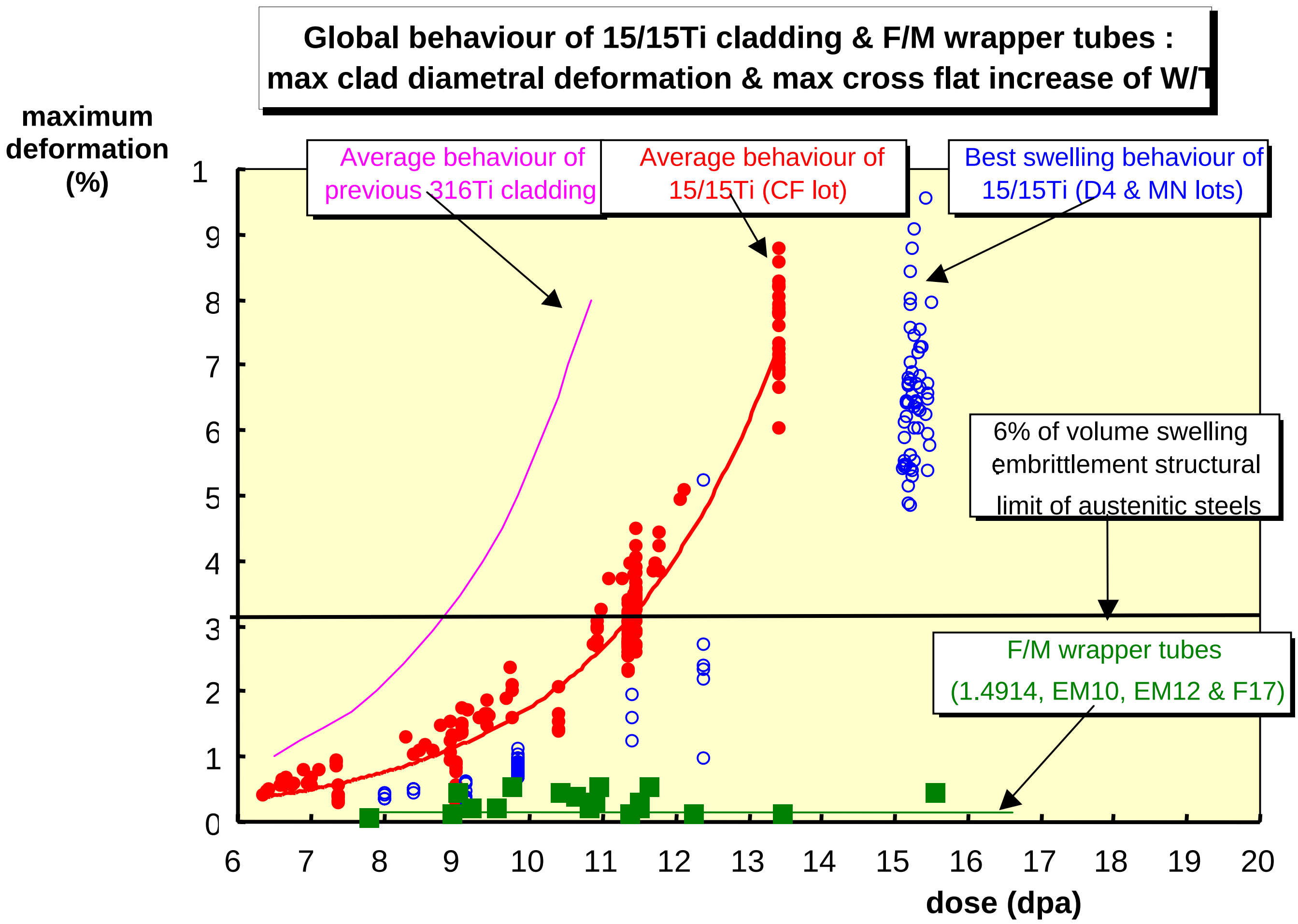


**Figure 2**
**Evolution of DBTT with irradiation temperature for different ferritic-martensitic steels - Ref. [8]**

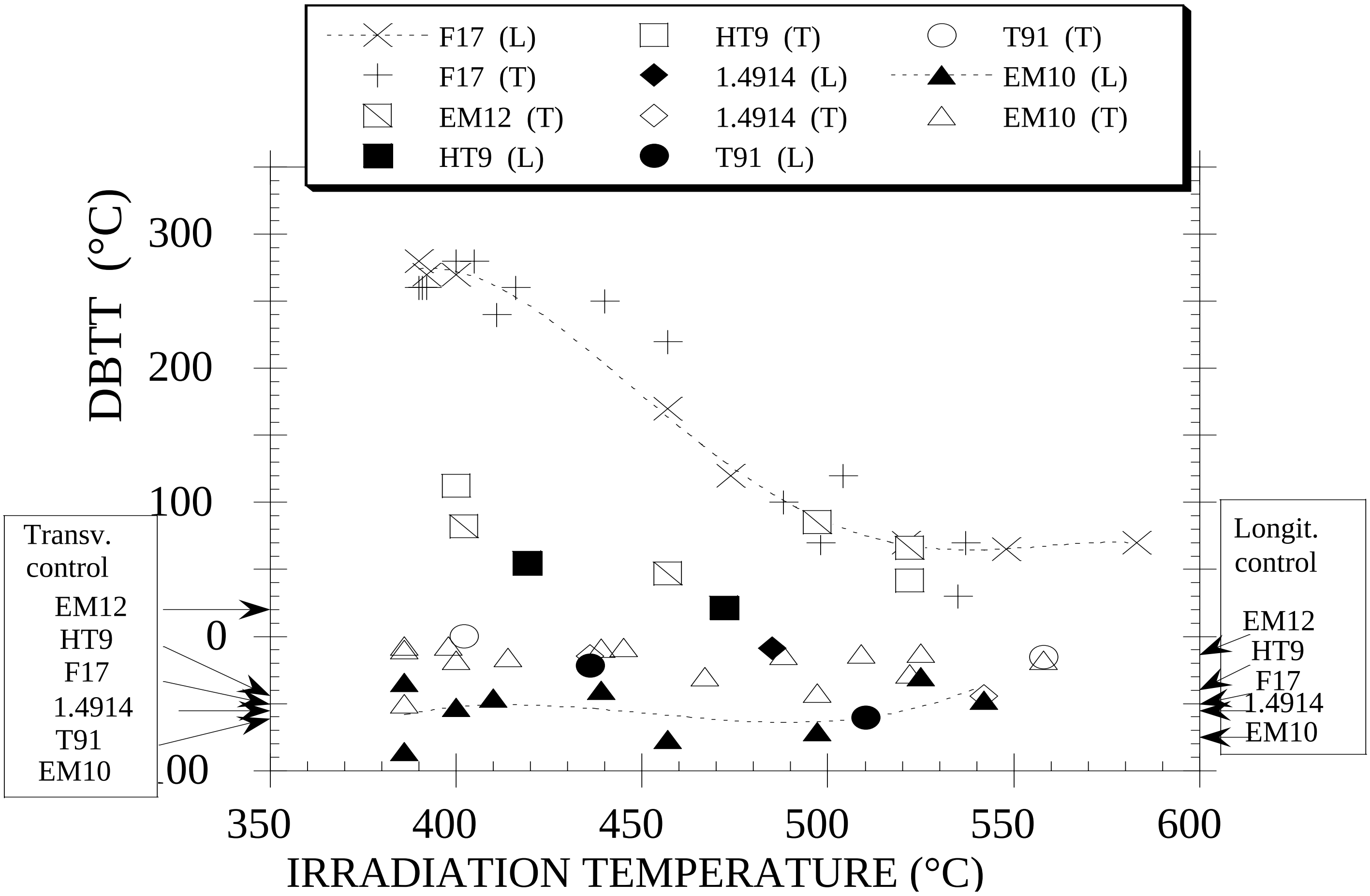
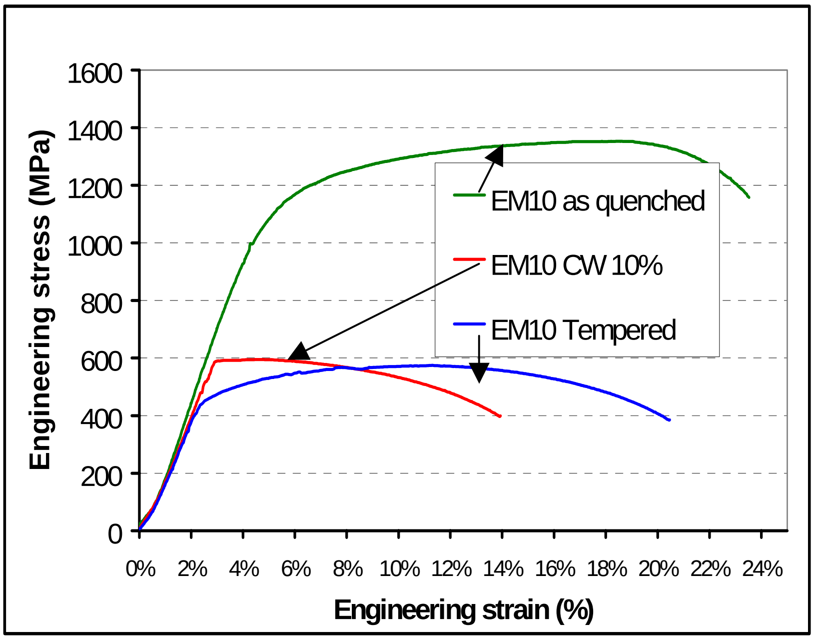
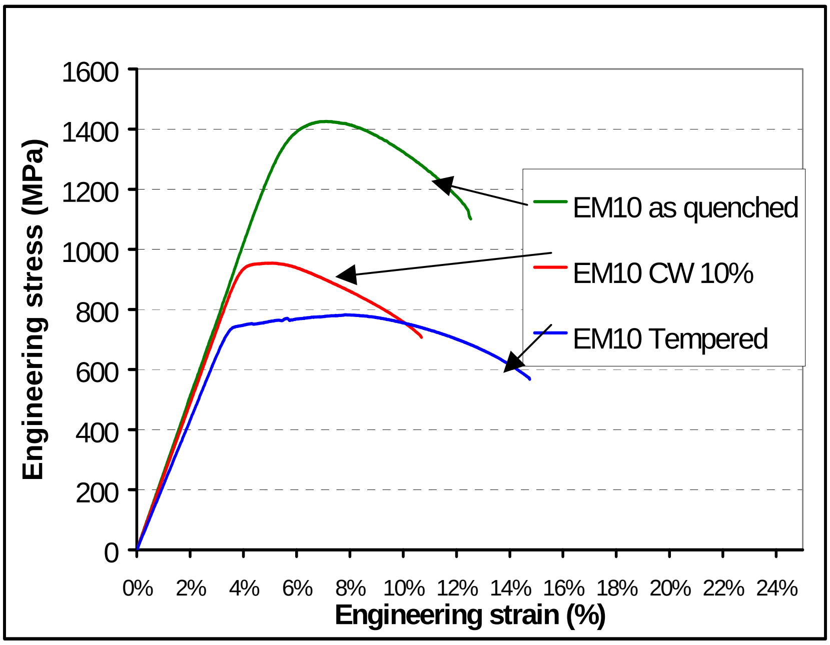


**Figure 3**
**Influence of initial metallurgical conditions on post-irradiation behavior of EM10 (non modified 9Cr-1Mo steel) - Ref. [9]**

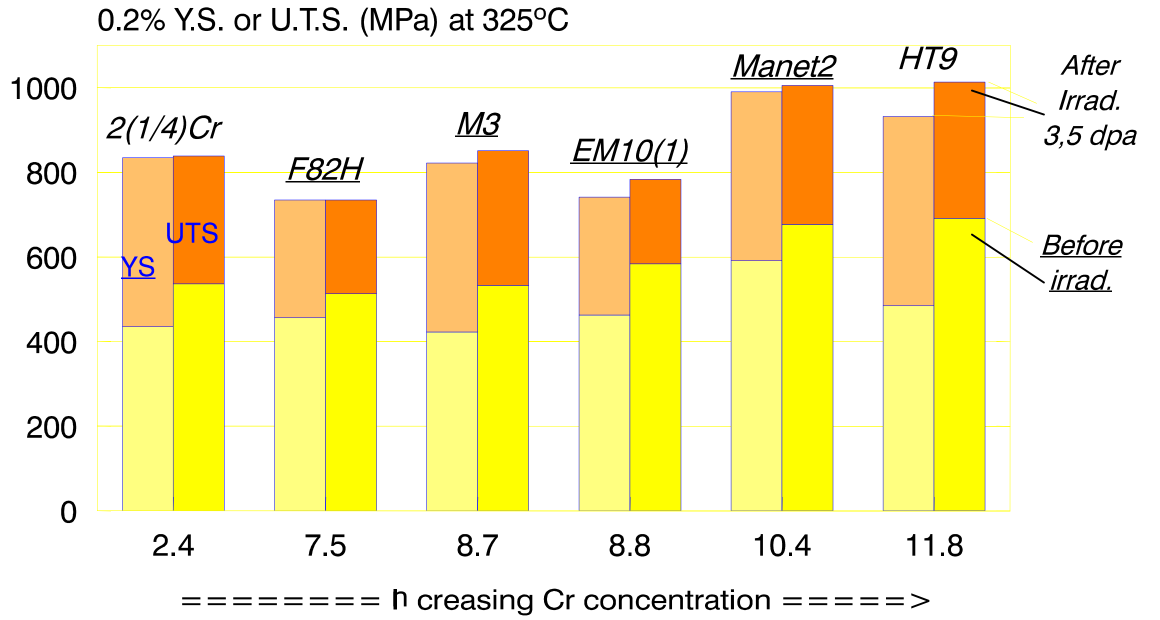


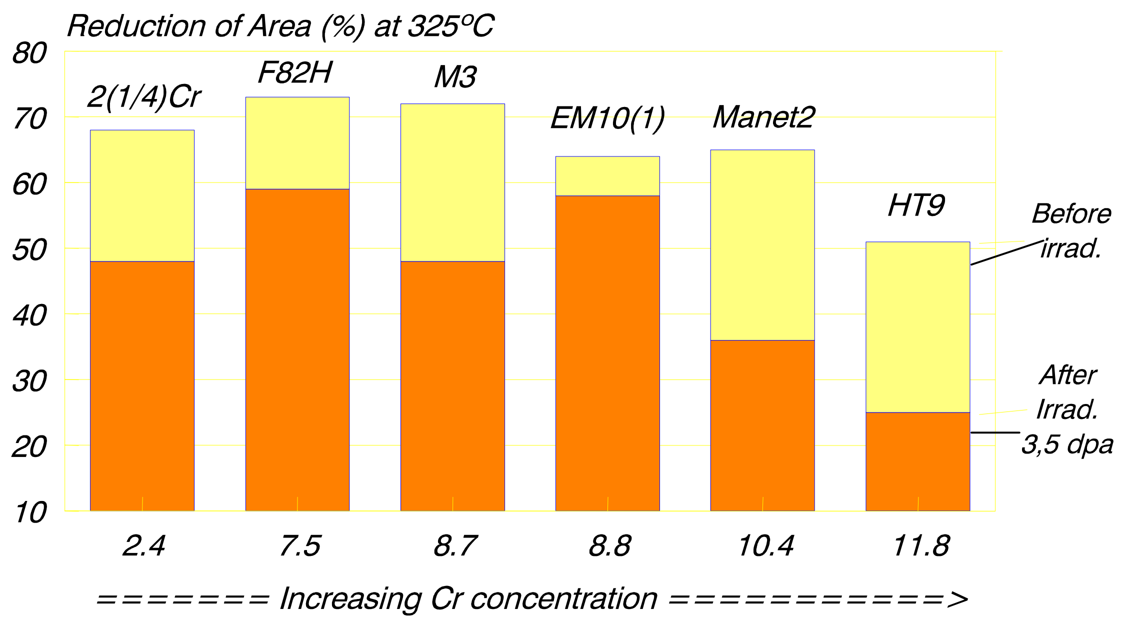

*Before irradiation ($T_{test}$=$T_{irrad.}$ =325°C)*




*After irradiation at 3.5 dpa ($T_{test}$=$T_{irrad.}$ =325°C)*

**Figure 4**
**Tensile properties at 325°C, before and after irradiation at 3.5 dpa, for tempered martensitic steels - Ref. [9]**



## FICHE DE VÉRIFICATION DU DOCUMENT

TITRE DU DOCUMENT : EFFECT OF ENVIRONMENT ON THE MATERIAL OF GT-MHR VESSEL

Rédacteur : M.T. CABRILLAT

| Domaines* de la Vérification | Résultats ** | | |
|---|---|---|---|
| **1) Vérification de la forme : respect de la PR1 G1 du MQ DER** | Conformité | | |
| Modèle de NT | C | NC | |
| Contenu des « cartouches » : page 1, page 2 et page courante | C | NC | |
| Plan du document et Pagination | C | NC | |
| Clarté de la mise en forme (traitement de texte) | C | NC | |
| Respect de l'orthographe et de la grammaire françaises | C | NC | |
| Formules, Figures, Tableaux, … (titres, unités, échelles, légendes, …) | C | NC | SO |
| **2) Vérification scientifique** | | | |
| Les documents de références et les documents applicables sont-ils mentionnés ? | Oui | Non | SO |
| L'étude s'appuie-t-elle sur une analyse de l'état de l'art ? | Oui | Non | SO |
| Le problème est-il clairement posé ? | Oui | Non | SO |
| Les hypothèses sont-elles clairement définies ? | Oui | Non | SO |
| Les données sont-elles appropriées et validées ? | Oui | Non | SO |
| Les méthodes employées sont-elles justifiées et validées ? | Oui | Non | SO |
| Les ordres de grandeur sont-ils cohérents ? | Oui | Non | SO |
| Les conclusions sont-elles justifiées ? | Oui | Non | SO |
| **3) Vérification de la clarté et de la cohérence d'ensemble** | | | |
| Le Titre est-il explicite et pertinent ? | Oui | Non | |
| Le Résumé est-il autoporteur, clair et conclusif ? | Oui | Non | |
| Le Résumé est-il cohérent avec l'Introduction et la Conclusion ? | Oui | Non | |
| Les Mots Clés sont-ils pertinents et exhaustifs ? | Oui | Non | |
| L'Introduction indique-t-elle suffisamment : les objectifs contractuels, le contexte technique, les objectifs précis, les enjeux, … | Oui | Non | |
| L'Introduction présente-t-elle l'articulation de la présentation ? | Oui | Non | |
| L'enchaînement des étapes de la présentation est-il compréhensible ? | Oui | Non | |
| La Conclusion précise-t-elle clairement le degré d'atteinte des objectifs contractuels ? | Oui | Non | |
| La Conclusion ouvre-t-elle des perspectives ? | Oui | Non | SO |
| **Bilan de la Vérification** | | | |
| Les modifications demandées ont toutes été prises en compte par le Rédacteur | Oui | Non | SO |
| Les modifications listées en PJ n'ont pas été suffisamment prises en compte par le Rédacteur et nécessitent l'arbitrage de l'Approbateur | Oui | Non | SO |
| | | | |

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| ** | C et Oui | Jugé Satisfaisant par le Vérificateur |
| ** | NC et Non | Jugé Non Satisfaisant par le Vérificateur : modifications demandées au Rédacteur |
| ** | S.O. | Jugé Sans Objet par le Vérificateur (la rubrique ne s'applique pas) |

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| **Vérificateur* :** | **Domaines* : 1, 2 et 3** | **Date :** | **Visa :** |
| **Approbateur :** | | **Date :** | **Visa :** |

* S'il y a plusieurs vérificateurs, chacun remplit une Fiche en précisant les domaines qu'il a vérifiés

# FICHE DE VERIFICATION AQ DE DOCUMENT

TITRE DU DOCUMENT : EFFECT OF ENVIRONMENT ON THE MATERIAL OF GT-MHR VESSEL

Rédacteur : M.T. CABRILLAT

| Page | Observations |
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| Vérification AQ : | F. Joyer | Date : | Visa : |
| Vérif Chef de Service : | J. Rouault | Date : | Visa : |

Après prise en compte des corrections par le rédacteur, retourner au vérificateur AQ :
- cette fiche
- les éventuelles pages annotées.
- le document réédité, après corrections

Cette fiche est à conserver avec la fiche de vérification du document.