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IN-PILE CREEP STRAIN AND FAILURE OF CW 316 Ti PRESSURIZED TUBES

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Abstract.

The in-pile creep and failure behavior of CW 316 Ti pressurized tubes irradiated in the same rig at 660-680 °C and 81.4 dpaF max in Phenix is presented and compared to monitors of the same heat.

The in-pile plastic strains are of the same order of what is expected from the monitors and are rather independent of the dose rate in the range 4 to 9×10^{-3} dpaF/h. Such a behavior supports the assumption that the out-of-pile deformation mechanisms are operative in pile and a certain balance occurs between modification of the microstructure, dynamic hardening and deformation mechanisms due to irradiation.

Examinations by fractography and optical micrography, show that the failures are intergranular either in-pile or out-of-pile. In both cases the damage consists in intergranular wedge cracks, and no cavitation can be observed by transmission electron microscopy.

Then the in-pile embrittlement which gives lower failure strain and time is to be associated with a decrease of the surface energy of grain-boundaries rather then growth and coalescence of cavities.

KEYWORDS : radiation damage, in-pile creep, embrittlement, Ti stabilized 316.

INTRODUCTION.

Thorough knowledge and understanding of the mechanical behavior of the fuel pin cladding over a wide range of temperature for stress or strain controlled loadings are needed for a reliable prediction of time and location of the pin failure during normal or not operations of the reactor.

In this paper the main concern will be the in-pile creep behavior of Ti stabilized 316 cold-worked stainless steel at high temperature. Experimental results are issued from the irradiation XT2 which was done in Phenix at ∿ 660-680 °C up to 81.4 dpaF max, corresponding to 8820 hours.

XT2 EXPERIMENT

Material.

The tested samples are 20 % CW 316 Ti pressurized tubes having the diameter and wall thickness of Phenix fuel pin cladding. The material consists in two experimental batches WY1 and WY2 issued from the same heat. The chemical composition is within the AFNOR standardization : Z6 CNDT 17-13. The main difference between them is the grain size : 19 µm for WY1 and 44 µm for WY2.

In-pile experiment.

The experimental rig consists in an eight-level capsule. An estimation of temperatures is obtained by post-irradiation tensile tests (see Appendix). The four upper levels have a temperature in the range 660-680 °C in good agreement with thermal calculation.

Figure 1 shows the temperature and dose profiles of XT2 experiment, table I-a the stresses applied to the tubes of WY1 and WY2 batches.

Out-of-pile experiment.

Creep tests were carried out on monitors. Applied temperatures and stresses for the creep tests analysed hereafter are given in table I-b.

T(°C) / σ_θ (MPa)	660-680
50	WY2
100	WY1

(a)

T(°C) / σ_θ (MPa)	600	650	700
50		WY1	WY1
100	WY1	WY1	WY1
200		WY2	

(b)

Table I : Batches, temperatures and stresses in XT2 experiment :
(a) in-pile ; (b) out-of-pile (monitors)

ANALYSIS OF THE OUT-OF-PILE RESULTS.

Examples of the time evolution of plastic strain for WY1 batch are given in figure 2 : a - 600 °C, b - 650 °C and c - 700 °C. At 600 °C under 50 MPa, strains and experimental accuracy are of the same order. At 550 °C no deformation is recorded up to 20 000 h.

Creep stages and failure occurence.

For 600 and 650 °C it is difficult to detect a primary stage. For 600 and 650 °C under 100 and 50 MPa respectively, no tertiary stage appears. For 650°C under 100 MPa, a tertiary stage seems to occur around 10 000 h without any specimen failure up to 15 000 h.

At 700 °C primary, secondary and tertiary stages seem to exist under 50 MPa at least. Times to failure are in the range of 10 000 h under 100 MPa and over 7 500 h under 50 MPa.

Stress dependence and deformation mechanisms.

The 600-650 °C and 700-750 °C ranges are to be considered separately.

In the 600-650 °C range, the stress dependence is quasi-linear : strains are $\sim$ 0.2% under 50 MPa and $\sim$ 0.5% under 100 MPa at 10^4 h (see figure 2-b). Such a dependence is in general agreement with diffusion creep. By comparison with theoretical prediction of COBLE [1] or NABARRO-HERRING [2], the WY1 strain rates could be mostly due to diffusion creep (see figure 3). Diffusion data used to do this comparison concern a 17% Cr - 12% Ni austenic alloy and are taken from [3]. Experimental strain rates have an order of magnitude that seems closer to NABARRO-HERRING prediction than COBLE one. This could reflect an inhibition of the COBLE creep ; the same behavior is observed in Nb or Ti stabilized 20Cr - 30Ni stainless steel in unaged condition [4], while ageing before creep improves the agreement with COBLE prediction [5].

At 700 °C, the stress dependence of strain rates is no more linear : active mechanisms involve dislocation motions under 50 and 100 MPa.

Creep damage.

Test (1) reported figure 2-b (WY1 batch, 100 MPa, 650 °C and 1.5 x 10^4h) was interrupted to look for creep damage, by optical and transmission electron microscopy (TEM). Grain boundaries display no damage neither creep cavities nor wedge cracks.

Under 200 MPa at 650 °C, failures of WY2 tubes occur between 2500 and 5000 h. Examinations by scanning electron microscopy (SEM) of the outer surface show that these failures result from very thin and localized cracks. Optical observation of a cross section of one end of this crack displays an non-through intergranular crack (fig.4). As no creep cavities can be found by TEM [6], the black spots on grain-boundaries (see fig.4) must be intergranular precipitation which essentially consists of σ phase as revealed by TEM [6]. Otherwise localized wedge cracks exist (their direction being 45° from the crack of fig.5) : they might have nucleated on the intergranular precipitates as shown figure 5.

ANALYSIS OF THE IN-PILE RESULTS.

Density changes are always less than ∿ 0.1 - 0.2 %. The few small voids observed by TEM in some areas [6] cannot account for such changes. The density results are to be explained by some other aspect of the microstructural evolution, especially precipitation. The plastic hoop strain is calculated by difference between the total hoop strain and the third of the swelling.

In-pile creep behavior of WY2 batch (50 MPa).

The measured gas pressure after irradiation shows that all the tubes kept their initial gas content. As shown on figure 6 dose or dpa rate evolutions of plastic strains can be splitted into two groups.

Plastic strains for the lower levels 1,2 and 3 ($T_I \lesssim 600$ °C) are hardly larger than the experimental accuracy. Nevertheless they are in qualitative agreement with an expression such as :

$$\varepsilon_\theta^P = \frac{3}{4} B \, \phi t \, \sigma_\theta$$

which is typical of the irradiation creep and where B is taken temperature independent according to plastic strain analysis of CW 316 Ti fuel pin cladding.

On the opposite, for the higher temperature (levels 5 to 8) the plastic strains are rather independent of the dose or dpa rate and thus cannot be described by the previous expression even with a larger creep modulus. Therefore we tend to conclude that the observed strains are mainly due to out-of-pile deformation mechanisms.

Finally let us point out that the out-of-pile and in-pile strains are of the same order of magnitude. Unfortunately a direct comparison cannot be made because the batches and temperatures are different.Nevertheless between 660 and 680 °C under 50 MPa, the knowledge of WY2 creep behavior leads to predict an out-of-pile strain in the range .3, 1.2% at 8820 h which essentially results from dislocation creep and is in good agreement with the in-pile strains ∿.6% (see fig.6).

Creep damage.

By checking the gas pressure, the tubes of the WY1 batch having an initial hoop stress of 100 MPa are found empty after irradiation in levels 5 to 8. Like for the monitors, the failures occur as very thin and localized cracks. An example observed on the outer surface by SEM is shown in figure 7. The crack surfaces are of intergranular nature and TEM examinations display no grain-boundary cavitation.

Under 50 MPa, no damage is noticeable neither crack nor grain-boundary cavitation.

Finally although better conditions for cavitation are fulfilled for the in-pile samples (smaller grain size, lower stress and higher temperature than for the out-of-pile tests), no grain-boundary cavity occurs and the creep damage remains of wedge-crack type. Nevertheless the failure strains and times of WY1 under 100 MPa (see fig.8) are smaller for in-pile tests thus showing some embrittlement occurs under irradiation.

SUMMARY AND DISCUSSION.

Deformation mechanisms.

At 660-680°C the in-pile creep strains of the 20% CW 316 Ti seem to be of the same order as out-of-pile and independent of the dose rate in contrast with the usual irradiation creep behavior at lower temperatures. Therefore the main operative deformation mechanisms may be the out-of-pile ones which give very similar strains in spite of the neutron flux.

Out-of-pile creep strains may be altered in several ways by irradiation :

i) the modificationsof the microstructure (especially precipitation and the variation of the dislocation density) as well as the point defect concentrations will affect the strain rate of out-of-pile deformation mechanisms [7, 8],

ii) fast neutron irradiation can enhance out-of-pile deformation mechanisms [9,10] or create new ones [9].

Fortunately for XT2 experiment in-pile and out-of-pile microstructures are very similar as far as dislocation density, crystallographic nature, morphology and density of precipitates are concerned [6]. So according to i) irradiation could essentially act through the dynamic hardening due to different point defect concentrations [8].

Irradiation gives rise to new mechanisms like SIPA [11] or intensifies the out-of-pile dislocation creep. Everytime the deformation is due to dislocation glide controlled by climb SIPA mechanism enhances it [9]. Irradiation is liable to intensify as well every creep rate controlled by anihilation of edge dislocation dipole in subgrain boundaries by climb [10].

So finding very similar values for the in-pile and out-of-pile strains means occurence of a certain balance between dynamic hardening and mechanism modification due to irradiation.

Creep damage.

The creep damage of the 20% CW 316 Ti is localized around the failure and consists mainly in wedge cracks with no cavitation. So the evolution of the creep damage should be described as crack nucleation and growth controlled by grain boundary sliding [12].

Then the in-pile embrittlement which gives a smaller failure strain and time is to be associated with a decrease of the surface energy of the grain boundaries rather than growth and coalescence of cavities on them.

Despite the microstructure difference and the higher He concentration ($\sim$ 20 ppm He in XT2 experiment), such a damage behavior seems to be very similar to the B-doped 316 one when crept out-of-pile after thermal irradiation [13].

APPENDIX

To obtain a good estimation of the irradiation temperature is a worrysome problem when no thermocouple is available. A rather reliable method is based on the comparison of the yield stresses of irradiated specimens and samples taken from objects with well known temperature : fuel pin cladding.

Figure A shows that in the range of 30 to 100 dpaF, the yield stress of fuel pin cladding at room temperature is independent of dose and thus can be described by a function of the sole irradiation temperature.

Yield stress measured for XT2 experiment gives then an estimation of the irradiation temperature ranging from 450 °C for the lower to 660-680 °C for the upper levels, which is in rather good agreement with thermal calculations.

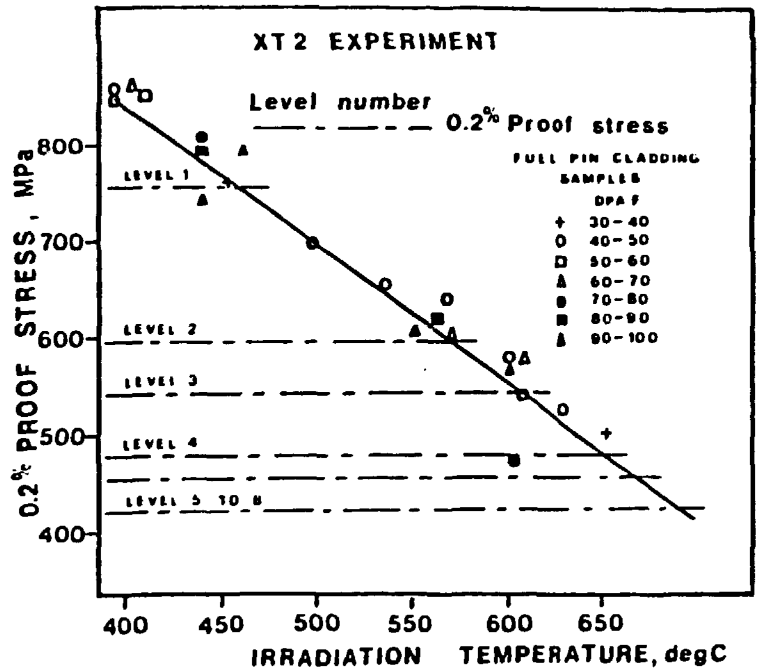

Figure A : Room temperature yield stress of CW 316 Ti fuel pin cladding and XT2 samples versus irradiation temperature

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FIGURE CAPTIONS.

Figure 1 : Temperature and dose profiles of XT2 in-pile experiment.

Figure 2 : Hoop strain of WY1 batch versus time at 600°C (a), 650°C (b) and 700°C (c).

Figure 3 : Comparison between experimental unitary strain rate and prediction of diffusion creep theories.

Figure 4 : Optical micrography of intergranular failure obtained on WY2 monitor (temperature : 650 °C ; hoop stress : 200 MPa).

Figure 5 : Internal wedge crack observed by SEM on a the same cross section as in figure 4.

Figure 6 : In-pile hoop strain of WY2 batch versus dose or dose rate.

Figure 7 : SEM observation of the outer surface of tubes from WY1 batch failed in-pile under 100 MPa at 660-680 °C.

Figure 8 : Temperature versus hoop strain at failure a and time to failure b (hoop stress : 100 MPa).

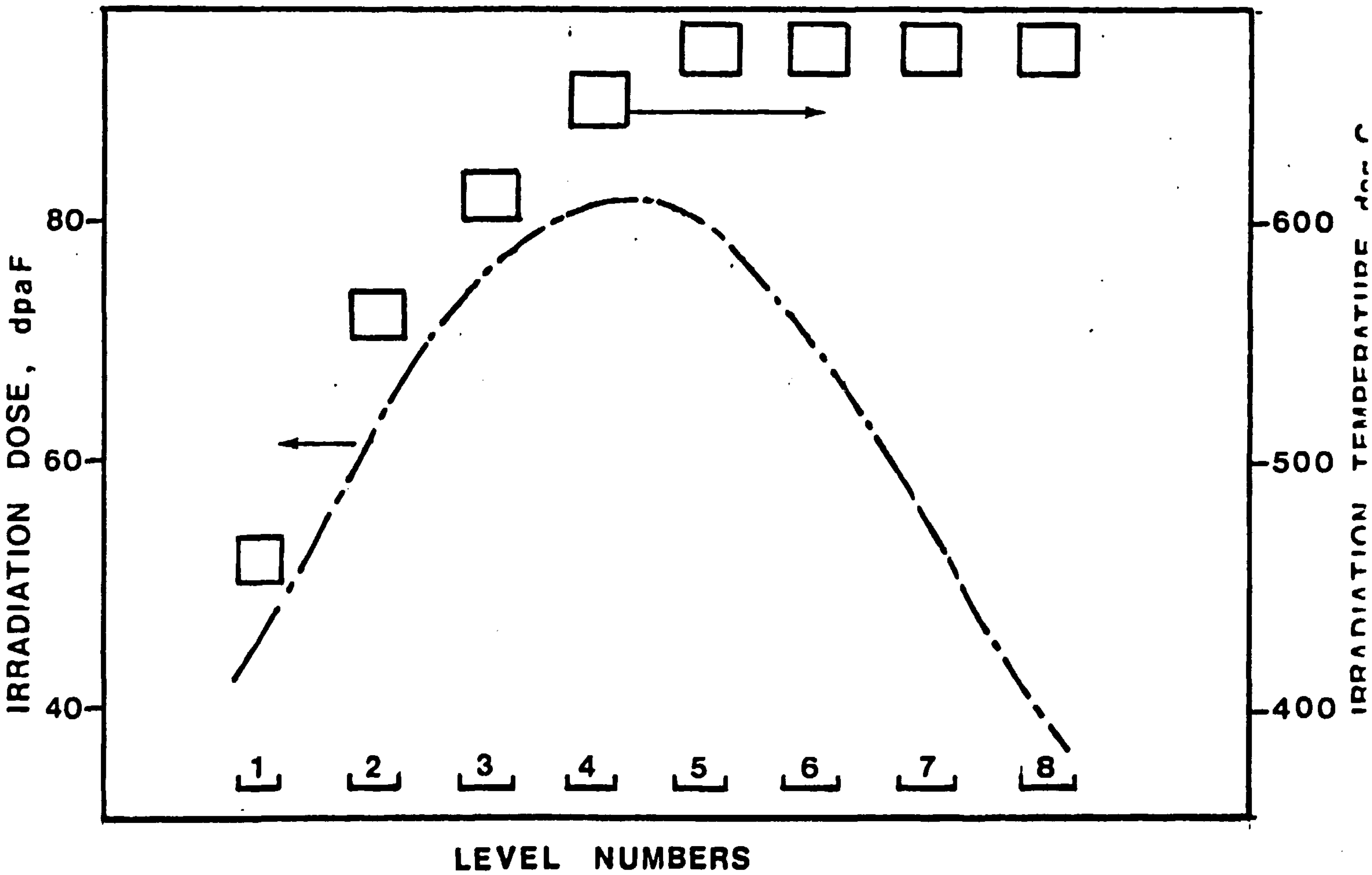
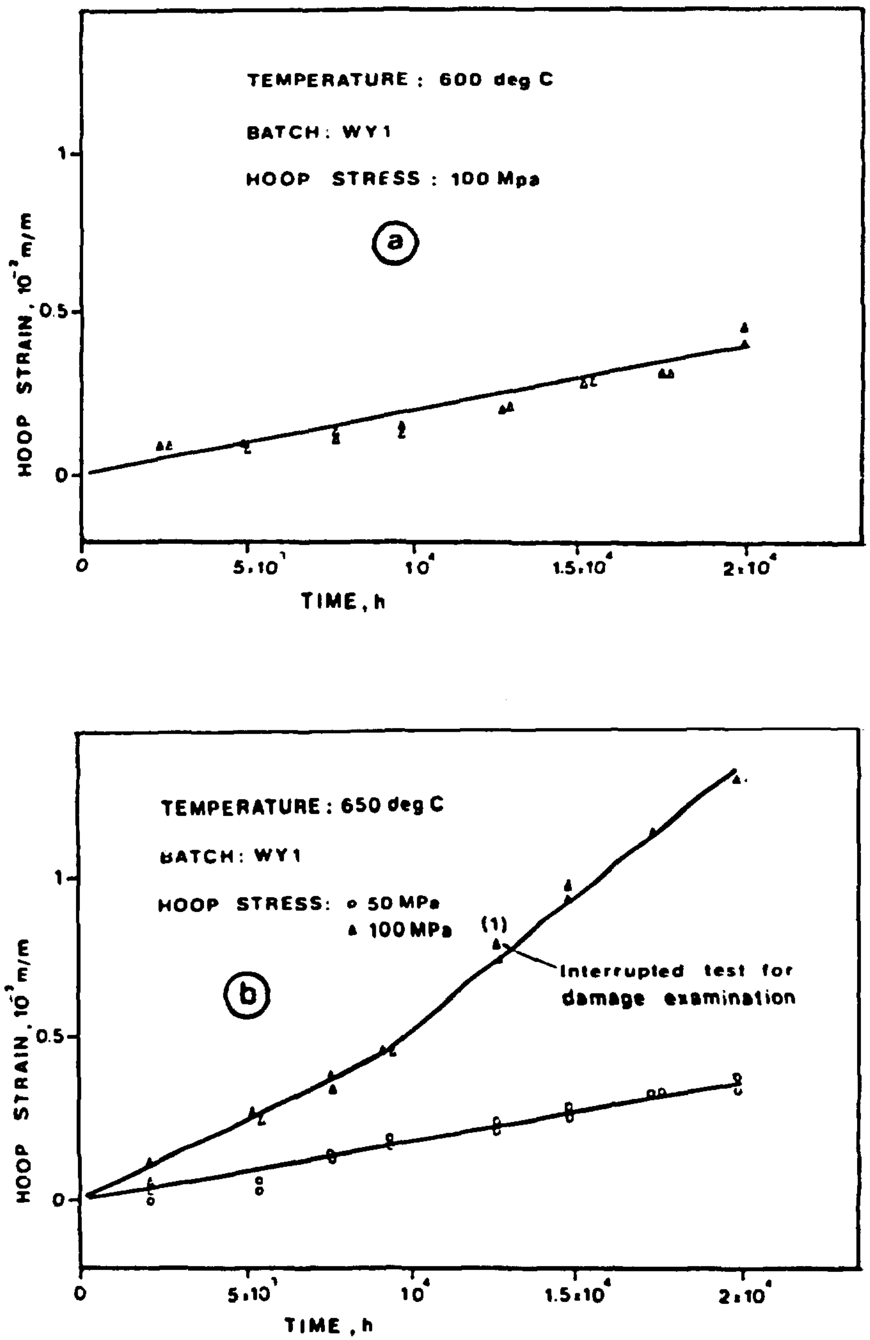

80
60
40
600
500
400
IRRADIATION DOSE, dpaF
1
2
3
4
5
6
7
8
LEVEL NUMBERS

Fig. 1


TEMPERATURE : 600 deg C
BATCH : WY1
HOOP STRESS : 100 Mpa
a
HOOP STRAIN, 10^{-2} m/m
TIME, h
b
TEMPERATURE : 650 deg C
BATCH : WY1
HOOP STRESS : o 50 MPa
▲ 100 MPa
(1)
Interrupted test for
damage examination
HOOP STRAIN, 10^{-3} m/m
TIME, h

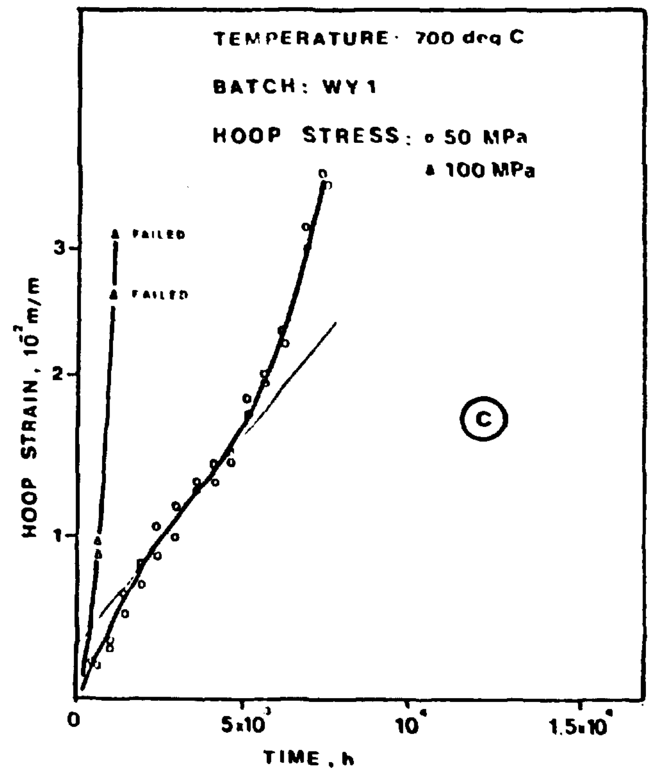

TEMPERATURE : 700 deg C
BATCH : WY 1
HOOP STRESS : o 50 MPa
▲ 100 MPa
FAILED
FAILED
c
HOOP STRAIN, 10^{-2} m/m
TIME, h

Fig. 2

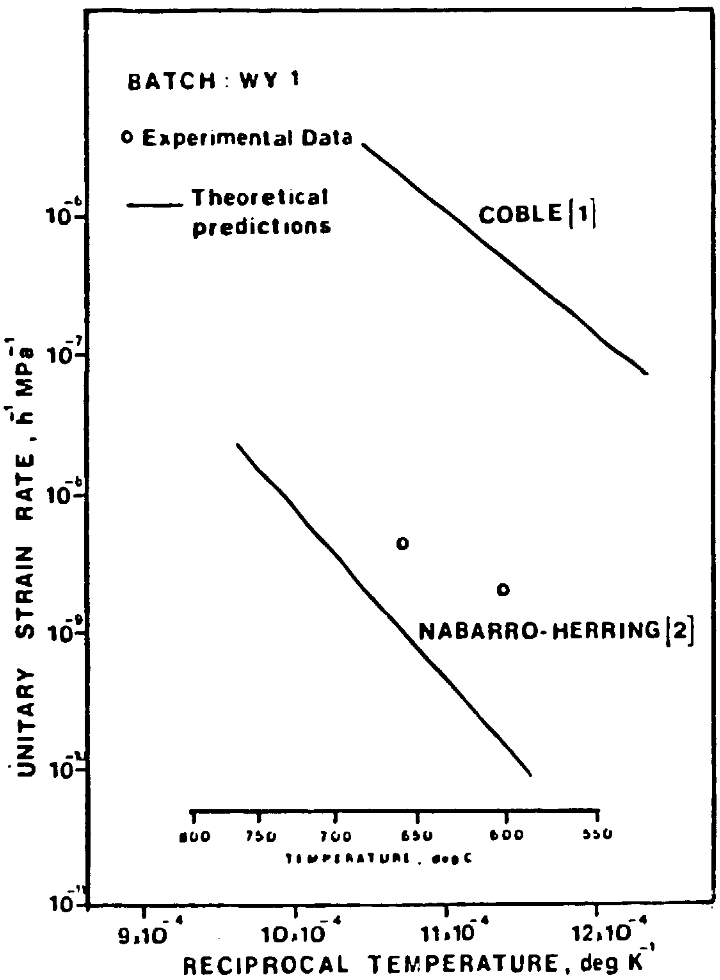

BATCH : WY 1
o Experimental Data
Theoretical predictions
COBLE [1]
NABARRO-HERRING [2]
UNITARY STRAIN RATE, h⁻¹ MPa⁻¹
800 750 700 650 600 550
9x10⁻⁴ 10x10⁻⁴ 11x10⁻⁴ 12x10⁻⁴
RECIPROCAL TEMPERATURE, deg K⁻¹

Fig. 3

Fig. 4

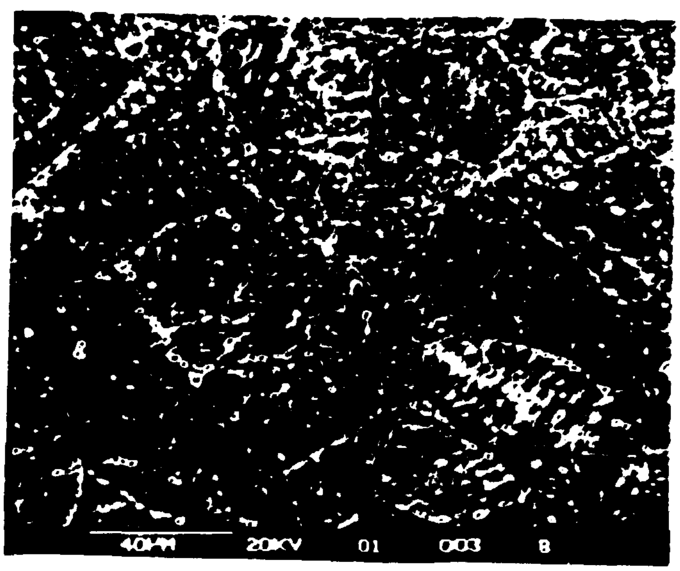


Fig. 5-a

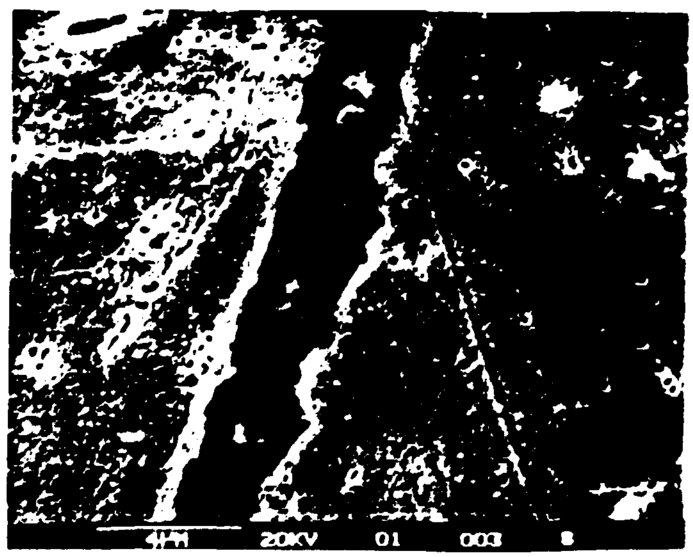


Fig. 5-b

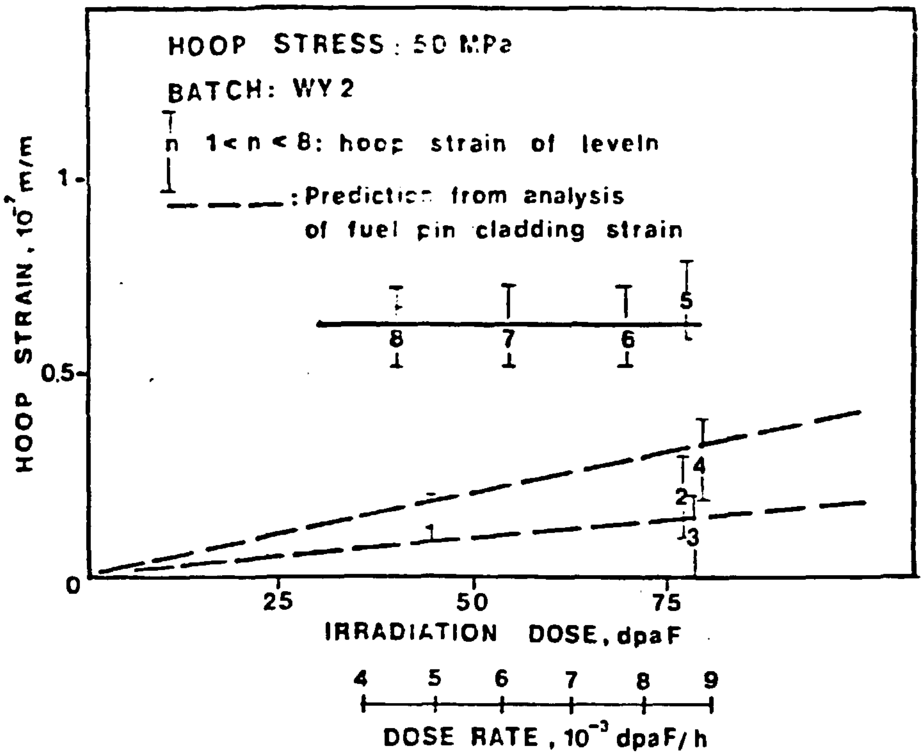

HOOP STRESS : 50 MPa
BATCH: WY 2
1 < n < 8: hoop strain of level n
: Prediction from analysis of fuel pin cladding strain
HOOP STRAIN, 10^{-2} m/m
IRRADIATION DOSE, dpaF
DOSE RATE, 10^{-3} dpaF/h

Fig. 6

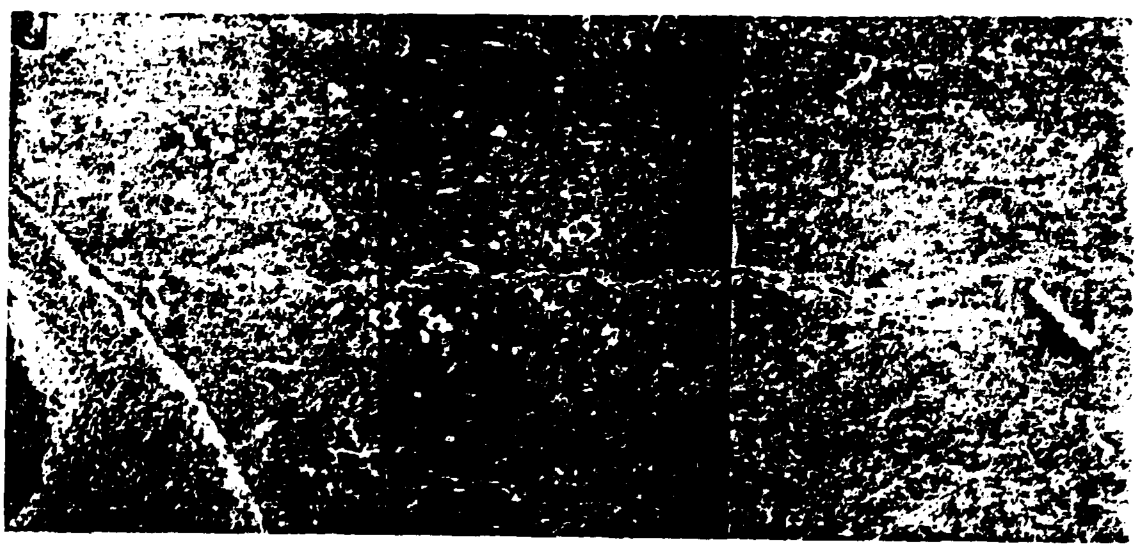


Fig. 7

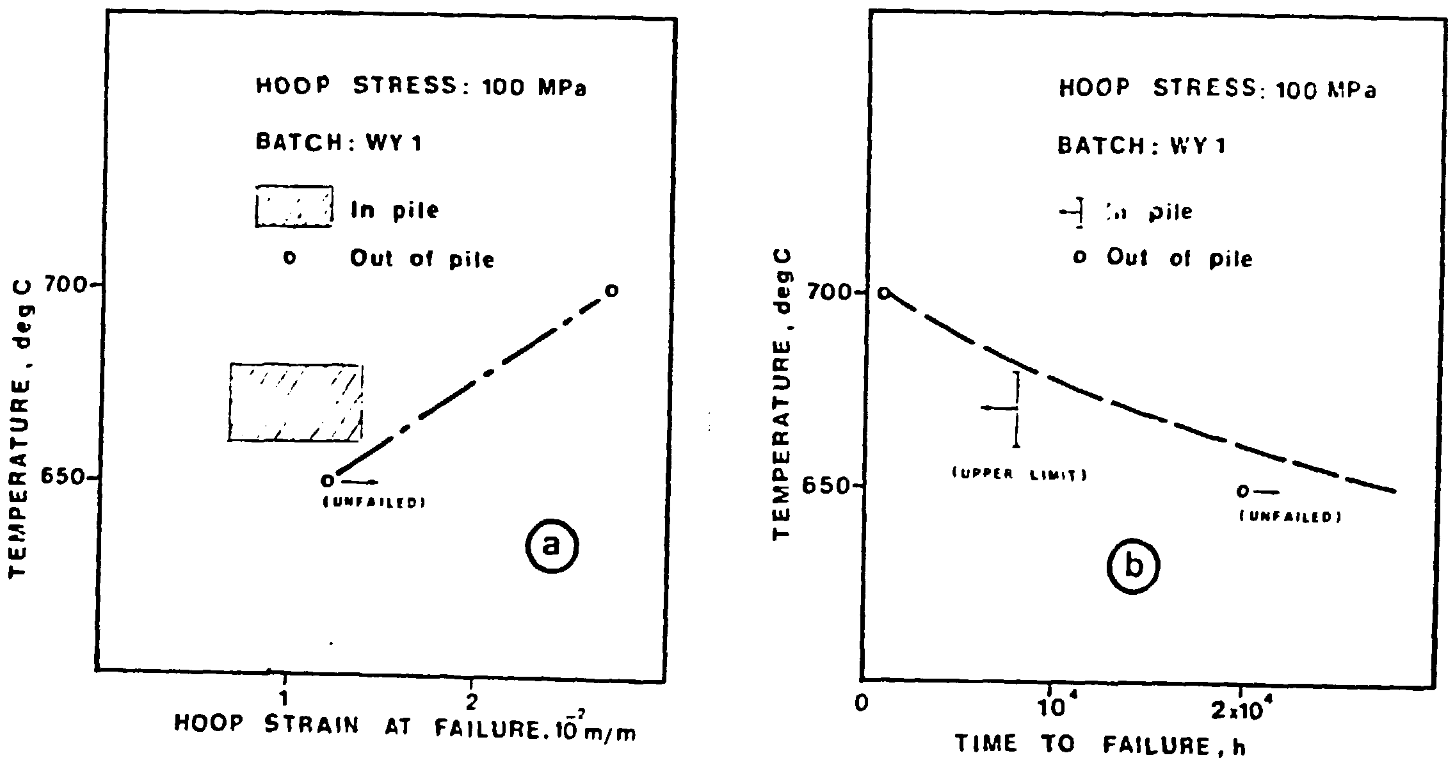

HOOP STRESS: 100 MPa
BATCH: WY 1
In pile
Out of pile
TEMPERATURE, deg C
700
650
(UNFAILED)
a
1
2
HOOP STRAIN AT FAILURE. 10^{-7} m/m
HOOP STRESS: 100 MPa
BATCH: WY 1
In pile
Out of pile
700
650
(UPPER LIMIT)
(UNFAILED)
b
0
10^4
2×10^4
TIME TO FAILURE, h

Fig. 8

20ème SYMPOSIUM INTERNATIONAL SUR LES EFFETS DE L'IRRADIATION SUR LES MATERIAUX

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ANALYSE DU COMPORTEMENT ET DES RUPTURES EN PILE DE TUBES PRESSURISES EN 316 Ti ECROUI

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RESUME.

On présente l'analyse du comportement en pile de tubes pressurisés irradiés dans une même expérience à 660-680 °C et jusqu'à 80 dpaF dans PHENIX.

Les variations de densité sont inférieures à 0,2% pour tous les échantillons et vraisemblablement dues à l'évolution microstructurale plutôt qu'à du gonflement comme l'indiquent les examens de microscopie électronique. L'étude de la microstructure révèle qu'il n'y a pas de différence fondamentale entre matériaux irradiés et moniteurs thermiques.

Les déformations plastiques obtenues en pile sont du même ordre de grandeur que celles des moniteurs testés hors pile et sont indépendantes du taux de création de défaut dans le domaine 4 à 9 x 10^{-3} dpaF/h. Un tel comportement suggère que les mécanismes de déformation hors pile restent opérant sous flux et qu'une certaine balance existe entre les phénomènes typiques de l'irradiation que sont les modifications de microstructure, de mécanismes de déformation et le durcissement dynamique.

Les ruptures obtenues en pile sur certains tubes présentent un faciès intergranulaire. Des examens micrographiques effectués sur moniteur révèlent, d'une part l'absence de cavités de fluage et d'autre part, la présence de nombreuses fissures secondaires adjacentes à la surface de rupture.

Ces résultats indiquent que le dommage de fluage du 316 Ti écroui est plutôt contrôlé par la déformation plastique.