INTRODUCTION.

It is generally known that the stable growth of cracks in the creep regime is governed by specific micromechanisms, mainly diffusion and/or deformation controlled, which lead to different types of damage processes /1/ going from ductile transgranular fracture to intergranular creep fracture through the transgranular creep fracture.

While the strain-stress field at crack tip for brittle materials or for highly constrained zones could be easily and carefully described by $K$, the stress intensity factor of the Linear Elastic Fracture Mechanics, for ductile materials $C^3$, the Creep Fracture Mechanics parameter, has been introduced.

It is known that both such parameters suffer from limitations in the correlation with CCG rate, essentially depending on the extension of the plastic zone for $K$ and the effectiveness of steady state creep condition into the localized process zone ahead the crack tip for $C^3$.

The only way to define the best correlating parameter is to evict which micromechanism is acting at the crack tip of a crack growing steadily by creep /2/: for this reason a metallographic post test examination of the creep crack growth specimens had been performed.

Aim of this report is to summarize the results of the microstructural survey performed by means of optical, scanning and transmission electron microscopies on the here tested six specimens.

The described activity had been done within the framework and financial support of the EEC Fast Breeder Coordinating Committee-Working Group for Code and Standards, Activity Group 3 "Materials". The EEC contract had been placed of INTERATOM and subcontracted of ENEL-CRTN, to well as at CEA-France and UKAEA-United Kingdom.

MATERIAL AND CCG DATA.

Material specifications and experimental techniques have been previously detailed /3/; Table 1 shows the material chemical composition.

The results of the Round Robin CCG tests on 316 L mod. stainless steel performed at ENEL are reported in Table 2 together with calculated engineering figures, such as the creep zone radius and the total blunting radius, computed using the formulas reported in the literature /4/ and /3/ respectively.
It must be noticed that test X04 has been reloaded after 2843 h for other 11,360 h and test X05 was interrupted after 1293 h because of grips failure, then continued till to 2679 h total testing time.

Comparing the initiation time of CCG, taken at the load line displacement vs time turning point, with transition time /4/ from elastic plastic regime to steady-state creep it appears quite clearly that \( C^* \) is the load parameter governing the CCG for this material, so the correlation of the CCG rate \( da/dt \) vs \( C^* \) was made and is described by the following power law:

\[
\frac{da}{dt} = 10^{-5} \cdot 0.76 \cdot C^*
\]

where \( da/dt \) is expressed in mm/s and \( C^* \) as N/m s.

This correlation of the CCG rate with \( C^* \) shows also the lowest scatter as reported in Fig. 1, with respect to \( K \) and \( \sigma_{net} \) correlations, even if it could be matter of discussion its validity throughout the overall CCG rate range.

**POST TESTS EXAMINATIONS.**

Microhardness tests, microstructural observations and blunting measurements had been performed on the surface orthogonal to the CCG plane in the middle of the specimen thickness, to characterize the crack at its maximum propagation.

For the fractographic studies the fracture surface had been observed after specific chemical deoxidation treatment.

At the crack initiation there are usually two twin cracks, growing at 45° degrees with respect to the precracking plane, and only one carries on the propagation; because of that, blunting radius measurements with appreciable accuracy could be made only observing both fracture surfaces rejoined.

By grids of microhardness Vickers 50 gr tests it had been found that for the specimens in which the crack propagated (X02, X03 and X06) the microhardness has very high values, till to 280 gr/\( \mu \)m, near to the fracture surface and it decreases departing from the crack to reach a mean and constant asymptotical value of 175 gr/\( \mu \)m, equal to that of the unloaded zone, at about 8-10 mm far to the crack surface, Fig. 2.

This trend is quite constant along the crack growing path and no specific microhardness increase related to the crack growth length had been evicted, Fig. 3.

In X05 specimen the maximum microhardness value was 210 gr/\( \mu \)m and the sharp decreasing trend was not observed; this specimen had been reloaded after grips' failure.

The general remarks of the microstructural observations are detailed as follow and then summarised in Table 3:

- In the crack propagated specimens (X02, X03 and X06) it had been evicted a grain deformation localised at initial crack tip, with the grain elongated in the direction of 125° degrees with respect to the precracking plane, Fig. 4. This strained zone has a dimension ranging from 1 to 2mm correlable with the initial \( \sigma_{net} \) stress applied, Table 3.
In cases of extensive propagation (X03, X05 and X06) grain boundary detaching is present, due to a large wedge type cavitation, Fig. 5, which involves a depth variable from 1 to 20 grains below the fracture surface correlable with initially applied stress, Table 3.

Carbides precipitation has been fairly evicted just in the specimen X04 along the grain boundary, Fig. 6, probably for its longest exposition time.

By fractographic observations it had been evicted that all the specimens have a type of fracture intergranular, Fig. 7, except X03 specimen that shows an alternatively intergranular and ductile-transgranular fracture along the crack path, Fig. 8.

This alternation of the type of fracture could be justify by the high crack tip shear strain acting there during the propagation at 45° degrees: so, as marked by the arrows in Fig. 4, when the crack growing direction is far away from the initial crack plane the crack is constrained to return in its original direction with an high local matrix strain and leaving a twin stopped short crack.

Transmission electron microscopy methods had been applied here to obtain dislocation density of X03 specimen. Microstructural observations, Fig. 9 and 10, and measurements had been performed in four positions at decreasing distances from the fracture surface, leading to the following results:

<table>
<thead>
<tr>
<th>Dist. from fracture surf.</th>
<th>unload.zone</th>
<th>30 mm</th>
<th>18 mm</th>
<th>3 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>-2</td>
<td>9</td>
<td>10</td>
</tr>
<tr>
<td>Dislocation density (cm²)</td>
<td>4x10</td>
<td>7.7x10</td>
<td>1x10</td>
<td>2.1x10</td>
</tr>
</tbody>
</table>

It comes out that dislocation density increases by about an order of magnitude, going from the unloaded zone (4x10⁹ cm²), up to the fracture surface (2x10¹⁰ cm²).

These results had been computed using the relationship:

\[
\rho = \frac{2N I}{L t}
\]

where \(N\) is the number of intersections between dislocations and circumferences of total length \(L\) placed on the micrographs of the area of interest, \(I\) is the micrographs magnification and \(t\) the foil thickness evaluated by the "thickness fringes" method.

\(\rho\) values obtained are affected by about a 10% error, due to the inherent inaccuracies of the experimental method applied. Moreover it should be noted that results relate to a single CCG specimen; an extention of this is needed to carry out a quantitative assessment of the relation between dislocation density and local plastic strain.

DISCUSSION AND CONCLUSIONS.

From the computed values of the CCG engineering parameters such as initiation time, transition time, crack tip blunting radius and creep zone dimensions, \(C^*\) was discriminated as the best Fracture Mechanics parameter both to describe the strain-stress
field at the crack tip during propagation and to correlate with CCG rates.

The microstructural survey allows to confirm generally this idea, even if evidences, into the explanation of the micromechanisms damaging the material at the crack tip, introduce specific limitation of the C* as deduced from power-law stable creep representativity: indeed, while the intergranular cavitation damage and the grain deformation are very localized near to the crack tip, and so, near to the fracture surface, microhardness values and dislocations’ density even if decreasing, departing from the crack plane, remain at high level testifying an high and widespread creep deformation on overall the specimen.

So, massive grain deformation seems to be due mainly to the shear stress at the crack tip. Cavitation, always of wedge type, appears to be mainly controlled by local matrix creep deformation and grain boundary sliding.

Carbides’ precipitation is present just in the longest duration test, indicative of a regime governed by time ageing.

Transgranular-ductile fracture is evicted in one case when the crack is long growing and the alternation of inter and transgranular type could be related with high shear strain at the tips of twin cracks.

The correspondence between the blunting radius figures calculated and metallographically measured are in fairly good agreement confirming the goodness of the proposed /3/ equation as well as of the hypothesis that the crack starts to growth at the turning point of the load line displacement vs time curve.

In conclusion it could be said that the micromechanisms acting at the crack tip during the creep crack propagation are mainly governed by deformation; moreover high values of crack tip localized strain, assumed during the incubation time, are indicative of a primary creep type mainly acting into the material.

So, even if a more quantitative description of the phenomena could arise only from the comparison of such survey with the other examinations performed at Interatoom, CEA and UKAEA, it could be concluded, at the moment, that surely K is not applicable but C*, to be better representative of the micromechanism acting during the CCG into the AISI 316 SS at 550°C, should be computed including the primary creep.

REFERENCES.

/1/Ashby M. F., Fields R. J., Weerasooriya T., Mechanisms in two austenitic and one ferritic steel, University of Cambridge, Department of Engineering, Report n.CUED/C/MATS/Tr.47.
TABLE 1 - Material chemical composition. (weight %)

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Cu</th>
<th>Co</th>
<th>Nb</th>
<th>W</th>
<th>Ti</th>
<th>B</th>
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</thead>
<tbody>
<tr>
<td>.02</td>
<td>.23</td>
<td>2.05</td>
<td>.02</td>
<td>17.5</td>
<td>2.4</td>
<td>12.5</td>
<td>.05</td>
<td>.05</td>
<td>.01</td>
<td>.07</td>
<td>.01</td>
<td>.0004</td>
</tr>
</tbody>
</table>

TABLE 2 - Creep Crack Growth data.

<table>
<thead>
<tr>
<th>Spec.</th>
<th>( \sigma_{\text{net}} )</th>
<th>( K_1 )</th>
<th>( a_{\text{init}} )</th>
<th>( a_{\text{fin}} )</th>
<th>Testtime</th>
<th>( V_{L,L,0} )</th>
<th>Propag.</th>
<th>( V_{L,L,i} )</th>
<th>( \Delta r_{\text{tot}} )</th>
<th>( r_{\text{creep}} )</th>
<th>( t_1 )</th>
<th>( t_{\text{meas}} )</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>MPa</td>
<td>mm·( \sqrt{\text{m}} )</td>
<td>mm</td>
<td>mm</td>
<td>hours</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
<td>hours</td>
</tr>
<tr>
<td>X01</td>
<td>295.4</td>
<td>33</td>
<td>26.08</td>
<td>500</td>
<td>642</td>
<td>0.94</td>
<td>NO</td>
<td>0.88</td>
<td>0.94</td>
<td>0.0604</td>
<td>6.42</td>
<td>642</td>
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<tr>
<td>X02</td>
<td>525.6</td>
<td>57.2</td>
<td>25.18</td>
<td>26.75</td>
<td>176</td>
<td>1.48</td>
<td>YES</td>
<td>4.62</td>
<td>0.53</td>
<td>0.1241</td>
<td>10.65</td>
<td>80</td>
</tr>
<tr>
<td>X03</td>
<td>517</td>
<td>57</td>
<td>25.56</td>
<td>32.06</td>
<td>308</td>
<td>4.62</td>
<td>YES</td>
<td>5.07</td>
<td>0.86</td>
<td>0.1880</td>
<td>10.65</td>
<td>75</td>
</tr>
<tr>
<td>X04</td>
<td>350</td>
<td>38.5</td>
<td>24.80</td>
<td>11360</td>
<td>2643</td>
<td>0.28</td>
<td>NO</td>
<td>1.08</td>
<td>0.28</td>
<td>0.0466</td>
<td>13.38</td>
<td>2643</td>
</tr>
<tr>
<td>X05</td>
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<td>49</td>
<td>23.55</td>
<td>27.72</td>
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<td>NO</td>
<td>0.52</td>
<td>0.21</td>
<td>-</td>
<td>19.87</td>
<td>11360</td>
</tr>
<tr>
<td>X06</td>
<td>472.5</td>
<td>52.7</td>
<td>24.33</td>
<td>26.39</td>
<td>583</td>
<td>1.85</td>
<td>YES</td>
<td>2.33</td>
<td>0.385</td>
<td>0.0858</td>
<td>9.43</td>
<td>93</td>
</tr>
</tbody>
</table>

\( V_{L,L,0} \) is the initial value of the load point displacement upon loading and before zero reset;

\( V_{L,L,i} \) is the \( V_{L,L} \) value at turning point, e.g. at crack and time initiation;

\( \Delta r_{\text{tot}} \) is the total computed blunting /3/;

\( r_{\text{creep}} \) is the creep zone at crack tip radius /3/;

\( t_1 \) is the values of the initiation time taken at turning points;

\( \Delta r_{\text{meas}} \) is the total metallographically measured blunting.

TABLE 3 - Correlation \( \sigma_{\text{net}} \) / phenomena extension.

<table>
<thead>
<tr>
<th>Spec.</th>
<th>Incubation.</th>
<th>( \sigma_{\text{net}} )</th>
<th>Radius</th>
<th>Cavitation</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>time (h)</td>
<td></td>
<td>deform.zone</td>
<td>depth</td>
<td></td>
</tr>
<tr>
<td>X01</td>
<td>-</td>
<td>295.4</td>
<td>-</td>
<td>-</td>
<td>No propagation</td>
</tr>
<tr>
<td>X04</td>
<td>-</td>
<td>350</td>
<td>-</td>
<td>-</td>
<td>No propagation</td>
</tr>
<tr>
<td>X05</td>
<td>500</td>
<td>396</td>
<td>-</td>
<td>2 grains</td>
<td>Reload test</td>
</tr>
<tr>
<td>X06</td>
<td>93</td>
<td>472.5</td>
<td>1 mm</td>
<td>5-10</td>
<td>&quot;</td>
</tr>
<tr>
<td>X03</td>
<td>75</td>
<td>517</td>
<td>1.2mm</td>
<td>10-20</td>
<td>Propagated</td>
</tr>
<tr>
<td>X02</td>
<td>80</td>
<td>525.6</td>
<td>2 mm</td>
<td>-</td>
<td>Prop. early int.</td>
</tr>
</tbody>
</table>

Fig. 1 - Creep Crack Growth Rates versus C*-Integral.
Fig. 2 - Specimen X03.
Microhardness vs distance from the fracture surface.

Fig. 3 - Specimen X03.
Microhardness vs crack propagation.

Fig. 4 - Specimen X03. Crack propagation profile.

Fig. 5 - Specimen X06, Type wedge cavitation.

Fig. 6 - Specimen X04. Intergran precipitation.
Fig. 7 - Specimen X05.
Intergranular fracture.

Fig. 8 - Specimen X03.
Ductile fracture.

Fig. 9 - Specimen X03.
Dislocations in the unloaded position.

Fig. 10 - Specimen X03.
Dislocations in the position near the fracture surface.