Morphology of the creep crack tip in P92 steel and its relation to microstructure

V A Yardley¹, T Matsuzaki², R Sugiura², A T Yokobori Jr.², S Tsurekawa⁴, Y Hasegawa³

¹Kumamoto University Graduate School of Engineering, Kurokami-machi 2-chome-39-1, Kumamoto 860-8555, Japan
²Tohoku University Graduate School of Engineering, Aramaki Aoba 6-6-01, Aoba-ku, Sendai 980-8579, Japan
³Steel Research Laboratories, Nippon Steel Corporation, Shintomi 20-1, Futsu, Chiba 293-8511, Japan
yardley@kumamoto-u.ac.jp

Abstract. The region around the tip of a creep crack obtained using an interrupted test on a C(T) specimen of the 9 wt.% Cr power plant alloy P92 has been examined using electron backscatter diffraction to study the relationship between the crack morphology and the martensitic substructure. Nucleation of voids was found to occur primarily on prior austenite grain and packet boundaries. This agrees well with previous results in which minimum creep rate decreased with increasing prior austenite grain size for small prior austenite grains. Growth and coalescence of voids to form a continuous crack appears to occur by recovery, recrystallisation and eventual rupture of the bridging regions between the voids.

1. Introduction
Ferritic 9-12 wt.% Cr heat-resistant steels, such as the W-containing 9 wt.% Cr alloy P92 (NF616) [1,2] have a tempered lath martensitic microstructure. Martensite is formed by a diffusionless transformation from the parent austenite, and has a fixed crystallographic relationship with the austenite. However, because of crystal symmetry, a number of equivalent orientational variants satisfy this relationship and, while the prior austenite grain boundary network is conserved, each prior grain breaks up into substructures containing some or all of the allowed variants. Morphologically, the microstructure consists of highly dislocated laths, around 0.5 μm in width. Laths of the same, or closely related, orientational variant are grouped into elongated, straight-sided blocks, and blocks with parallel long axes occur together in regions called packets, within a prior austenite grain (PAG) [3-6].

This microstructure, together with the fine carbides formed after tempering, contributes to the long-term creep resistance of these steels under service conditions [7,8]. However, exposure to high temperatures and stresses can lead to the nucleation and growth over time of voids and cracks.

Safe plant operation thus requires accurate prediction of safe component lives under operating conditions and the choice of appropriate inspection intervals. The compact tension C(T) specimen with side groove is a standard geometry used in the determination of creep crack growth rates [9]. A C(T) specimen of P92 steel with fine PAG (~30 μm diameter) has been examined using optical microscopy and SEM after testing to failure at 873K in air with a constant load of 311.8 MPa [10]. Creep curves measured during this test are shown in figure 1. The fracture surface showed a dimpled appearance characteristic of ductile fracture, and in a cross-section parallel to the tensile axis and direction of
crack growth, the surface had a zig-zag or toothed shape, with the width of one 'tooth' approximately 500 μm. Lines of voids were observed running parallel to the local inclination of the crack surface and extending beyond the turning points of the zig-zags.

In the present work, the creep test on fine-PAG P92 has been repeated but interrupted part-way through to obtain a metallographic specimen containing a growing crack. Using electron backscatter diffraction (EBSD), which enables mapping of the crystal orientation as a function of position [11], the relationship between the morphology of the crack and voids and the martensitic substructure, in terms of packets, blocks and prior austenite grains, has been investigated.

2. Experimental

The composition of the P92 steel is given in table 1. The material was normalised for 2 hours at 1343K and tempered for 2 hours at 1053K.

The creep tests were interrupted after 300 hours; it can be seen from figure 1(b) that at the point of interruption, marked with a round dot, the sample is in the steady-state creep regime. The area around the crack tip was sectioned in a plane parallel to the tensile axis and crack growth direction and prepared for EBSD observation using a final stage of polishing with 0.05 μm colloidal silica for around 2 hours [12]. A sample of the as-received, untested material was also observed using EBSD for comparison.

3. Results

3.1. As-received sample

As a result of the orientation relationship between martensite and its parent austenite, the martensitic variants also have fixed orientation relationships with one another, leading to a characteristic distribution of misorientation angles. The high-angle part of the misorientation angle distribution obtained using EBSD for the as-received sample is shown in...
A large peak can be observed at 60º and a smaller one at 54.5º, with a trough in between. The smaller peak is displaced by about 5º from the position it would take if the ideal Kurdjumov-Sachs orientation relationship were exactly obeyed.

This misorientation angle distribution for the as-received specimen can be used for comparison to test whether recovery or recrystallisation have occurred in different regions of the interrupted test specimen.

### Table 1. Composition of P92 sample, in mass %.

| C    | Si  | Mn | P  | S  | Cr | Mo | W  | Nb | V  | N  | B  |
|------|-----|----|----|----|----|----|----|----|----|----|----|
| 0.09 | 0.16| 0.47| 0.01| 0.001| 8.72| 0.45| 1.87| 0.06| 0.21| 0.05| 0.002|

3.2. Interrupted sample
The area around the crack tip in the interrupted test specimen can be divided into three regions according to microstructure and crystallographic characteristics.

**Region I:** Void nucleation region. Small voids of irregular size are found. Figure 3 is an EBSD image quality (IQ) map of the void nucleation region, with the boundary of a prior austenite grain and the boundaries of the packets within it, as determined from crystallographic analysis, marked with white and black lines respectively. It can be seen that the majority of voids are located on the prior austenite grain boundary, with some others on packet boundaries and a few within packets. The misorientation profiles from both the bulk and the areas in the immediate vicinity of the voids are unchanged from that for the as-received sample. This suggests that the voids have formed without any gross microstructural recovery or plastic deformation.

**Region II:** Void growth and bridging region. Large, circular voids are present. Some of these, from their shape, are clearly made up of two or more small voids which have coalesced. The voids are surrounded with layers of fine, equiaxed grains. Between the voids are bridging ligaments exhibiting grain rotation, as demonstrated by the crystal orientation map, and recovery and recrystallisation, as can be seen from the map of “image quality”, characterising the clarity of the electron backscatter diffraction pattern. High-angle misorientation profiles from areas immediately around the voids show a loss of the characteristic two-peak martensitic profile.

**Region III:** Continuous crack region: Voids have coalesced to form a continuous crack extending from the edge of the sample. The surfaces of the crack are covered with a layer of recovered or recrystallised material, as demonstrated by the high-angle misorientation profile, but away from the immediate vicinity of the crack, the misorientation profile is similar to that of as-received material. Lines of 'failed' voids, which did not form a successful crack, are also present extending beyond the zig-zags of the continuous crack.

4. **Discussion and conclusion**

From the results presented above, a mechanism of fracture can be proposed. As a result of the specimen geometry, bands oriented at 45º to both the tensile axis and the overall crack growth direction experience the highest stress and have the greatest propensity for void formation. Voids nucleate mostly at prior austenite grain boundaries, with some at packet boundaries and within the
packets. Nearer the crack tip, voids grow and coalesce by recovery, recrystallisation and eventual rupture of the bridging ligaments between them. This leads to the observed crack with a layer of recovered or recrystallised grains on its surface and the dimpled appearance of the fracture surface.

The zig-zag morphology is formed by nucleation and growth of lines of voids both above and below the plane of the crack. Out of the two lines of voids, the one in which bridging occurs first becomes part of the crack. The other remains as one of the observed 'failed' lines of voids. Zig-zags change direction after extending far enough away from the crack plane that they no longer experience sufficient stress for void coalescence.

It has been found experimentally in CrMoV martensitic steels that the minimum creep rate decreases with increasing PAG diameter for small PAG diameters [15]. The present results, showing void nucleation occurring on PAG boundaries, are consistent with this, since larger PAG give fewer possible void nucleation sites per unit volume. In addition, it has been found in plain-carbon steels that the packet size increases with increasing PAG size [16], so the number of available packet boundary sites for void nucleation will also decrease as PAG size increases. However, the relationship between PAG size and minimum creep rate breaks down for PAG diameters > 50 μm [15], and increasing the PAG diameter beyond a critical value leads to the onset of brittle cracking along PAG boundaries [15,17]. Recent work suggests a similar dependence of fracture mode on PAG size in 12 wt.% Cr P122 steel [18]. Further EBSD observations of cracked 9-12 wt.% steel specimens with various thermal histories and thus different PAG sizes may help to test the existence of such a transition and, if it does exist, determine the critical PAG size corresponding to the transition and its dependence on such factors as composition and test temperature and stress.

Acknowledgements
The authors acknowledge the 129 committee of the Japan Society for the Promotion of Science (chairman: Prof. T. Yokobori) for support of this work and provision of samples.

References
[1] Nippon Steel Technical Report No. 50, 1991
[2] ASTM Standard A335M - 06 Standard Specification for Seamless Ferritic Alloy-Steel Pipe for High-Temperature Service, 2006
[3] Marder A R and Krauss G 1969 Trans. ASM 62 957
[4] Maki T, Tsuzaki K and Tamura I 1980 Trans. ISIJ 20 207-214
[5] Morito S, Tanaka H, Konishi R, Furuhara T and Maki T 2003 Acta Mater. 51 1789-1799
[6] Kitahara H, Ueji R, Tsuji N and Minamino Y 2006 Acta Mater. 54 1279-1288
[7] Maruyama K, Sawada K and Koike J 2001 ISIJ Int. 41(6) 641-653
[8] Masuyama F 2001, ISIJ Int. 41(6) 612-625
[9] ASTM E1457-07: Standard Test Method for Measurement of Creep Crack Growth Times in Metals, 2007
[10] Matsuzaki T, Sugiuera R, Yokobori A T and Yardley V A 2008 EBSD study of effect of material microstructure in limiting creep crack growth in high-Cr heat-resistant steels Japan Institute of Materials Autumn Meeting, Kumamoto, Japan
[11] Adams B L, Wright S I and Kunze K 1993 Metall. Trans. A 24A 819-831
[12] Nowell M W, Witt R A and True B W 2005 Microscopy Today July 44-48
[13] Kurdjumow G and Sachs G 1930 Z. Physik 64 325-343
[14] Wright S I and Nowell M M 2006 Microsc. Microanal. 12 72-84
[15] Kimura K, Ohi N, Shimazu K, Matsuo T, Tanaka R and Kikuchi M 1987 Scripta Metall. 21 19-22
[16] Morito S, Yoshida H, Maki T and Huang X 2006 Mater. Sci. Eng. A 438-440 237-240
[17] Hong S and Yu J 1989 Scripta Metall. 23 1057-1062
[18] Sugiuera R, Yokobori A T, Takamori S, Tabuchi M, Fuji A, Yoda M, Kobayashi K, and Yokobori T 2007 Mater. Trans. 48 2928-2936