Relation between Creep Rupture Strength and Substructure of Heat Resistant Steel

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Creep rupture tests were performed at around 650°C on a low carbon steel, a W-bearing low carbon steel, a low carbon steel containing 0.1% of TaN and a 0.1%C–8%Cr–2%W–0.2%V–0.04%Ta steel (F-82H), structural material for fusion reactors. The equivalent obstacle spacing for mobile dislocations is calculated using only rupture data according to the creep theory developed by some of the authors. Thin films taken from the ruptured specimens were examined under a transmission electron microscope (TEM). The observed sub-grain size or the calculated inter-particle distance roughly coincides with the equivalent obstacle spacing. This indicates that the microscopic variable, i.e. the obstacle spacing for mobile dislocations, such as sub-grain size or inter-particle distance, can be directly calculated from only the macroscopic variable, i.e. time to rupture or creep rate.

KEY WORDS: creep theory; sub-grain; precipitation.

1. Introduction

Many creep equations have been developed, where the variables such as creep rate, time to rupture, temperature, stress, grain size, sub-grain size, dislocation density and so on are correlated with each other. Creep phenomena of metals and alloys have been well understood qualitatively based on these equations. In contrast, to understand creep phenomena microscopically, a concept of effective stress, i.e. applied stress minus internal stress, has been discussed for a long time. Equivalent stress to the internal stress is sometimes called athermal stress, threshold stress, frictional stress, or back stress. The internal stresses have been explained in terms of microstructural parameters. These stresses are usually measured by changing the applied stress in a steady state creep range, however the problems on measuring the internal stress have been discussed elsewhere. One of the merits of the introduction of the effective stress is that the stress exponent for the effective stress decreases to the theoretical values, i.e. 3–5, while the stress exponent for the applied stress is very large, e.g. larger than 10, in some cases.

A creep equation should describe the relation between creep rate and microstructural parameters, because creep rate is clearly affected by microstructure. In other words, it is expected to calculate an internal stress or a microstructural parameter from the observed creep rate directly. Certainly, for some dispersion hardened alloys which show threshold stress, \( \sigma_{th} \), in an applied stress, \( \sigma \), vs. creep rate, \( \dot{\epsilon} \) diagram, \( \dot{\epsilon} = \dot{\epsilon}_0 (\sigma - \sigma_{th})^m \), even though the data are limited within a short time, where \( \dot{\epsilon} \) and \( m \) are a constant and an effective stress exponent, respectively. If \( \sigma_{th} \) is assumed to be an Orowan stress, we can estimate an inter-particle distance from \( \sigma_{th} \). However, in general, to calculate an internal stress using the existing creep equations of a power law type is impossible, because for many cases threshold stress does not observed in a \( \sigma-\dot{\epsilon} \) diagram. This means that internal stress is changing during creep or creep equations concerning internal stress have not yet been complete.

For these circumstances, some of the authors have developed a new creep concept and related equations which can predict the equivalent obstacle spacing for mobile dislocations using only time to rupture. A previous work reported that an average inter-particle distance of a ferritic steel containing small amount of TaN measured using TEM roughly coincided with the equivalent obstacle spacing calculated from rupture data.

To control the distribution of fine particles like TaN is very important for developing superior heat resistant steels. Therefore, it is significant to develop further the previous work.

In this study, further creep rupture tests for the above mentioned TaN containing steel together with the reference steels have been conducted and the sub-grain size and/or the particle size are measured using TEM and the results are compared with the equivalent obstacle spacing calculated from the rupture data.

2. Equivalent Obstacle Spacing

The new theory and related equations have not been so general, an essence of the theory has been reviewed as follows. According to the new theory time to rupture, \( t_r \), is
expressed as

\[ t_i = t_{i0} \exp \left( \frac{Q - \sigma V}{RT} \right) \] ........................(1)

where \( \sigma, T, R, Q, V \) and \( t_{i0} \) are the applied stress, the testing temperature in K, the gas constant, the activation energy in the absence of the applied stress (apparent activation energy for simplicity), the activation volume and the material constant, respectively. The followings are assumed in introducing Eq. (1): (i) steady state creep rate is controlled by dislocation creep, (ii) dislocation velocity is controlled by a thermally activated process and (iii) the Monkman–Grant (M-G) relation is valid.\(^{(15)}\)

Equation (1) has been applied to the rupture data beyond 100 thousands hours of many heat resistant steels and all of the data plotted in a log \( t_i \) vs. \( \sigma \) diagram for each steel have been explained by several number of straight lines.\(^{(14)}\) Moreover, physical meanings of the material constants of Eq. (1) have been discussed in the previous work.\(^{(15)}\)

It is well known that the apparent activation energy is close to the activation energy of self-diffusion for many pure metals.\(^{(1)}\) However, for many practical heat resistant steels the values of \( Q \) which are calculated by Eq. (1) or the conventional power law equations are known to be in either case much larger than the activation energy of self-diffusion of the matrix.\(^{(3,14,18)}\) The differences are mainly caused by elastic interactions between mobile dislocations and strengthening factors, such as sub-grain boundaries, sessile dislocations, precipitations and so on. Spacing of these obstacles for mobile dislocations is denoted as \( d \). A dislocation approaches to the obstacles under the residual and/or applied stress and is pinned due to the elastic interactions and the dislocation does not contribute to macroscopic creep deformation. Subsequent dislocations have also elastic interactions with not only the obstacles but also the pinned dislocations. That is, the pinned dislocation becomes a new obstacle to the subsequent mobile dislocations. When the interaction energy becomes the maximum under the applied stress, the mobile dislocations climb the obstacles followed by gliding a considerable distance and the mobile dislocations contribute to macroscopic creep deformation.

Among the elastic interactions, we assume the interaction energy, \( Q_{\text{int}} \), between a mobile dislocation with an edge character and the obstacle dislocation with an edge character as a maximum interaction. Then, \( Q \) can be rewritten as

\[ Q = Q_d + Q_{\text{int}} \left( 1 - \frac{d \ln \mu}{d \ln T} \right) \] ........................(2)

where \( Q_d \) is the activation energy for self-diffusion of the matrix, \( \mu \) is the shear modulus of the matrix at a testing temperature and a parentheses of the second term means the correction due to the temperature dependence of the shear modulus.

The obstacle spacing, \( d \), can be expressed as

\[ \frac{d - \frac{\mu b}{\pi (1 - \nu)} \ln \left( \frac{d}{2r_0} \right)}{V} \] ........................(3)

where \( p \) is an adjustable parameter, \( b \) is the length of the Burger’s vector, \( \nu \) is the Poisson’s ratio and \( r_0 \) is the distance between the nearest neighbor atoms. A model proposed is based on an elastic interaction between a straight mobile edge dislocation and a straight obstacle dislocation with an edge character. However, in many cases dislocations observed are wavy or tangled and the pinning force depends on characters of the obstacle particles, such as diameter and misfit parameter. Therefore, an adjustable parameter, \( p \), is needed. If an obstacle is small and/or it has a weak interaction with a dislocation, \( p < 1 \). And when an obstacle dislocation has a pure edge character, \( p = 1 \). \( p = 0 \) for a pure screw character, that is, obstacle with a screw character does not affect a mobile dislocation. Therefore, \( \Delta d/p \) in Eq. (3) is an effective obstacle spacing or equivalent to the obstacle spacing for mobile edge dislocations and we denoted \( \Delta d/p \) as equivalent obstacle spacing. \( \Delta d/p \) can be calculated analytically from Eq. (3), assuming that \( d \) in logarithm of Eq. (3) is constant, \( 1 \mu \). The values of \( \Delta d/p \) calculated using Eq. (3) of the Cr–Mo steels roughly coincide with the inter-particle spacing of the precipitates observed under TEM.\(^{(15)}\) It is also reported in the previous work\(^{(15)}\) that the values of \( \Delta d/p \) of a low carbon steel were calculated using a few independent data within a narrow range of temperature and stress and the values were analyzed as a function of time and temperature and, as a result, the precipitation of Mo\(_2\)C during creep was predicted.

### 3. Experimental Procedure

Top three experimental steels with chemical compositions listed in Table 1 were melted in a 10 kg vacuum induction furnace. The ingots were hot-rolled to 12 mm thickness, where the final pass was made at 1000°C. FETA1 is a pure iron and is a reference material. FETA2 is ultra low carbon steel containing about 0.1% TaN. FETA4 is intended to contain a small amount of W, the value of which corresponds to the maximum solubility of W at 650°C. According to Thermo-Calc, a calculation system of thermodynamic data,\(^{(19)}\) in FETA4 any compound such as Laves phase, WC and Fe\(_2\)C does not precipitate at 650°C, around which temperatures creep rupture tests were performed.

The hot-rolled plates were normalized at 1000°C for FETA1 and FETA4 and at 1200°C for FETA2, at which temperature TaN is dissolved in the matrix.\(^{(20)}\) In order to complete the precipitation of TaN and to relieve the residual stress, FETA1, 2 and 4 are aged at 650°C for 500 h be-

| Steel | C   | Si  | Mn  | P   | S   | Cr  | V   | Al  | Ta  | W   | N   | O   | Fe  |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| FETA1 | 0.0031 | 0.008 | 0.076 | <0.002 | 0.0010 | na  | na  | 0.020 | na  | na  | 0.0019 | 0.0010 | bal |
| FETA2 | 0.0028 | 0.008 | 0.074 | <0.002 | 0.0007 | na  | na  | 0.030 | 0.093 | na  | 0.0130 | 0.0011 | bal |
| FETA4 | 0.0026 | 0.077 | 0.092 | <0.002 | 0.0011 | na  | na  | 0.027 | 0.031 | 0.0018 | 0.0010 | bal |
| F-52H | 0.598 | 0.10 | 0.15 | 0.003 | 0.003 | 7.71 | 0.18 | 0.005 | 0.04 | 2.1 | 0.0043 | 0.0028 | bal |

| na | not analysed | bal | balance |

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**Table 1.** Chemical composition (mass%) of the experimental steels.
F-82H is originally a hot rolled plate of 15 mm, which was manufactured in a semi-production scale using a 5 ton ingot aiming for use in fusion reactors. The hot rolled plate was re-normalized at 1200°C for 30 min and tempered at 800°C for 2 h.

TEM discs were taken from a uniformly deformed portion of the ruptured specimens. Thin films were prepared in 2% perchloric acid–methanol using a single jet polishing apparatus. TEM observations were conducted on JEOL JEM-2010 and Hitachi HF 2000 operated at 200 kV.

4. Results and Discussion

4.1. Creep Properties

Figure 1 shows creep curves of FETA1 and FETA4. At 600°C accelerate creep is observed after a considerable amount of transition creep, but at 650°C transition creep is hardly observed for both steels. Rupture life of W-bearing steel, FETA4, is little longer than that of FETA1 at 650°C, however, creep behavior of both steels is quite similar.

Figures 2 and 3 show creep curves and $\varepsilon-\dot{\varepsilon}$ curves of FETA2 and F-82H, respectively. Creep rate of a tempered martensitic steel with a small amount of M$_{23}$C$_{6}$ and TaC, F-82H, is accelerated after about 2% of transition creep. On the contrary, a ferritic steel with a small amount of TaN, FETA2, shows only a small amount of transition creep. The difference should be explained by the recovery of martensite in F-82H during transition creep.

4.2. Regression Analyses and Rupture Strength

Figure 4 shows a stress vs. time to rupture diagram for the experimental alloys. Although usually logarithm of time to rupture is plotted against logarithm of stress, in Fig. 4 the applied stress is plotted on a linear scale according to the new creep theory.

Regression analyses were made using Eq. (1) on the rupture data, assuming that material constants of Eq. (1), $\dot{\varepsilon}$, $Q$ and $t_{0}$, are independent of testing temperature, stress and time. The calculated material constants and the correlation coefficients, $r$, are listed in Table 2. The high values of the correlation coefficient show that the material constants calculated are highly reliable. Using these constants the rupture strengths for a given time were estimated. The results were also listed in Table 2. In the table the estimated values were omitted when $\dot{\varepsilon}/RT\leq 1$, because Eq. (1) is invalid. In Table 2, $C$ is defined as $C = -\log t_{0}$ and the constant, $C$, corresponds to the well-known Larson–Miller constant.

Strictly speaking, the material constants of Eq. (1) are varying with temperature, stress and time. However, the
linear relationships have been well established for many heat resistant steels by plotting in the semi-logarithmic diagram the creep rupture data of widely ranged test conditions, which indicates that the values of \( Q, V, C \) of the practical heat resistant steels can be regarded as constant for a wide range of test conditions.\(^{14}\) Moreover, the deformation mechanism should be the same for specimens tested under the narrow ranging temperature and stress. Therefore, it is reasonable to assume that in this study \( Q, V, C \) are independent on temperature, time and stress.

Yield strengths at 650°C of F-82H and a low carbon steel corresponding to FETA1 are 300\(^{25}\) and 80 MPa,\(^{26}\) respectively. Yield strengths of FETA2 and 4 are not yet available. However, Vickers hardness numbers, HV10, of FETA 1, 2, 4 and F-82H are 66, 99, 73 and 168, respectively, and if the yield strength at 650°C is proportional to the hardness number at room temperature, the yield strengths of FETA2 and 4 can be estimated as 200 and 145, respectively. Thus, the creep tests were judged to be done below the yield strength for each steel. In addition to the fact that the creep tests were done within a narrow range of test conditions, this leads that the material constants, \( Q, V, C \), are expected to change little during the creep test.

The values of \( Q \) for FETA1 and FETA4 are little larger than the activation energy for self-diffusion of Fe.\(^{27}\) The values of \( Q \) for the other steels strengthened by Ta are much larger than those of FETA1 and FETA4. The values of \( C \) for the experimental steels are ranging from 15 to 35 and are varying similarly with the trend of \( Q \). The values of \( V \) for FETA1, 2 and 4 are much larger than that of F-82H. These material constants listed in Table 2 seem to be rather larger or smaller than the values determined from the long term rupture data,\(^{14}\) but it should be reminded that the constants are obtained through the short time creep tests.

Comparing the rupture strengths of FETA1 with those of FETA4, the effect of solid solution strengthening by W in ferrite is found to be clear at rather low temperature and short time, but not so remarkable at around 650°C. It is well known that the addition of a small amount of Mo in a carbon steel, \( i.e. \) the same level comparing with the W content of FETA1 in at%, increases rupture strength remarkably.\(^{28,29}\) However, testing temperature of these literature data is rather low, \( i.e. \) 400–500°C, and N content of each steel examined is much larger than that of FETA4. The latter causes the interaction between interstitial and substitutional atoms,\(^{30}\) which is responsible for the difference in the Mo/W effects between the literature data and FETA4. Extrapolated rupture strengths of FETA1 at 650°C are the same or higher than those of FETA4. This is not an essential behavior, but this may be caused by the character of data, \( i.e. \) the higher strength of FETA4 at low temperatures and shorter time.

On the other hand, rupture strengths of Ta-containing steels, FETA2 and F-82H, are much higher than those of the reference steel, FETA1. FETA2 is strengthened by TaN\(^{30}\) and F-82H is strengthened by TaC besides martensite and M\(_{23}\)C\(_6\).\(^{30,31}\) Comparing the data of FETA2 with F-82H, it is probable that \( t_{r} \) of FETA2 exceeds that of F-82H at a low stress level, though the number of data is limited, because the slope of the stress vs. time to rupture relation of F-82H is much larger than that of FETA2. FETA2 is aged at 650°C for 500 h before the tests, so that the new precipitation of TaN during the test can be neglected. Though TaC particles in F-82H are stable for a long time,\(^{30}\) the amount TaC is less than the amount of TaN in FETA2, so that the inter-particle distance of TaC is larger than that of TaN as mentioned later (Table 4). Besides this fact, the recovery of martensite and coarsening of M\(_{23}\)C\(_6\) are responsible for the predicted reversal of the rupture strength at a longer time.

### Table 2. Material constants of Eq. (1) and the estimated rupture strengths (MPa).

| Steel | \( Q \) (kJ/mol) | \( V \) (cfs) | \( C \) | \( \sigma \) (MPa) at 600°C | \( \sigma \) (MPa) at 650°C |
|-------|------------------|--------------|--------|-----------------|-----------------|
|       |                  |              |        | 100 h | 1000 h | 10000 h | 100 h | 1000 h | 10000 h |
| FETA1 | 245              | 1500         | 15.5   | 98.5 | 27.6 | 19.3 | 10.0 | 18.8 | 9.5 | invalid |
| FETA2 | 818              | 2078         | 35.8   | 99.6 | 89.6 | 81.5 | 73.5 | 72.2 | 63.7 | 55.2 |
| FETA4 | 342              | 1569         | 15.7   | 99.5 | 29.3 | 18.7 | 8.0  | 18.5 | 7.2  | invalid |
| F-82H | 682              | 804          | 32.4   | 99.7 | 132.8 | 112.9 | 91.2 | 91.8 | 69.8 | 47.9 |

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**Fig. 5.** TEM micrograph of ruptured FETA1 tested at 650°C under 20.8 MPa (\( t_{r} = 57.6 \) h).
Figures 8 and 9 show TEM micrographs of ruptured FETA2. The test was made at 650°C under 76.3 MPa ($t_r = 32.2$ h and rupture elongation = 18.3%). The finer sub-grains than those of FETA1 are shown in Fig. 8. In a sub-grain tangled dislocations with fine particles are seen (Fig. 9). In the previous work, it was reported that the fine particles were TaN and the average diameter was 13 nm. Besides these fine particles, rectangular-shaped particles with 77 nm $\times$ 23 nm in average were observed frequently and the X-ray analysis on the extracted residues revealed that only TaN is a major constituent. Therefore, these coarse precipitates were considered to be un-dissolved TaN particles.

F-82H shows tempered martensite structure with the prior austenite grain size of 88 $\mu$m. Schäublin et al. have already presented a typical TEM image of F-82-H as normalized and tempered. They observed lath martensite structure with dislocation density of $0.86 \times 10^{14}$ m$^{-2}$. In the similar alloy with F-82H it is reported that high dislocation density decreases rapidly with increasing creep strain up to minimum creep rate and after the minimum creep rate dislocation density within lath martensite does not change largely and cell structure is gradually formed. Figure 10 shows a TEM micrograph of ruptured F-82H. The test was
made at 650°C under 78 MPa (τc=446.7 h and rupture elongation=18%). The finer sub-grains or cell structure than those of FETA1 and 4 are shown in Fig. 10. In the steel M23C6 and TaC are formed in the sub-grains and/or on the sub-grain boundaries, lath boundaries or cell boundaries and the size of M23C6 of the normalized and tempered state is ranging from about 0.05 to 0.6 μm.30,31) TaC particles could not be distinguished clearly in the thin films from the other contrast, because of the existence of both M23C6 particles and bend contour lines in addition to the small size of TaC. However, the previous work30) reported that the average size of TaC extracted on a replica film is 27 nm in the same steel of the normalized tempered state and the size does not drastically change even after the thermal aging at 650°C for 30 000 h.

4.4. Comparison of d/p with Substructure

(1) Sub-grain Size

Sub-grain size is measured as the average linear intercept on the TEM micrographs. The results are shown in Table 3. In the calculation both grain-boundaries and lath boundaries are treated as sub-grain boundaries. The observed sub-grain sizes of FETA1 and FETA4 are large as compared with those of the other steels hardened by precipitated particles.

It is well known that the sub-grain size is proportional to the inverse applied stress independent of testing temperature.1,2) At the same time, the sub-grain size is known to be varying largely specimen-by-specimen. Kadoya and Shimizu35) summarized the sub-grain sizes of pure iron and α-iron containing solute atoms and precipitates of an NaCl type (MX) including literature data and presented a sub-grain size–stress diagram which can be read as

\[
\log(d_s) = 1.87 - 0.8\log(\sigma)
\]

where \(d_s\) is the sub-grain size in μm and \(\sigma\) is the applied stress in MPa.

The sub-grain size can be calculated using Eq. (4). In Table 3 the observed sub-grain size and the calculated values for the average creep stress are listed. These coincide roughly with each other. Strictly speaking, the data listed in Table 3 may be an occasional coincidence, because an equation like Eq. (4) is generally valid for steady state creep or minimum creep rate, on the other hand, the observed sub-grain sizes were measured using the ruptured specimens. Moreover, it is reported27,33–35) that the sub-grain size sharply changes with increasing creep strain until minimum creep rate occurs and, then, gradually increases with increase in creep strain and the sub-grain size of a near rupture specimen is factor of 5 larger than that for the minimum creep rate in a maximum case and below factor of 2 larger in a minimum case, which depends on material. However, Table 3 shows that the calculated values of \(d_s\) are fairly correlating with the observed sub-grain size and the difference between the observed sub-grain size and the calculated values is only about 50%. Therefore, Eq. (4) is judged to be valid for evaluating the sub-grain size, \(d_s\), corresponding to the steady state creep of a experimental steel tested under the average stress.

(2) Inter-particle Distance

The inter-particle distance of TaN in FETA2 is calculated using the observed average particle size, 13 nm.16) In the calculation it is assumed that (i) all of Ta in the steel form fine TaN, (ii) the existence of the rather large TaN of several 10's nm in size is ignored and (iii) density of TaN is 14.5 Mg/m³. The result, 0.13 μm, is listed in Table 4. The value is smaller than the inter-particle distance obtained as the linear intercept on TEM micrographs, 0.55 μm.16)

In the above calculation the coarse TaN particles were ignored. The area fraction of the coarse particles is only about 20%. Even if it is taken into account, the inter-particle distance of TaN in FETA2 increases only about 10%. As mentioned above it is very difficult to measure separately the inter-particle distances of TaC and M23C6 of F-82H under TEM. Therefore, the inter-particle distances of F-82H are calculated in the similar manner for FETA2. The amount of precipitated M23C6 in a normalized and tempered state is assumed as 1.96% using the thermodynamics data calculation system19) and the average size of M23C6 is 130 nm.28) It is reported that the average particle size is 0.27 nm

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### Table 3. Comparison of the observed sub-grain size for the rupture specimens with the calculated values.

| Steel | Creep stress Mpa | Observed sub-grain size μm | Sub-grain size Calculated using Eq.(4) μm | Ratio (Observed/Calculated) |
|-------|-----------------|-----------------------------|------------------------------------------|-----------------------------|
| FETA1 | 20.8            | 9.5                         | 6.5                                      | 1.45                        |
| FETA2 | 76.3            | 1.4                         | 2.3                                      | 0.61                        |
| FETA4 | 20.8            | 7.1                         | 4.8                                      | 1.48                        |
| F-82H | 78.0            | 3.8                         | 2.3                                      | 1.54                        |
| Av.   |                 |                             |                                          | 1.27                        |

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### Table 4. Characteristics of microstructure and equivalent obstacle spacing.

| Alloy | Hardness HV10 | precipitate | Amount of precipitation mass% | Average particle size nm | Average inter-particle distance(μm) | Calculated sub-grain size using Eq.(4) μm | Equivalent obstacle spacing d/p μm | Time to rupture | min. creep rate |
|-------|---------------|-------------|-------------------------------|--------------------------|-------------------------------------|------------------------------------------|-----------------------------------|-----------------|-----------------|
| FETA1 | 66            | trace       | 0.1%                          | 13 (3)                   | 0.13                                | 2.3                                      | 1.85                              | 1.95            | 1.24            |
| FETA2 | 99            | TaN         | 0.1(1)                        | 27 (2)                   | 0.41                                | 2.0                                      | 0.18                              | 0.14            |                 |
| FETA4 | 73            | trace       | 0.2(2)                        | 130 (4)                  | 0.37                                |                                          |                                    |                 |                 |
| F-82H | 168           | TaC         | 0.027(2)                     | 1.98(2)                  | 0.37                                |                                          |                                    |                 |                 |

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(1) Estimated from chemical composition
(2) Reference No. 31
(3) Calculated from the amount of precipitation and the average size
(4) Reference No. 30
(5) Calculated from the amount of precipitation and the average size

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and the amount of Ta in the extracted residues of the normalized and tempered state is chemically analyzed as 0.025% and the value was converted into the amount of TaC as 0.027%. The results are listed in Table 4. The inter-particle distances of TaC and M23C6 are just comparable and are 0.41 and 0.37 μm, respectively.

(3) Equivalent Obstacle Spacing and Substructure
The equivalent obstacle spacing, d/p, is calculated using Eq. (3) and rupture data for each steel. The results are listed in Table 4. In the calculation it is assumed that a single deformation mechanism is operating for all tests shown in Fig. 4. The values of d/p of FETA1, FETA4, FETA2 and F-82H are large in the order of magnitude, but the values of d/p of FETA1 and FETA4 are much larger than those of FETA2 and F-82H.

The calculated sub-grain size of FETA1 and FETA4 for the average stress is comparable to, but little larger than d/p for each steel. This means that residual dislocations in sub-grains may play a role of strengthening besides the sub-boundaries. The values of d/p of FETA2 and F-82H were just comparable to the average inter-particle distance rather than the calculated sub-grain size. Figure 11 shows the correlation between the equivalent obstacle spacing and substructure. It is found from the figure that for a plain low carbon steel or a W-bearing steel sub-boundary is a basic strengthening factor and for precipitation hardened steels inter-particle distance is a basic strengthening parameter.

(4) Validity of the Assumptions
From the above discussion it is clear that the calculated values of d/p that are obtained using only rupture data are quantitatively related with the typical parameters of microstructure, i.e. sub-grain size or inter-particle distance. However, further discussions are needed on the following two points: (i) these parameters may be changing during creep deformation, though the TEM observations were made on the rupture specimens and (ii) what is the reasonable value of p.

On the first point, the new theory should be true originally for the steady state, however the M-G relation is also hold for many steels, and therefore Eq. (1) is valid. It is reported that the most important change in microstructure occurs during transient creep. Therefore, the values of d/p obtained should represent the microstructure corresponding to the steady state deformation, if the M-G relation is hold in the experimental steels. Figure 12 shows the relation between minimum creep rate and time to rupture. It is assumed that both steady state creep rate and minimum creep rate are the same. It is found from Fig. 12 that the linear relations are proved for each steel, though the M-G constant, minimum creep rate times time to rupture, is low for FETA2, because of the shortness of rupture elongation for this alloy. For the minimum creep rates the values of d/p can be newly calculated using the similar equations of Eqs. (1)–(3) and the results are also shown in Table 4 and Fig. 11. It is clear in Table 4 that there are no essential difference between the values of d/p obtained using the rupture data and minimum creep rate for each steel. In other word, it is surprising that the values of d/p calculated for minimum creep rate of FETA1 and 4 roughly coincide with the calculated sub-grain size corresponding to minimum or steady state creep, if the effect of residual dislocations observed in the sub-grains is taken into account.

Figure 11 shows that the values of d/p calculated from minimum creep rate or time to rupture of FETA2 and F-82H completely coincide with the estimated inter-particle distance. The inter-particle distance of FETA2 is estimated as a value corresponding to the creep deformed state. Concerning this steel the change in the inter particle distance during creep can be neglected, because the pre-aging treatment at 650°C for 500 h was made. Therefore, this coincidence is significant. Concerning F-82H, the inter-particle distance of M23C6 is estimated as a value of a normalized and tempered state. The amount of precipitates of M23C6 may not change, but the particle size should be increasing gradually, so that the inter particle distance of M23C6 may increase during creep. However, the change may be not so large, because the test duration is short. On
the other hand the inter particle distance of TaC is estimated as a value of a normalized and tempered state, however, it is reported that the size and the amount of TaC do not change during very long thermal aging. In any way, the estimated inter particle distance of FETA2 and F-82H shown in Fig. 11 are one order smaller than the sub-grain sizes. Therefore, for these steels, inter particle distance, not sub-grain size is judged to be a main strengthening factor.

On the second point, reminding the assumption of a thermal activation process it may be acceptable to neglect the resistance for mobile dislocations with screw character, because macroscopic deformation cannot be explained by only screw dislocations. Therefore, \( p = 1 \) is a reasonable assumption.

The above discussion indicates that the values of \( d/p \) calculated using the time to rupture or the minimum creep rate represent the spacing of obstacles for mobile dislocations during steady state or accelerating creep, i.e. a major portion of creep deformation and does not strongly affected by the initial condition of microstructure and, therefore, it is clear that the calculated equivalent obstacle spacing roughly coincides with the microstructural parameters, i.e. sub-grain size or inter-particle distance which control rupture life. This means that the new creep theory and the related equations are valid and applicable to the interpretation of creep of heat resistant steels.

5. Conclusion

Creep rupture tests under a constant load and microstructural observations under a transmission electron microscope were performed on a W-bearing low carbon steel, a TaN containing low carbon steel, a high chromium heat resistant ferritic steel with a small amount of TaC and \( M_23C_6 \) together with a low carbon steel and the following conclusions are obtained.

1) The equivalent obstacle spacing of \( \alpha \)-iron without precipitates is comparable with the sub-grain size which is estimated as the sub-grain size during steady state creep, but within the test conditions it is lightly smaller than the sub-grain size. Dislocations in the sub-grains may also play a role of a strengthening factor.

2) In the high chromium ferritic heat resistant steel and the TaN containing low carbon steel, the equivalent obstacle spacing is comparable with the inter-particle distance. This means that the dispersion of particles is an essential strengthening factor in these steels.

3) The creep deformation model which induces Eqs. (1)–(3) is valid for the heat resistant steels tested. This means that macroscopic variables such as time to rupture and creep rate are analytically and quantitatively correlated with the microscopic parameters such as inter-particle distance and sub-grain size through Eqs. (1)–(3).

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