Damage Assessment of Heat Resistant Steels through Electron BackScatter Diffraction Strain Analysis under Creep and Creep-Fatigue Conditions*

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Abstract

EBSDElectron BackScatter Diffraction analyses were conducted for studying the quantitative microstructural metrics of creep and creep-fatigue damage for austenitic SUS304HTB boiler tube steel and ferritic Mod.9Cr piping steel. KAMKernel Average Misorientation maps and GOSGrain Orientation Spread maps were obtained for these samples and the area averaged values KAM_{ave} and GOS_{ave} were obtained. While the increasing trends of these misorientation metrics were observed for SUS304HTB steel, the decreasing trends were observed for damaged Mod.9Cr steel with extensive recovery of subgrain structure. To establish more universal parameter representing the accumulation of damage to compensate these opposite trends, the EBSD strain parameters were introduced for converting the misorientation changes into the quantities representing accumulated permanent strains during creep and creep-fatigue damage process. As KAM values were dependent on the pixel size (inversely proportional to the observation magnification) and the permanent strain could be expressed as the shear strain which was the product of dislocation density, Burgers vector and dislocation movement distance, two KAM strain parameters $M_{eKAMnet}$ and $M_{e\delta KAMave}$ were introduced as the sum of product of the noise subtracted KAM_{net} and the absolute change from initial value δKAM_{ave} with dislocation movement distance divided by pixel size. $M_{e\delta KAMave}$ parameter showed better relationship both with creep strain in creep tests and accumulated creep strain range in creep-fatigue tests. This parameter can be used as the strain-based damage evaluation and detector of final failure.

Key words: EBSD, Creep, Creep-Fatigue, Damage, Austenitic Stainless Steel, High Chromium Steel, Strain

1. Introduction

Austenitic stainless steels and high chromium tempered martensitic heat resistant steels are widely used as key component materials to endure the severe condition of steam pressure and temperature in modern fossil power plants[1]. However those material degradation and damage accumulation processes might not be fully understood

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quantitatively according to the complex nature of microstructural change. Although the enormous efforts have been done to reveal the micromechanism of degradation and damage during creep process using Transmission Electron Microscope (TEM) or other sophisticated equipment, the quantification of the phenomena is still a challenging task[2].

Recently, Electron BackScatter Diffraction pattern (EBSD) method has been successfully applied to investigate the crystallographic characteristics of dislocation substructure and grain/subgrain morphologies[3]. Among the EBSD parameters, quantities related to misorientation could represent the change of local crystal rotation but it could not have been directly correlated with the plastic strains or creep strains.

Meanwhile, several extensive studies have been conducted on the relationships between inelastic strains and EBSD parameters, such as on the relationship between the distribution of misorientation and crystal slip [3], on the crystal deformation parameter[4] and on the effect of grain size and EBSD pixel step size [5,6].

The permanent creep or plastic strain can be interpreted in principle as the resultant quantity from the combination of dislocation density and dislocation motion. Misorientation measured by EBSD is apparently correlated with the dislocation distribution but it does not represent the mobile dislocation density itself. Moreover, EBSD parameters are inevitably affected by the observation magnification i.e. the pixel step size[7].

To establish the evaluation method of accumulated permanent strain during creep and creep-fatigue damage process, special attention was paid on the change of EBSD parameters KAM(Kernel Average Misorientation) and GOS(Grain Orientation Spread) based on experimental results. KAM based strain parameters were introduced for obtaining good correlations with the macroscopic strains both for creep and creep-fatigue damage process.

2. Experimental Procedure

2.1. Materials

Tested materials used are the plate of simulated SUS304HTB steel as austenitic stainless steel for boiler tube material and the plate of simulated Mod.9Cr steel as- tempered martensitic steel for piping material of high temperature component. Table 1 and Table 2 show the chemical composition and the tensile properties at ambient temperature.

Table 1 Chemical composition (mass %).

Material	C	Si	Mn	P	S	Cr	Mo	Ni	V	Cu	N
SUS304HTB	0.06	0.53	1.45	0.025	0.012	18.38	0.22	10.04	<0.01	0.12	0.029
Material	C	Si	Mn	P	S	Cr	Mo	Ni	V	Nb	N
Mod.9Cr	0.084	0.385	0.0375	0.10	0.001	8.34	0.89	0.28	0.018	0.074	0.048

Table 2 Tensile properties at ambient temperature.

Material	0.2% proof stress (MPa)	Tensile strength (MPa)	Elongation (%)
SUS304HTB	253	594	61
Mod.9Cr steel	526	690	29

2.2. Creep Test

Creep tests were conducted on round bar specimens using single lever type creep testing machines. Figure 1 shows the specimen geometry with 6mm diameter and 30mm gage

length. Tables 4 and 5 show the creep rupture and creep interruption test conditions with corresponding nominal creep strain for interruption test specimen and nominal creep rupture strain for ruptured specimen.

The center portions of specimens shown as hatched area were longitudinally sectioned for observation sampling. For EBSD observation, mid-sections of the specimens were polished by sand paper (No.180-No.2000) and by buff with Alumina slurry (particle size $3\mu\text{m}$ to $0.1\mu\text{m}$). Sample surface preparation was conducted up to the final polishing with colloidal silica.

Fig.1. Creep test specimen and sampling portion.

Table 4 Creep rupture and interruption test conditions for SUS304HTB.

Test temperature (°C)	Stress (MPa)	Imposed time (h)	Time fraction t/t_r	Creep strain ε_c
650	130	75.4	0.1	0.021
		166	0.22	0.086
		377.3	0.5	0.096
		616.3	0.82	0.159
		754.6	1	0.445

Table 5 Creep rupture and interruption test conditions for Mod.9Cr steel.

Test temperature (°C)	Stress (MPa)	Imposed time (h)	Time fraction t/t_r	Creep strain ε_c
600	130	471	0.1	0.008
		943	0.2	0.009
		2347	0.5	0.021
		3762	0.8	0.036
		4704	1	0.112
650	100	53.7	0.1	0.007
		108.1	0.2	0.008
		267	0.5	0.015
		427.1	0.8	0.045
		533.8	1	0.169

2.3. Creep-fatigue Test

Creep-fatigue tests were conducted on round bar specimens by strain-controlled electric-hydraulic servo testing machine with induction heater. Figure 2 shows the specimen geometry with 8mm diameter and 20mm gage length. Figure 3 shows the schematic of the stress-strain hysteresis loop for tensile strain hold wave shape where inelastic strain range $\Delta\varepsilon_{ie}$ and creep strain range $\Delta\varepsilon_c$ are defined. Tables 6 and 7 show the creep-fatigue failure and interruption test conditions with representative strain ranges measured at $N_f/2$ cycles for fractured specimens. The failure cycles N_f were determined as the cycles at 25% drop of peak stress after deviation point from the steady state of peak stress changes with cycles.

The tension-compression strain rate was set as 0.01%/s.

The center portion of specimens shown as hatched area was longitudinally sectioned and polished for observation after the creep-fatigue tests. For EBSD observation, mid-sections of the specimens were polished in the similar manner for creep test specimen.

Fig.2. Creep-fatigue test specimen and sampling portion.

Fig.3 Schematic of the stress-strain hysteresis loop for tensile strain hold waveshape.

Table 6 Creep-fatigue failure and interruption test conditions for SUS304HTB

Test temperature (°C)	Total strain range $\Delta\epsilon_t$	Inelastic strain range $\Delta\epsilon_{ie}$	Creep strain range $\Delta\epsilon_c$	Hold time (min)	Number of cycles N	Cycle fraction N/N_f
650	0.01	0.0075	0.00047	10	128	0.17
					370	0.5
					740	1

Table 7 Creep-fatigue failure and interruption test conditions for Mod.9Cr steel

Test temperature (°C)	Total strain range $\Delta\epsilon_t$	Tensile strain rate $\Delta\epsilon_{ie}$	Compressive strain rate $\Delta\epsilon_c$	Hold time (min)	Number of cycles N	Cycle fraction N/N_f
650	0.01	0.0085	0.00068	10	106	0.1
					265	0.25
					529	0.5
					794	0.75
					1058	1
		0.0088	0.00082	30	230	0.5
					460	1

2.4. EBSD Observation

EBSD observation was conducted using the EBSD system (Tex SEM Laboratory: OIM 4.6) instrumented in Scanning Electron Microscope (SEM), JSM7000FS by JEOL. In this study, KAM maps and GOS maps were made from the observation data.

Figure 4 shows the procedure for calculating KAM and GOS values from subject maps. The calculated variables are hereafter denoted in italics like *KAM* and *GOS*. *KAM* estimated from the adjacent neighboring pixel angles is strongly dependent on step size d_s as defined in Fig.4, which is inversely proportional to the magnification of observation but *GOS* is supposed more independent on d_s because of the averaging effect of step sizes using overall intragranular spacing. The averaged values over observed areas are calculated as the averaged *KAM* and *GOS* values for all pixels in a map and denoted as KAM_{ave} and GOS_{ave} .

Fig.4. Definition of EBSD parameters.

The magnifications for SEM/EBSD observation were $\times 250 (d_s = 2\mu\text{m})$, $\times 500 (d_s = 1\mu\text{m})$, $\times 1000 (d_s = 0.5\mu\text{m})$ for SUS304HTB and $\times 2000 (d_s = 0.25\mu\text{m})$, $\times 3000 (d_s = 0.167\mu\text{m})$, $\times 5000 (d_s = 0.1\mu\text{m})$ for Mod.9Cr steel keeping the same position for changing the magnification. The higher magnifications for Mod.9Cr steel than SUS304HTB is due to smaller size of boundaries corresponding to blocks inside the prior austenite grains.

3. Results

Figures 5 shows the KAM maps for as-received, creep damaged and creep-fatigue damaged SUS304HTB samples. The area of misorientation angle over 1° increased with the increase of time fraction and cycle fraction showing the change of dislocation substructure. The solid lines show the boundaries of the misorientation over 15° which correspond to the austenite grain boundaries or twin boundaries. The change of *KAM* distribution with damage accumulation is more extensive in creep-fatigue tests than in creep tests, though the observation magnification is smaller in creep-fatigue tests than creep tests.

Fig.5. KAM maps for creep and creep-fatigue damaged SUS304HTB.

Figures 6 shows the KAM maps for as-received, creep damaged and creep-fatigue damaged Mod.9Cr steel samples. While the gradual decrease in the area of KAM over 1° (mainly below 2°) and block size coarsening were observed during the accumulation of creep damage, extensive decrease in the area of KAM over 1° and block size coarsening were observed even in the very early stage of creep-fatigue damage accumulation.

Fig.6 KAM maps for creep and creep-fatigue damaged Mod.9Cr steel.

Figure 7 shows the relationship between KAM_{ave} and time or cycle fraction for creep and creep-fatigue damaged SUS304HTB. The increasing trend in KAM_{ave} was observed for SUS304HTB. The step size effect is apparent.

Figure 8 shows the relationship between GOS_{ave} and time or cycle fraction for creep and creep-fatigue damaged SUS304HTB. The effect of step size is not so apparent as compared

with the case of KAM_{ave} . The increasing trend in GOS_{ave} during creep damage accumulation was observed while monotonic trend was not observed especially over 0.5 in N/N_f during creep-fatigue damage process.

Fig.7. Relationships between KAM_{ave} and time or cycle fraction for creep and creep-fatigue tests of SUS304HTB.

Fig.8. Relationships between GOS_{ave} and time or cycle fraction for creep and creep-fatigue tests of SUS304HTB.

Figure 9 shows the relationship between KAM_{ave} and time or cycle fraction for creep and creep-fatigue damaged Mod.9Cr steel. The initial decreasing trend in KAM_{ave} was observed showing some differences in the overall trend between creep and creep-fatigue.

Figure 10 shows the relationship between GOS_{ave} and time or cycle fraction for creep and creep-fatigue damaged Mod.9Cr steel. The initial decrease trend was observed in

GOS_{ave} but the effect of step size is not so apparent in creep compared with KAM_{ave} and the difference between creep and creep-fatigue was also observed.

Fig.9. Relationships between KAM_{ave} and time or cycle fraction for creep and creep-fatigue tests of Mod.9Cr steel.

Fig.10. Relationships between GOS_{ave} and time or cycle fraction for creep and creep-fatigue tests of Mod.9Cr steel.

4. Discussion

As the misorientation metrics themselves are not the direct measures of plastic or creep permanent strains, a new parameter is required for representing accumulated permanent strains during the creep and creep-fatigue damage process. We call this parameter as “EBSD strain parameter” [7]. The theoretical basis for defining this parameter is as follows.

If we assume the misorientation angle M is caused by the existence of total n dislocations with Burgers vector b within the unit pixel of characteristic step size d_s , the misorientation angle M is approximated as;

$$M = \frac{nb}{d_s} = \frac{n}{d_s^2} b d_s \approx \rho b d_s \quad (1)$$

where, ρ is the dislocation density approximated as n/d_s^2 for the thickness of unity.

The permanent strain induced by dislocation motion can be expressed by the following equation by the dislocation theory.

$$\gamma_p = \rho_m b d_B \quad (2)$$

where, ρ_m : dislocation density moved during the permanent deformation, d_B : dislocation mobile distance between boundaries defined as the border over the special misorientation value depending on the materials.

By substituting Eq.(1) into Eq.(2), we obtain the following relationship.

$$\gamma_p \approx M \frac{d_B}{d_s} \quad (3)$$

In Eq.(3), M is attributed as KAM related values in this study. We use two quantities here as the KAM related values KAM_{net} and δKAM_{ave} as follows.

$$KAM_{net} = KAM_{ave} - KAM_0 \quad (4)$$

$$\delta KAM_{ave} = |KAM_{ave} - KAM_i| \quad (5)$$

KAM_{net} in Eq.(4) means the net value of KAM_{ave} subtracting the common minimum value KAM_0 irrespective of damage level. KAM_0 is introduced here as the assumption that there is a unique baseline value of KAM irrespective of observation magnification and damage amount like a “background noise level” caused by the EBSD equipment itself.

δKAM_{ave} in Eq.(5) means the absolute value of the change in KAM_{ave} from initial value KAM_i obtained from the unused materials. Here KAM_i was attributed here to the undamaged materials value of KAM_{ave} . δKAM_{ave} can be obtained more easily than KAM_{net} without determining KAM_0 .

Figures 11 and 12 suggest almost linear relationship between KAM_{ave} and step size d_s . The step size $2\mu m$ is excluded from the regression for SUS304HTB in Fig.11 because this size is thought to be too large for maintaining adequate resolution of intragranular misorientation distribution. Then the following linear equation between KAM_{ave} and d_s can be obtained for various degree of damage by regression.

$$KAM_{ave} = A_K d_s + KAM_0 \quad (6)$$

where, A_K is regression constant but has different value according to damage.

The regression by Eq.(6) was actually conducted in the following manner.

1) The unique value for KAM_0 was set as the unused material's mean value over different d_s for SUS304HTB while as the average of lower bound values in creep and creep-fatigue over different d_s for Mod.9Cr steel. The values of KAM_0 were 0.00549 for SUS304HTB and

0.00698 for Mod.9Cr steel in radian scale respectively.

2)The individual slope value of A_K was determined by minimizing the sum square of the residuals from Eq.(6), i.e., $\sum |KAM_{ave} - (A_K d_s + KAM_0)|^2$, the values were 0.0096 for SUS304HTB and 0.0043 for Mod.9Cr steel respectively in radian scale.

Note that from Eq.(4) and Eq.(6), KAM_{net}/d_s is independent on d_s , i.e., observation magnification within the limited range, which is a convenient result for KAM measure.

Fig.11. Regression analysis for determining the unique KAM_0 value of SUS304HTB.

Fig.12. Regression analysis for determining the unique KAM_0 value of Mod.9Cr steel.

We define the dislocation mobile distance d_B as the grain boundary spacing for SUS304HTB and the block boundary spacing for Mod.9Cr steel according to the microstructural features. Figure 13 shows the definition of boundary spacing d_B from KAM maps for SUS304HTB and Mod.9Cr steel. The scanning lines are inclined 45° to the loading axis to measure the dislocation mobile spacing along assumed maximum shear stress direction, which may cause the permanent strain, though this rule is not mandatory. The boundary is identified as the grain boundary or twin boundary with over 15 degrees for SUS304HTB, and as prior austenite grain boundary or block boundary with over 5 degree for Mod.9Cr steel. The boundary spacing d_B is defined as the distance between two adjacent intersections of neighboring boundaries on the same scanning line.

Then KAM_{net} strain parameter $M_{eKAMnet}$ and δKAM_{ave} strain parameter $M_{e\delta KAMave}$ can be

defined as follows analogous to Eq.(3) using KAM_{net} or δKAM_{ave} as M .

$$M_{\epsilon KAM_{net}} \equiv KAM_{net} \frac{d_B}{d_s} \quad (7)$$

$$M_{\epsilon \delta KAM_{ave}} \equiv \delta KAM_{ave} \frac{d_B}{d_s} \quad (8)$$

Fig.13. Definition of characteristic boundary spacing.

Figures 14 and 15 show the change of boundary spacing with time fraction and cycle fraction in creep and creep-fatigue tests for SUS304HTB and Mod.9Cr steel. The trends are almost opposite for those two materials, that is, SUS304HTB shows decreasing trend while Mod.9Cr steel shows rather increasing trend. The major cause of the decrease in d_B for SUS304HTB may be attributed to the deformation of grains toward the loading axis and the increase in precipitation areas.

Fig.14. The change of boundary spacing against time fraction or cycle fraction for creep and creep-fatigue tests of SUS304HTB.

Fig.15. The change of boundary spacing against time fraction or cycle fraction for creep and creep-fatigue tests of Mod.9Cr steel.

Figures 16 and 17 show the relationship between the strain parameters with creep strain ε_c or accumulated creep strain $N\Delta\varepsilon_c$ for creep-fatigue tests for SUS304HTB. The creep strain values corresponding to t/t_r were listed in Tables 4 and 5. $N\Delta\varepsilon_c$ is the product of number of cycles N (interrupted cycles or cycles to failure N_f) and creep strain range $\Delta\varepsilon_c$ which is interpreted as the accumulated tensile (unbalanced) creep strain component. $M_{\varepsilon KAMnet}$ and $M_{\varepsilon \delta KAMave}$ show almost similar increasing trend with the increase in creep strain components, especially indicating good correspondency between creep and creep-fatigue data approximately below the creep strain value of 0.2. This result suggests that the strain parameters may represent the one-way(or unbalanced) accumulation of strain during the damage process except in the later stage of damage. As the saturating trends near failure can be interpreted as the localization of strain at the site of major cracks, the onset of saturation can be identified as the final failure stage of the damage process.

Fig.16. Relationships between KAM_{net} strain parameter $M_{\varepsilon KAMnet}$ and creep strain or accumulated creep strainrange for creep and creep-fatigue tests of SUS304HTB.

Fig.17. Relationships between δKAM_{ave} strain parameter $M_{\epsilon\delta KAM_{net}}$ and creep strain or accumulated creep strainrange for creep and creep-fatigue tests of SUS304HTB.

Figures 18 and 19 show the relationship between the strain parameters and with creep strain ϵ_c or accumulated creep strain $N\Delta\epsilon_c$ for creep-fatigue tests for Mod.9Cr steel. $M_{\epsilon KAM_{net}}$ cannot represent the common increasing trend for creep and creep-fatigue against the strain accumulation while $M_{\epsilon\delta KAM_{ave}}$ shows more common monotonic increase trend especially in the relatively early stage of creep and creep-fatigue. There also can be observed the end of increasing trends which suggest the strain localization probably due to major crack growth.

Therefore, $M_{\epsilon\delta KAM_{ave}}$ is thought here as the better parameter for evaluating strain-based damage accumulation in creep and creep-fatigue process both for ferritic and austenitic heat resistance steels.

Fig.18. Relationships between KAM_{net} strain parameter $M_{\epsilon KAM_{net}}$ and creep strain or accumulated creep strainrange for creep and creep-fatigue tests of Mod.9Cr steel.

Fig.19. Relationships between δKAM_{ave} strain parameter $M_{\delta KAM_{net}}$ and creep strain or accumulated creep strainrange for creep and creep-fatigue tests of Mod.9Cr steel.

5. Conclusion

EBSD analyses for creep and creep-fatigue damage imposed materials were conducted on SUS304HTB and Mod.9Cr steel. Obtained results are as follows.

- (1)EBSD misorientation parameters KAM_{ave} and GOS_{ave} showed opposite trend for SUS304HTB and Mod.9Cr steel. SUS304HTB showed increasing trend and Mod.9Cr steel showed decreasing trend with respect to time fraction and cycle fraction for creep and creep-fatigue damaged samples.
- (2)Boundary spacing for SUS304HTB showed decreasing trend with damage accumulation mainly due to formation of twin boundaries, while that of Mod.9Cr steel showed rather increasing trend due to subgrain structure recovery.
- (3)The strain parameter $M_{\delta KAM_{ave}}$ showed common monotonic increasing relationship with the accumulation of creep strain component in the relatively early stage of creep damage and creep-fatigue damage for either material. The end of the increasing trend was probably due to the onset of predominant strain localization due to major cracks which should be investigated in more detail.

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