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# Grain Boundary Deformation at High Temperature Tensile Tests in ODS Ferritic Steel

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The tensile test of the recrystallized ODS ferritic steels was performed in the loading direction for the longitudinal and 45° inclined with respect to the grains alignment. The testing temperature was 800°C and the strain rate was $10^{-4}$ $s^{-1}$. A clear serration structure was observed at near the grain boundaries at the surface of 45° specimen ruptured. This is a clear evidence of the occurrence of the grain boundary sliding in 45° direction. For the total strain of 12% in 45° direction, grain boundary deformation induced by sliding was estimated about 9%, whereas the amounts of the transgranular strains was 2% measured by EBSD analysis. The grain-subdivision was also identified near grain boundaries by FIB analysis, which could be caused by a dynamic recrystallization during the localized grain boundary deformation.

KEY WORDS: ODS ferritic steel; tensile test; grain boundary sliding; serration; misorientation; electron backscattering diffraction; recrystallization.

## 1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic steels are significantly strengthened at high-temperatures by dispersing a high number density of nano-size oxide particles in the ferrite matrix with superior radiation resistance. These steels are being developed by Japan Atomic Energy Agency (JAEA) as one of the most promising cladding materials for sodium-cooled fast breeder reactors. The ODS ferritic/martensitic steels are also expected as advanced heat-resistant steels for a next generation fossil power plant with extremely high thermal efficiency, toward reducing $CO_2$ emission. There are two types of ODS ferritic/martensitic steels; one is 9CrODS martensite steel which has grain structure controlled by a reversible $\alpha/\gamma$ phase transformation,[1–4)] the other is 12-15CrODS ferritic steel which has a recrystallized structure.

For the recrystallized 12CrODS ferritic steel manufactured in the form of a cladding tube, it was shown that the creep rupture strength is considerably reduced in the hoop direction as compared with that in the longitudinal direction, when the tube was internally pressurized.[5)] One of the authors pointed out that this strength reduction in the hoop direction could be attributed to the grain boundary sliding which took place along the boundary inclined 45° with respect to the stress axis. Terada *et al*. also observed a trace of the grain boundary sliding at specific coincidence site lattice (CSL) boundary in the recrystallized ODS ferrite by means of a transmission electron microscopy.[6)] It is still not very clear how the grain boundary sliding affect on the strength and fracture mode of the ODS ferritic steels at high temperatures.

In this study, in order to improve the hoop strength of the ODS ferritic steels, we conduct tensile tests at 800°C for the recrystallized ODS ferrite with grain boundaries parallel to and inclined 45° directions with stress axis, and the amounts of strains induced by grain boundary and intra-grain deformations was quantitatively evaluated.

## 2. Experimental Procedure

### 2.1. Tensile Test

The composition of the ODS ferrite is Fe-15Cr-2W-0.35$Y_2O_3$ (mass%), and their bars were manufactured by a hot exclusion at 1,150°C after mechanical alloying of each powder. The sheets were cut from the bars, and were cold-rolled with the reduction of 85%. Then the cold-rolled specimens were annealed at 1,150°C to make a recrystallization with the elongated large grains along the cold rolling direction. The tensile test specimens were prepared in parallel to the longitudinal direction, designated as LD, and inclined 45 degree direction, 45°, with respect to the elongated grains along the rolling direction by means of an electric discharge processing machine. This appearance is schematically shown in **Fig. 1**. The size of the specimen is 5 mm in gauge length, 1.2 mm in width and 0.5 mm in thickness, as shown in **Fig. 2**. The tensile test was carried out at 800°C with a strain rate of $10^{-4}s^{-1}$ under argon gas atmosphere to prevent a surface oxidization. The ruptured elongation was measured by a scale through a change of the gage length of both

 

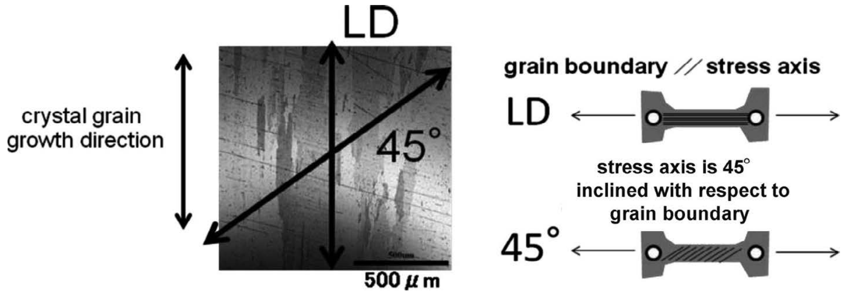


**Fig. 1.** The relationship of the direction of crystal grain growth and stress axis in tensile specimens cut from the recrystallized ODS steels.

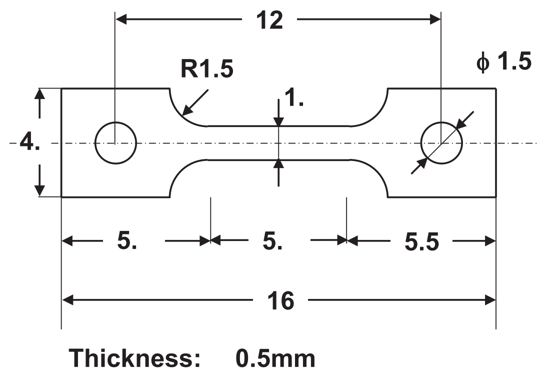


**Fig. 2.** A schematic view of tensile test specimen.

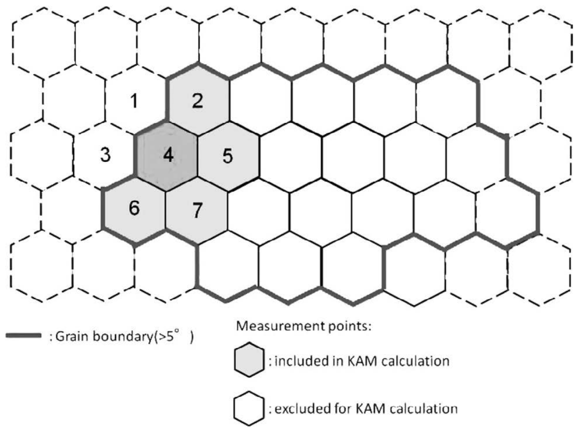


**Fig. 3.** A schematic Model of KAM measurement.

ruptured pieces after the tensile test.

### 2.2. Microstructural Observations

Microstructural observations were conducted by optical microscopy (OM) and scanning electron microscopy (SEM) to estimate grain boundary sliding. Besides, a focused $Ga^+$ ion beam (FIB) with a JEOL JFIB-2300 system was utilized to make a 10 $\mu$m deep hole and to directly observe grain boundaries from the thickness direction of the plate specimen.

The electron back scattering diffraction patterns (EBSD) analyses were conducted to assess contribution of the intra-grain deformation by using a JEOL JSM-6500F field-emission scanning electron microscope (FE-SEM) equipped with TSL software; OIM-Analysis 4 (TexSEM Laboratories). EBSD analyses provide information of crystalline misorientation among grains and plastic deformation behavior of ODS ferritic steel with the resolution of 0.2 $\mu$m. For the EBSD analyses, specimens were polished with #400~4000 emery paper, 1 $\mu$m diamond paste and alumina colloidal silica.

There is Kernel Average Misorientation (KAM) to assess a quantitative rotation angle of the crystalline.[7–10)] KAM value means an average of misorientation angle of surrounding six points for the center point. A schematic view is shown in **Fig. 3**. KAM value for center point-4 is calculated as an intra-grain misorientation according to Eq. (1), when $\alpha_{1,4}$ and $\alpha_{3,4}$ are higher than 5° that are defined as grain boundary; namely point-1 and point-3 are out of grain occupied by center point-4.

$$\mathrm{KAM} = \frac{\alpha_{2,4}+\alpha_{5,4}+\alpha_{6,4}+\alpha_{7,4}}{4}, \quad \text{................ (1)}$$

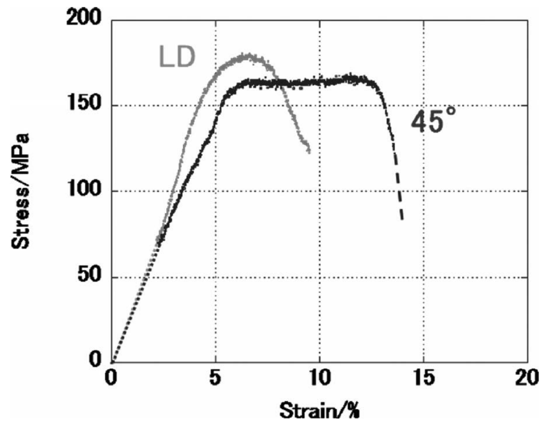


**Fig. 4.** Stress strain curves in the recrystallized ODS ferritic steel at 800°C with a strain rate of $10^{-4}s^{-1}$.

where $\alpha_{i,j}$ is the misorientation angle between *i* and *j* points. Kimura *et al*. noted that KAM value has linear relation with an amount of plastic deformation for completely annealed nuclear grade austenitic steels SUS316NG.[7)]

## 3. Results and Discussion

### 3.1. Intragranular Deformation

**Figure 4** shows the stress-strain curves for the loading directions of LD and 45° with respect to the elongated grains along the rolling direction. The test temperature was a 800°C and strain rate of $10^{-4}s^{-1}$. For the 45° direction, the stress is slightly reduced at the initial startup of strain, and the rupture strain is further extended by keeping almost constant stress as compared with LD direction. The ruptured elongation that was measured by scale for the ruptured specimens is 11.6% for 45° and 9.2% for LD. These results for the 45° specimen could be attributed to the easy plastic deformation at the grain boundaries from the lower stress level. The amount of plastic deformation by necking was estimated to be about 1% for 45° and 3% for LD.

**Figure 5** shows an inverse pole figure (IPF) analyzed by EBSD for the ODS ferritic steel before and after the tensile test for LD and 45° directions. Microstructure near the fracture area is slightly fine. From the results of average KAM value in the gauge section, increase of the transgranular misorientation angles from before to after tensile test at the LD and 45° directions turns out to be 0.32° and 0.24°, respectively. From these misorientation angles, the amount of transgranular deformation was derived by using the following method. When each crystal grain is plastically deformed, crystal grains are subject to constraint from the surrounding grains, because the slip system operated in each grain is different. The transgranular misorientation angle ($\theta_{rad}$) can be correlated by Read[11] as Eq. (3.1) for a low angle boundary using Burgers vector (b) and a space between dislocations (l),

$$\theta_{rad} = \frac{b}{l}. \quad (3.1)$$

**Figure 6** illustrates a simplified transgranular model, and grain diameter ($D$) is expressed as

$$D = n \cdot l, \quad (3.2)$$

where n is the number of dislocations. Ashby suggested that geometrically necessary dislocations (GNDs) are generated in order to accommodate misfit of the deformed grains, and dislocation density ($\rho_g$) and intragranular strain ($\varepsilon$) were correlated by using the following relation,[12]

$$\rho_g = \frac{n}{D^2} \approx \frac{\varepsilon}{4bD}. \quad (3.3)$$

From Eq. (3.1) to Eq. (3.3), the transgranular strain ($\varepsilon$) can be expressed in terms of the misorientation angle ($\theta_{rad}$) which can be measured by EBSD,

$$\varepsilon = 4\theta_{rad}. \quad (3.4)$$

By substituting the measured values of $\theta_{rad}$ of 0.32° and 0.24° into Eq. (3.4), the amounts of the deformed transgranular strains are obtained to be 2.2% and 1.7% for LD and 45° directions, respectively.

From OIM analysis of the recrystallized ODS steel in Fig. 5(a), it was found that the ratio of CSL boundary (Σ3~29) is 0.83 and the ratio of random grain boundary (Σ29~49) is 0.17, which suggests that the specimen of the recrystallized ODS ferritic steels are mainly composed of CSL boundaries.

### 3.2. Grain Boundary Deformation

**Figure 7** shows SEM micrographs of the fractured area for LD and 45° specimens. It is found that fracture surface in LD specimen is 45 degree inclined with respect to the loading direction and the fracture take place in the mixture of intergranular and transgranular modes. On the other hand, 45° specimen is completely fractured along the grain boundaries.

**Figure 8** represents SEM micrographs on the surface of 45° specimen and its higher magnification at area of grain boundaries. A serration type pattern is clearly observed at all of the grain boundaries. This pattern seems to be formed, when slip lines are twisted across the grain boundaries. This finding provides a clear evidence of severely localized deformation at grain boundaries. A similar behavior is reported by Watanabe for Fe-0.8at%Sn alloy in creep test at 700°C.[13] The grain boundary sliding could be incorporated in this type of grain boundary deformation. The amount of strain induced by the grain boundary deformation at the entire parts of the gauge length was estimated to be about 10% by using about 2 μm for the average width of the serration which contributes equivalently to the longitudinal elongation and 250 for the number of grains within the gauge length (5 mm) on the basis of SEM observation.

The FIB was conducted at the area near the grain boundaries of 45° specimen. **Figure 9** shows scanning ion microscopy (SIM) of the holed area by using ion-induced secondary electrons. At the cross section, small grains and some voids are clearly observed at near grain boundaries. Such voids could be formed by grain boundary sliding. The

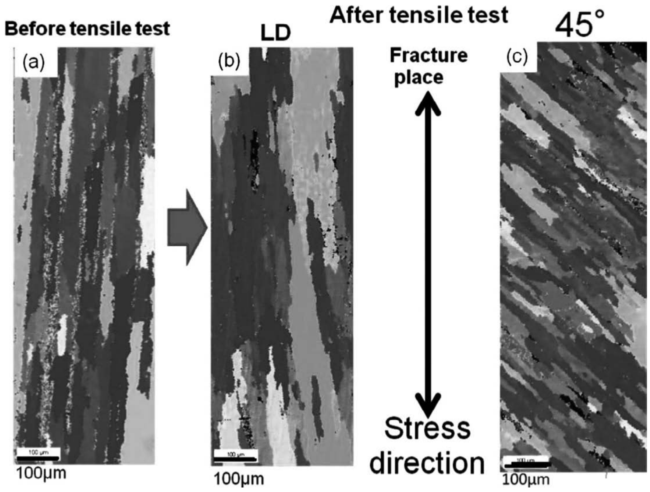


**Fig. 5.** Inverse Pole Figure (IPF) of the ODS steel before and after tensile test using EBSD measurement. (a) before tensile test, (b) after tensile test in LD direction, (c) 45° direction.

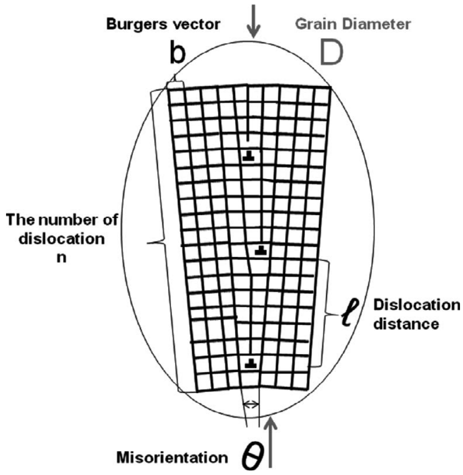


**Fig. 6.** A schematic view of transgranular plastic deformation which represents a relationship between the misorientation and dislocation alignment.

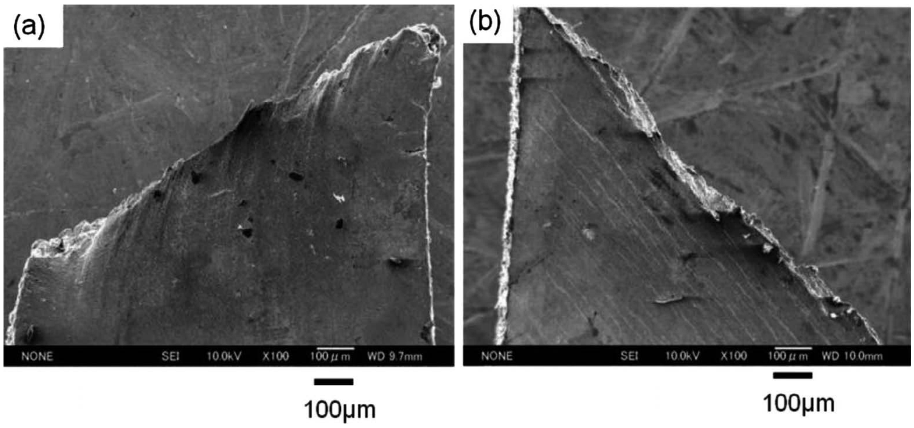


**Fig. 7.** SEM microscographs of fracture area after tensile test at 800°C : (a) LD and (b) 45° directions.

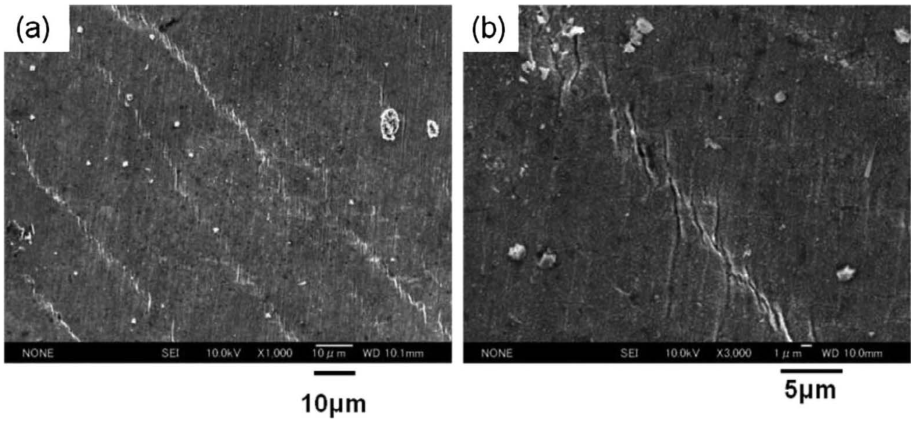


**Fig. 8.** SEM micrographs of fracture area after tensile test at 800°C in the recrystallized ODS ferritic steel.

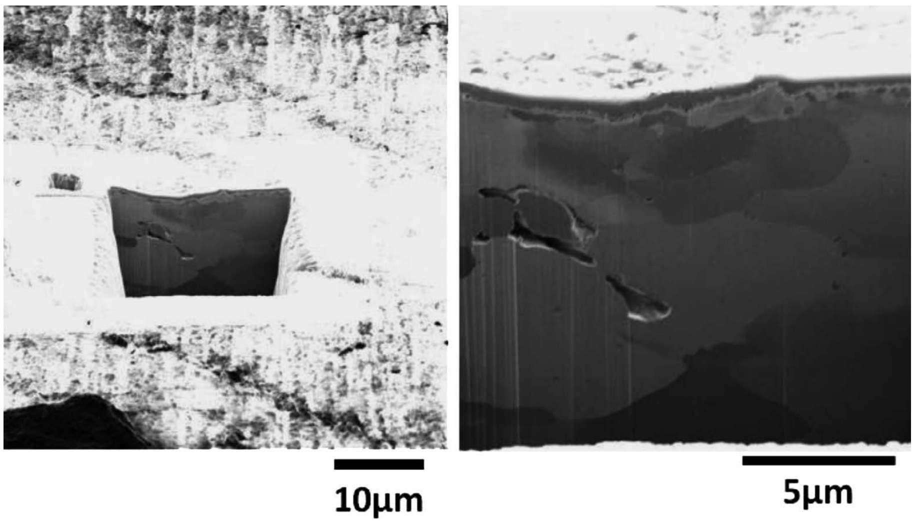


**Fig. 9.** Micrographs by FIB observation after tensile test at 45° direction (Tilt 60°).

formation of the grain-subdivision near the grain boundaries could be caused by the dynamic recrystallization during the localized grain boundary deformation. The driving force for the grain-subdivision could be accumulation of the deformed strain at near grain boundaries. Similar observation by FIB was reported by Motoyashiki *et al.*,[14] showing that cracks are formed along slip bands in a dual phase steel. These evidences clearly demonstrate an occurrence of the grain boundary sliding.

High Temperature deformation of the ODS ferritic steel consists of the grain boundary deformation, transgranular deformation induced by crystalline rotation and necking. On the basis of results of tensile tests, **Fig. 10** shows the contribution of each deformation process. The amount of a total plastic deformation is 9% for LD and 12% for 45°, respectively. For 45° direction, 2% of the transgranular deformation and 1% of necking are observed, thus an amount of grain boundary deformation can be estimated to be 9%. This value is similar to one estimated from the serration width. In addition, grain boundary deformation in 45° direction is

 

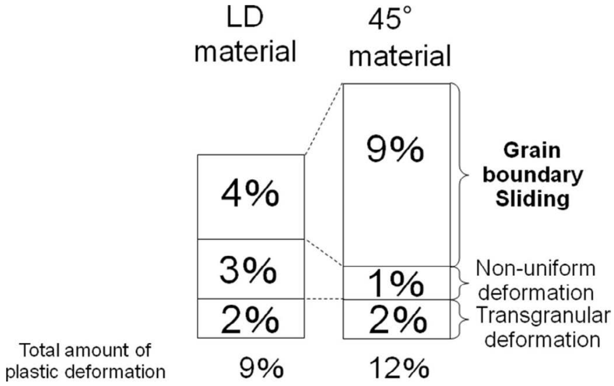


**Fig. 10.** Mechanism of high temperature deformation in ODS ferritic steel.

twice higher than that in LD direction. Since grains themselves of the ODS ferritic steel are reinforced by the fine dispersion of oxide particles, it is recognized that grain boundary deformation is a dominant process for the high temperature deformation.

## 4. Conclusion

The tensile test was performed at longitudinal and 45° directions for the recrystallized ODS ferritic steel at 800°C and a strain rate of $10^{-4}$ s$^{-1}$. The main results are summarized as follows;

(1) The clear serration structure was observed at the grain boundaries on surface of the ruptured specimens. This is a clear evidence of the occurrence of the grain boundary sliding in 45° direction.

(2) For the total strain of 12% in 45° direction, grain boundary deformation induced by sliding was estimated to be about 9%, whereas the amounts of the transgranular strains was 2%, which was measured by EBSD analysis.

(3) The grain-subdivision was identified at near grain boundaries by FIB analysis, which could be caused by a dynamic recrystallization during the localized grain boundary deformation.

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