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Oxide particle–dislocation interaction in 9Cr-ODS steel

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ABSTRACT

Oxide Dispersion Strengthened (ODS) ferritic/martensitic steels have an excellent high temperature strength primarily due to a dislocation pinning effect of nanometric oxide particles. In the present work, the interaction between oxide particles and dislocations in 9CrODS ferritic steel was investigated by both static TEM observation and in-situ TEM observation under dynamic straining conditions. The primary concerns of those observations were the obstacle strength of oxide particles and the type of interactions: attractive or repulsive. In the static observation, the majority (~90%) of all interaction geometries was characterized as repulsive type. In the in-situ straining experiments, the obstacle strength α of oxide particles was estimated to be no greater than 0.80. The experimentally-determined obstacle strength is smaller than that of Orowan type impenetrable obstacle, whereas those oxide particles are, in theory, ideally strong obstacles. The gap between predicted and measured obstacle strength is attributable to cross-slip motion of screw dislocations on the oxide particles.

1. Introduction

Reduced activation ferritic/martensitic steels (RAFMs) are candidate for blanket and first wall in fusion reactors because of their maturation in technology and good radiation resistance [1]. However, RAFMs suffers from the loss of strength at above 823 K [2]. In order to increase their creep properties at higher temperatures, ODS (oxide dispersion strengthening) is one of the most effective method. For example, the tensile strength of 9CrODS steel is 200 MPa higher than those of non-ODS 9Cr steels at 923 K [3]. This is because that fine stable oxide particles, which is dispersed densely in the matrix, hinder dislocation motion at higher temperatures.

The interaction between such impenetrable fine precipitates and dislocations have been discussed by many researchers using theoretical analyzation and computer simulations [4– 7]. Few other researchers have been used in-situ tensile method combined with transmission electron microscope (TEM) to reveal the interaction [8– 11]. There are two opposite interactions: one is repulsive, e.g. the Orowan interaction, and the other is called Slorovitz interaction, which is attractive mechanism especially adopted at high temperature. Most of the researchers concluded that dislocations overcome the impenetrable precipitates by the Orowan interaction.

In this study, we apply in-situ TEM tensile test to a 9CrODS steel. In-situ TEM tensile experiment is one of the attractive method to give an answer to the mechanism of interaction between dislocation and obstacles, because dislocation motions are directly observed during the experiment. However, there are very few reports which apply in-situ TEM tensile test to ferritic steels, because of bad resolution caused by magnetism. We overcome the limit of resolution by a newly-devised experimental procedure and succeeded the test with a generic 200 kV TEM. We measure bow-out angles of dislocation and convert angles into an obstacle strength (α), aiming to estimate the depinning mechanism of dislocation from oxide particles. The analysis method deciding threshold stress and the interaction mechanisms by α was reported by several theses [6,12–14]. We apply room temperature to the present work, as a former step of the experiment at high temperature.

2. Experimental procedure

The starting material is a 9CrODS steel cladding tube, measuring φ 8.87 mm × thickness 0.66 mm × length 330 mm, which composition is described in Table 1. Its final heat treatment condition was a 1323 K for 1 h annealing and a 423 K/h furnace

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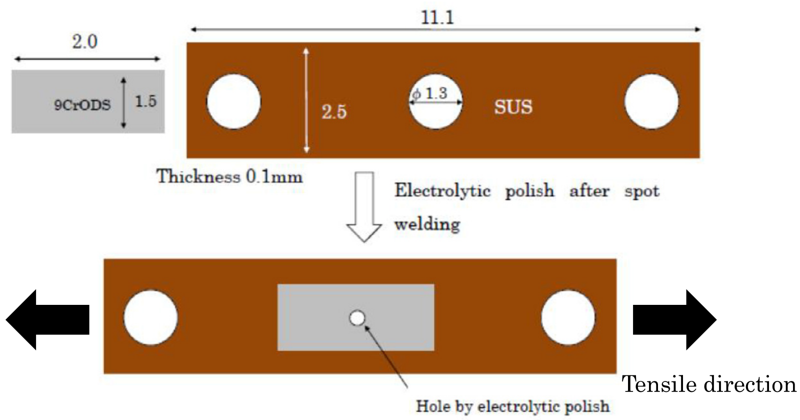

Fig. 1. Method of making in-situ TEM tensile specimen.

Table 1
Composition of 9CrODS steel tube.

Fe	Cr	C	W	Ti	Y_2O_3
Bal.	9	0.13	2	0.2	0.35

cooling. The tube was cut and grinded into 0.1 mm thick. We applied two types of specimen; one is for usual TEM observation and the other is for in-situ TEM tensile experiment. For the former experiment, the specimen was punched into φ3.0 mm disk. The surface of the disk was deformed by using Vickers' hardness tester to observe dislocations tangling with oxide particles. For the latter experiment, the specimen was cut into small rectangles measuring 2.0 mm × 1.5 mm, enough to avoid magnetism originated from ferritic steel in 200 kV TEM equipment. We attached the rectangle to a non-magnetic SUS304 plate (with holes measuring φ1.3 mm at both edges and center) by spot welding. The shape of the tensile specimen is shown in Fig. 1. Both of the final specimens were electro polished before TEM observation.

Both usual and in-situ tensile observation were performed by TEM (JEOL JEM-2010) with acceleration voltage of 200 kV. For tensile experiment, a single tilt tensile holder (GATAN Model654) was applied. The moving dislocation was captured by a CCD camera with a capturing speed of 1/50 s.

3. Results

3.1. Static analysis

Prior to the in-situ TEM tensile experiment, we judged interaction mechanism between dislocations and oxide particles by observing the deformed 3 mmφ TEM specimen. Two kinds of interaction are considered: repulsive or attractive. One of the typical repulsive interaction is Orowan interaction. In this case, dislocation leaves an Orowan loop around a particle after depinning. The typical attractive interaction is called Srolovitz interaction [15,16]. If the dislocation reaches particle's surface by Srolovitz interaction, the dislocation is looked like disappeared at the surface of the particle, i.e., new dislocation segment is generated from the particle. It was difficult to judge the detail of the interaction perfectly by the static observation after deformation; so we only judged which kind of interaction is dominant. Fig. 2 shows bright fields images and dark field images from which we judged the interaction. If a repulsive interaction occurs, the strain contrasts appear originated from dislocation around the particles. If an attractive interaction occurs, dislocation segments disappear near the surface of the particles. The repulsive and the attractive interaction are pointed by arrows with solid and dotted lines, respectively. In such a way, we judged the interaction at around 21 particles in this specimen; 20 interactions were repulsive and 1 interaction was attractive. From the present work, we can say that the repulsive interaction is dominant in 9CrODS steel.

3.2. In-situ TEM tensile test

Fig. 3 shows a snapshot taken from the in-situ TEM tensile experiment. We traced bow-out dislocations by two ellipses, draw two tangent lines along each ellipse, and finally measured the angle between the two lines (Fig. 3b; Wulff's construction method [17]). The bow-out angle φ' measured in Fig. 3 was $\varphi' = 92°$. It should be noted the φ' is measured on the observed plane projected to a fluorescent screen in TEM, and should be corrected to the real angle on the slip plane in the specimen (see Fig. 4). In the case of Fig. 3, the tensile direction **t** is deduced as [1 -2 1] (determined from the diffraction pattern (Fig. 3c) and the actual direction of the TEM tensile holder). The observed plane **z** is (012). Presuming that the screw dislocation is dominant in thin film [18], Burgers vector is drawn by arrow **b** (parallel to the dislocation line). Considering that the major slip system in BCC metal is <111> {011} or {112}, direction **b** was deduced as [1 -1 1]. The slip plane judged by the maximum Schmid factor is (011). Finally, the slip system of dislocation is determined [1 -1 1] (011). From the orientation relationship drawn in Fig. 4, the following formula is derived;

$$\cos\theta = \frac{\cos\phi'}{\cos\phi} \tag{1}$$

θ is the inner angle between the projected plane and the real slip plane. In the case of Fig. 3, cosθ is calculated as 0.95. Applying the $\varphi' = 92°$ and $\cos\theta = 0.95$, $\cos\emptyset = -0.0367$ and $\emptyset = 92.1°$. Increase in stress τ by particles is described as the following formula,

$$\tau = \alpha\frac{Gb}{\lambda} \tag{2}$$

where G is modulus of rigidity, b is Burgers vector and λ is dispersion length. α is called obstacle strength and is described as the following formula;

$$\alpha = \cos\left(\frac{\emptyset}{2}\right) \tag{3}$$

Fig. 5 shows the results of φ and α carried out from 10 bow-out dislocations. The φ is roughly gathered between 90° and 120°,

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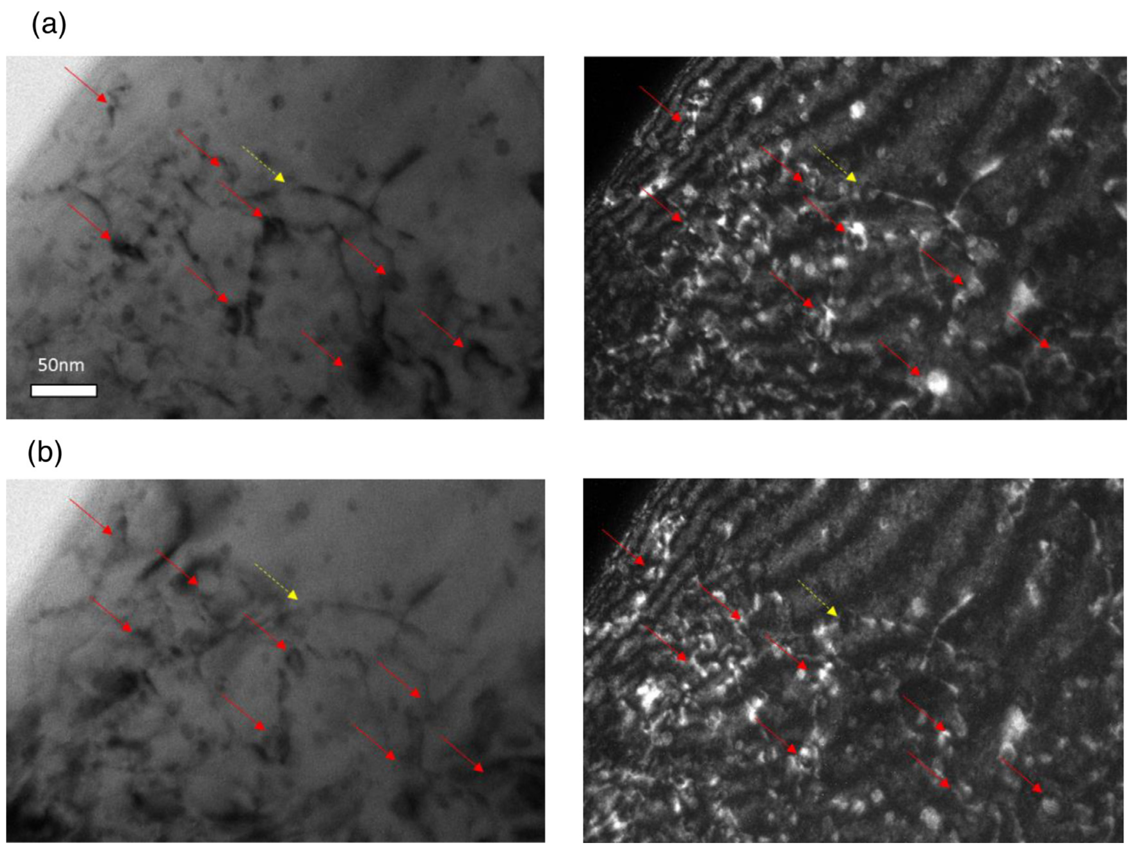

Fig. 2. TEM image (Bright field and Dark field) of interaction between interaction oxide particles and dislocations. Due to considering only dislocation overlapping above particle, we took two different tilt images ((a) and (b)) at same place. Dotted arrows point attractive interaction, solid line arrows point repulsive interaction.

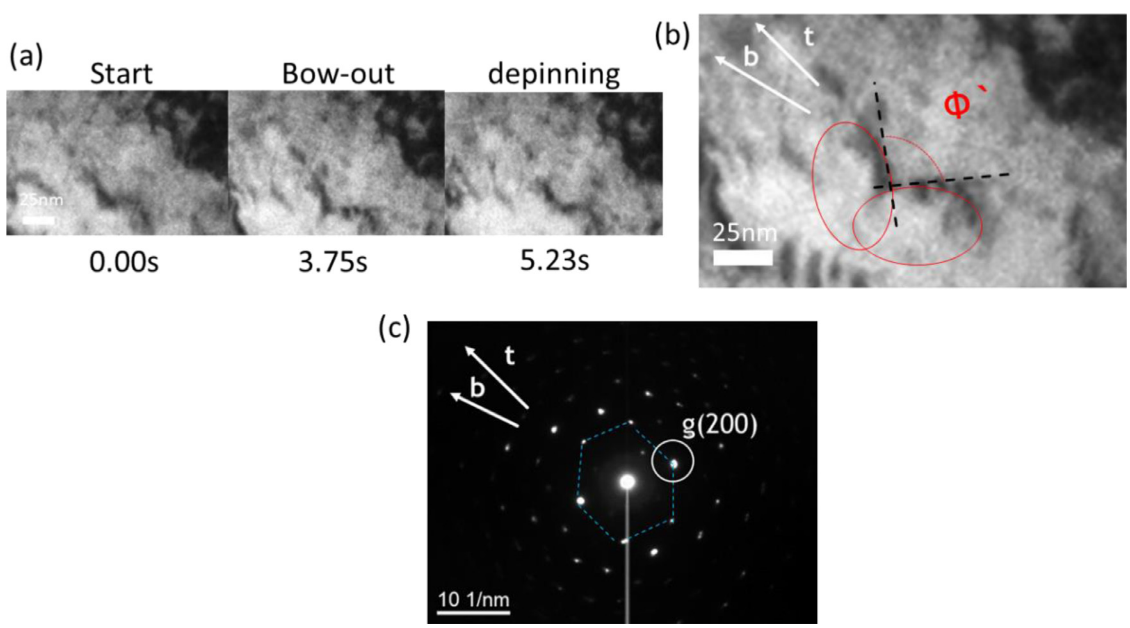

Fig. 3. (a) TEM image captured movie in in-situ TEM tensile test. (b) This is stated that the method of dislocation bow-out angle, the angle between two tangents of ellipse tracing dislocation shape is bow-out angle. Arrow t is the tensile direction, arrow b is the dislocation line direction and burgers vector. (c) The diffraction pattern of Fig. 3 (a) and (b). Looked spots surround dotted lines , observed plane z is (012). Arrow b and t is same in Fig. 3 (b).

which corresponds to α ranging from 0.5 to 0.7. The average φ and α is 106° and 0.59, respectively.

In the present work, we presumed that the observed dislocation as pure screw dislocation. However, active dislocation is usually mixed type. For stricter analysis, the continuous observation of gliding dislocation could be applied [19].

4. Discussion

4.1. The dominant interaction at room temperature

The present static TEM observation shows that most of the interactions between oxide particles and dislocation are repulsive. In

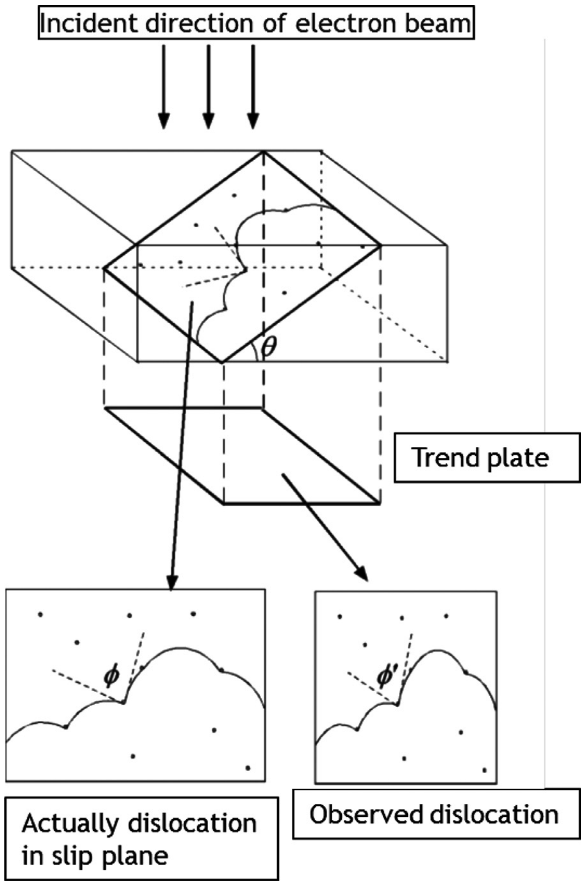

Fig. 4. Relationship between slip plane (angle ϕ) and observed plane (angle ϕ') [17]. Θ is angle between slip plane and observed plane.

the present study, we applied 9CrODS steel, in which most of the oxide particles are $Y_2Ti_2O_7$ [20]. $Y_2Ti_2O_7$ particles in an ODS ferritic steel have partial-coherence towards the ferrite matrix [21]. However, we consider that the coherency of the particles has no effect on the pattern of the interaction in this experiment. Although the attractive interaction occurs in the incoherent interface, it is generally said that this interaction occurs at high temperature [15]. Bartsch et al. and Yoshizawa et al. mentioned that oxide particles in the ODS ferritic steels showed attractive interaction with dislocation [9, 22]. Their experiments were performed at higher temperature at above 600 °C. Bartsch observed the interactions with the incoherent oxide particles. On the other hand, Yoshizawa treated a 9CrODS steel which has the very similar composition to that of our material. We conclude that the repulsive interaction is dominant in the present work, not the attractive interaction (Slorovitz interaction), because we observed them at room temperature.

4.2. The detailed mechanism of the interaction

The most likely repulsive interaction which reported in the literature is Orowan interaction. Bako et al., studying about the PM2000 [4], and many reports of MD simulation support Orowan interaction. For Orowan interaction, there are some different values of α reported from the literature [23, 24]. One is $\alpha = 0.81$ calculated by MD simulation [23] and another is $\alpha = 0.84$ in the geometric calculation [24]. The maximum value of α in the present work reaches 0.80; the value agrees with the simulated values in the literature. On the other hand, the average value of α, 0.59, is quite smaller than those of the literature. Some other mechanisms of interaction should be considered as well as Orowan interaction.

If the G of the particle is not higher than that of the matrix, dislocation can "cut" the particles and then the values of α becomes much smaller than those of Orowan interaction [12, 13]. However, $Y_2Ti_2O_7$ particles in 9CrODS steel are impenetrable particles whose G is much higher than that of the Fe-Cr matrix [25]. Therefore, cutting process must not occur in this experiment.

– If the moving dislocation is a screw type or a mix (screw and edge) type, cross-slip can be considered to be the suitable mechanism as well as Orowan interaction. Fig. 6 shows some examples how dislocation overcomes oxide particles by cross-slip based on the present results. Fig. 6(a) explains the observed result in Fig. 3(b): screw dislocation changes its slip plane when it faces a particle (the upper and the middle image); then, two segments of edge dislocation are created (the middle image); the two edge segments having same Burgers vector but opposite direction of displacement annihilate each other, and remained screw dislocation goes through a parallel plane to the initial slip plane (the lower image). In the case of Fig. 6(a), the slip direction for the edge dislocation must not hit the particle. Some other interactions in the present work appeared to be as Fig. 6(b): screw dislocation changes its plane as same as the case of Fig. 6(a), but only one edge segment is created (the upper and the middle image); in this case the created edge segment moves its slip plane just in front of the particle and circumvent the particle (the lower image). The case of Fig. 6(b) looks energetically easy than the case of Fig. 6(a). Although many interactions such as Fig. 6(b) were also observed in the present experiment, there was no obvious image from which the bow-out angles could be measured.

There is another type of repulsive interaction, called Hirsch interaction [7, 26]. Hirsh interaction is a mixture of Orowan interaction and the cross-slip interaction. Y.Xiang et al. supported this mix-type interaction [5]. With Hirsch interaction, dislocation loops are created in adjacent of the particles and not surround the particle as Orowan loop. In the present work, we could not observe such a tiny loop in adjacent of the oxide particles because of the limit of resolution caused by magnetism of the specimen. It is necessary to improve the specimen to avoid magnetism of steels or to try the experiment with big oxide particles.

There is some literature which studies particle-dislocation interaction by in-situ TEM tensile test at high temperature [8–11]. They have been reported that dislocation climb is observed as bypass process. The present work was done only at room temperature and clime motion of the dislocation should be difficult. The present work is a former step of the experiment at high temperature, as mentioned in the introduction section. We will consider the dislocation climb in the further studies.

Table 2
bow-out angles from 10 interactions.

Angle (φ)
86
92.1
135
128
73
119
101
102
114
110

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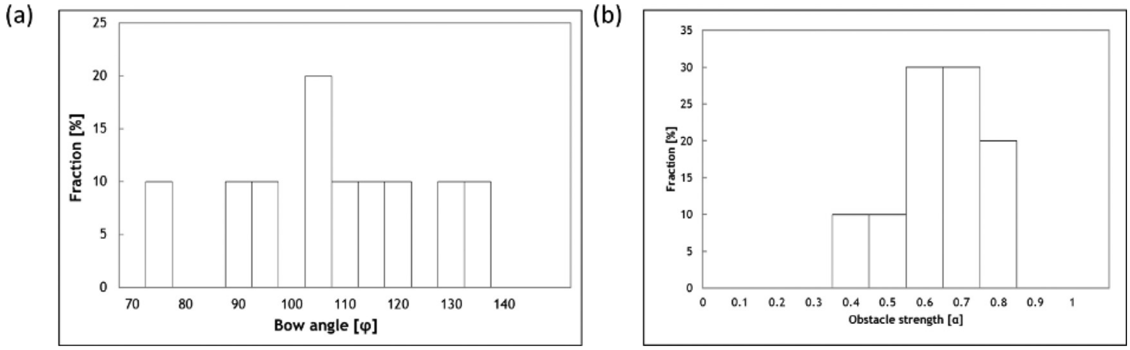

Fig. 5. (a) Histogram of dislocation bow-out angle (measured from 10 interactions). (b) Histogram of obstacle strength α.

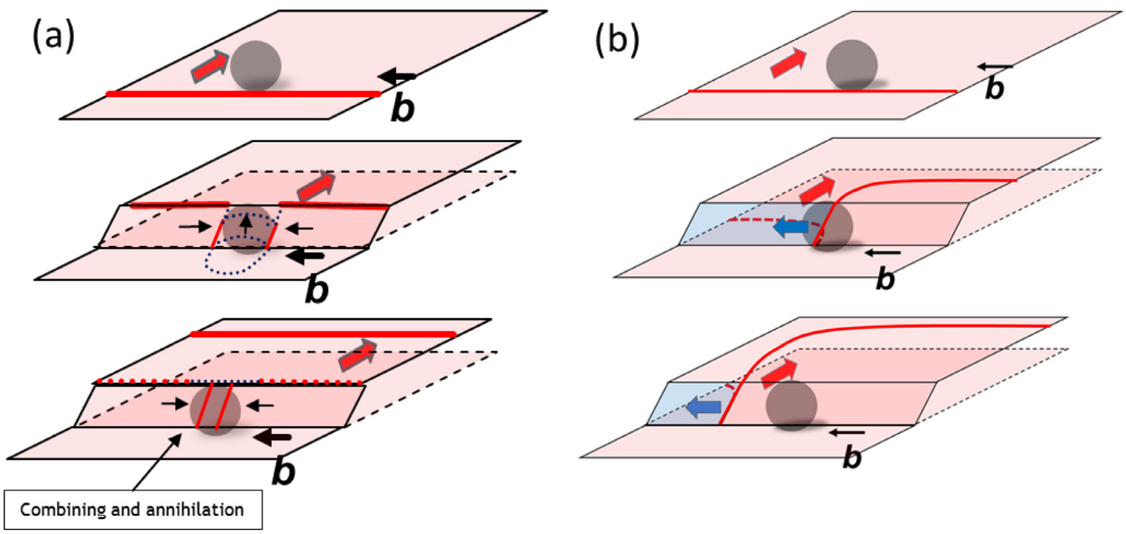

Fig. 6. On the basis of the observed movie result with in-situ TEM tensile experiment, bypass mechanism of dislocation from particle by double cross-slip. b is Burgers vector of dislocations.

5. Conclusion

A static observation and in-situ TEM tensile test was applied to analyze the interaction between oxide particles and dislocation in 9CrODS. More than 90 percent of the interaction was deduced as repulsive. The dislocation bow-out angles observed by in-situ TEM tensile test was converted to obstacle strength (α). The average value of α was 0.59, and maximum value of α was 0.80. While the maximum value is close to the value of Orowan interaction, not a few values of α are much smaller than that of Orowan interaction. Based on the observation with in-situ TEM tensile experiment, we considered that cross-slip system is suitable as the interaction mechanism between oxide particles and dislocation in 9CrODS steel.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.nme.2016.06.014.

Reference

[1] A. Tavassoli, E. Diegele, R. Lindau, N. Luzginova, H. Tanigawa, J. Nucl. Mater. 455 (2014) 269–276.
[2] A.-A.F. Tavassoli, J. Nucl. Mater. 302 (2002) 73.
[3] Y. Li, T. Nagasaka, T. Muroga, A. Kimura, S. Ukai, Fus. Eng. Des. 86 (2011) 2495.
[4] B. Bako, D. Weygand, M. Samaras, J. Chen, M.A. Pouchon, P. Gumbsch, W. Hoffelner, Phil. Mag. 87 (2007) 3645–3656.
[5] Y. Xiang, D.J. Srolovitz, L.-T. Cheng, E. Weinan, Acta Metall. (2004) 1745.
[6] S.M.H. Haghighat, R Schäublin, Phil. Mag. Lett. 93 (2013) 575–582.
[7] T. Hatano, Phys. Rev. B 74 (2006) 020102(R).
[8] J. Malaplate, F. Mompiou, J.-L. Béchade, T.V.D. Berghe, M. Ratti, J. Nucl. Mater. 417 (2011) 205–208.
[9] M. Bartsch, A. Wasilkowska, A. Czyrska-Filemonowicz, U. Messerschmidt, Mater. Sci. Eng. 272 (1999) 152.
[10] D. Häussler, B. Reppich, M. Bartsch, U. Messerschmidt, Mater. Sci. Eng. A309-310 (2001) 500.
[11] D. Häussler, M. Bartsch, U. Messerschmidt, B. Reppich, Acta Mater. 49 (2001) 3647–3657.
[12] K. Tougou, K. Nogiwa, K. Tachikawa, K. Fukumoto, J. Nucl. Mater. 442 (2013) 350.
[13] K. Nogiwa, N. Nita, H. Matsui, J. Nucl. Mater. 367 (2007) 392.
[14] B. Reppich, Acta Mater. 46 (1) (1998) 61–67.
[15] D.J. Srolovitz, R.A. Petkovic-luton, M.J. Litton, Phil. Mag. A 48 (1983) 795–809.
[16] J.H. Schröder, E. Arzt, Scripta Metall. 19 (1985) 1129–1134.
[17] K. Nogiwa, a doctoral thesis, Tohoku University, (2006) Sendai, Japan.
[18] Y. Matsukawa, Y.N. Osetsky, R.E. Stoller, S.J. Zinkle, Phil. Mag. 88 (2008) 581.
[19] Y. Matsukawa, G. Liu, J. Nucl. Mater. 425 (2012) 54–59.
[20] S.-W. Kim, T. Shobu, S. Ohtsuka, T. Kaito, M. Inoue, M. Ohnuma, Mater. Trans. 50 (2009) 917–921.
[21] M. Klimiankou, R. Lindau, A. Moslang, J. Nucl. Mater. 329-333 (2004) 347.
[22] A. Yoshizawa, T. Fujita, F. Yoshida, H. Nakashima, Tetsu-to-Haganè (Japanese J. ISIJ) 82 (1996) 865.
[23] A.J.E. Foreman, M.J. Makin, Phil. Mag. 14 (1966) 911.
[24] U.F. Kocks, Phil. Mag. 13 (1966) 541.
[25] Y. Jiang, J.R. Smith, G.R. Odette, Acta Mater. 58 (2010) 1536.
[26] P.B. Hirsch, Canad. J. Phys. 45 (1967) 663.