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Change in the fractal dimension of the grain boundaries in pure Zn polycrystals during creep

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The effects of creep deformation on the shape of grain boundaries were investigated on pure Zn polycrystals at 373 K. The fractal dimension of the grain boundaries D ($1 \leq D \leq 2$) was estimated by the box-counting method. There was then discussion on the relationship between the value of D, the microstructures, and the creep or plastic strain in the deformed specimens of metallic materials.

The fractal dimension of the grain boundaries (D) increased with increasing the creep strain in pure Zn polycrystals, but the increase in the value of D levelled off when the creep strain exceeded about 0.30. The value of D decreased as the creep stress decreased. The increase in the value of D with the creep strain was correlated with the increase in the density of slip lines in the grains that formed the ledges and steps on grain boundaries. The value of D on the plane in parallel with the tensile axis was slightly larger than that on the plane transverse to the tensile axis. The mean shear strain on grain boundaries estimated from the value of D was correlated with the creep or plastic strain in the deformed specimens.

1. Introduction

Fractal geometry [1] has been successfully applied to the quantitative evaluation of complex patterns of fracture surfaces [2–10] and microstructures [11–16] in various kinds of materials. The shape of grains or grain boundaries has been estimated by the fractal dimension on the deformed or deformed and annealed metals and alloys [11–15] or heat-treated specimens of heat-resistant alloys [5, 6, 16]. Hornbogen [11] has revealed that in a Cu–Zn–Al alloy the fractal dimension of the grain boundaries D ($1 \leq D \leq 2$) increases when the amount of hot work is increased. Nishihara [12] and one of the present authors [15] have obtained similar results on the cold-worked specimens of pure iron. The increase of the fractal dimension with cold work can be correlated to the increase in the density of slip lines in the grains that form the ledges and steps on grain boundaries, where the slip lines meet [15].

It has been found [17] that in an austenite steel the fractal dimension of the grain boundaries increases as the creep strain increases. $Cr_{23}C_6$ carbide particles that precipitated on grain boundaries, however, seem to affect the change in the ruggedness of grain boundaries with the creep strain. Streitenberger *et al.* [14] have estimated the fractal dimension of the grain boundaries in the deformed specimen and in the deformed and annealed specimen of pure Zn polycrystals by the slit island method. They have found that the rugged grain boundaries formed by compressive deformation are gradually smoothed during recovery and recrystallization caused by subsequent annealing. Ramsey [18] has observed cell formation and slip lines in pure Zn polycrystals deformed at high temperatures. He has found that grain boundary migration occurs in the high-temperature deformation, and the migrating "front" clearly exhibits a rugged feature. Thus, the change in the shape of grain boundaries under stress seems to be different from that in a simple annealing.

In this study, the effects of creep deformation on the fractal dimension of the grain boundaries D ($1 \leq D \leq 2$) are investigated using pure Zn polycrystals at 373 K. The value of D is estimated by the box-counting method [1, 19] and is correlated to the creep strain and the microstructures such as slip lines. The mean shear strain on grain boundaries is then estimated from the value of the fractal dimension on the basis of the fractal geometry model. A discussion is also made on the relationship between the mean shear strain on grain boundaries and the creep or plastic strain in the deformed specimens of metallic materials.

2. Experimental

Commercial pure Zn polycrystals (99.99 wt % Zn) were used in this study. Forged bars 16 mm in diameter and 85 mm in length were solution heated for 3.6 ksec at 473 K and then air cooled. The average grain diameter of the as–heat-treated specimens was about 12 μm. The heat-treated specimens were machined into creep rupture test pieces 6 mm in diameter and 30 mm in gauge length. Creep experiments were carried out using these test pieces under the initial creep stresses of 14.7, 19.6, and 29.4 MPa at 373 K. For simplicity, the initial creep stress is hereafter referred to as "creep stress" or "stress." Test pieces were crept to a certain creep strain in the range from 0 to 0.877. The crept test pieces were longitudinally or transversely sectioned and then polished with diamond paste of 3 μm grain size. These specimens were finished by polishing with diamond paste of 0.25 μm and finally etched with 10% chloric acid in water [20].

Optical micrographs of grain boundaries in these specimens were taken at the magnification of 1000×. The slip line spacing was examined on the plane in parallel with the tensile direction (longitudinal direction) in the crept specimens. The fractal dimension of the grain boundaries in the two-dimensional plane D ($1 \leq D \leq 2$) was estimated by the box-counting method [1, 19]. In this study, the value of D was examined on the plane in parallel with, or transverse to, the tensile direction. Optical micrographs were taken into a personal computer (NEC PC-9821 xV13) by using an image scanner (Epson GT-9000) and then were processed using an installed software. The fractal dimension of a grain boundary D' was estimated by using software in the computer that was developed in our laboratory. These procedures were similar to those developed by X. W. Li *et al.* [9]. In the box-counting method, the data points of grain-boundary length L and scale length of the fractal analysis r were fitted to the following equation to obtain the fractal dimension D' by the regression analysis [1, 19]

$$\log_{10} L = \mathrm{k} + (1 - D') \log_{10} r, \qquad (1)$$

where k is a constant. Gokhale *et al.* [8] reported that there is a statistical variation in the fractal dimension D'. Therefore, in this study, as well as in the previous study [17], the fractal dimension of the grain boundaries D is obtained as an averaged value of D' over randomly chosen 20-grain boundaries in each specimen.

3. Experimental

3.1. Creep-rupture properties and microstructures

Fig. 1 shows the creep curves in the specimens of pure Zn polycrystals at 373 K. The creep curves involves the transient, the steady-state and the tertiary creep regimes. The elongation increases a little (from 0.606 to 0.877) with decreasing the creep stress. Transgranular fracture occurred in all the specimens tested at 373 K. Fig. 2 shows the optical micrographs in the specimens of pure Zn polycrystals crept under a stress of 29.4 MPa. The tensile direction is horizontal in Fig. 2a to e and vertical in Fig. 2f. Almost straight grain boundaries are visible in the undeformed specimen (Fig. 2a). Grain boundaries become increasingly serrated as the creep strain increases (Fig. 2b–2f). The slip lines become visible in the crept specimens when the creep strain exceeds about 0.10 (Fig. 2c–f). The grain shape remains almost unchanged even in the specimen of about 0.50 creep strain, as known from the comparison of Fig. 2e (the plane in parallel with the tensile axis) and Fig. 2f (the plane transverse to the tensile axis) with Fig. 2a.

Table I lists the mean slip line spacing and the slip line spacing range in the specimens of pure Zn polycrystals crept at 373 K. Both the mean slip line spacing and the slip line spacing range decrease with increasing the creep strain, whereas both values at the same

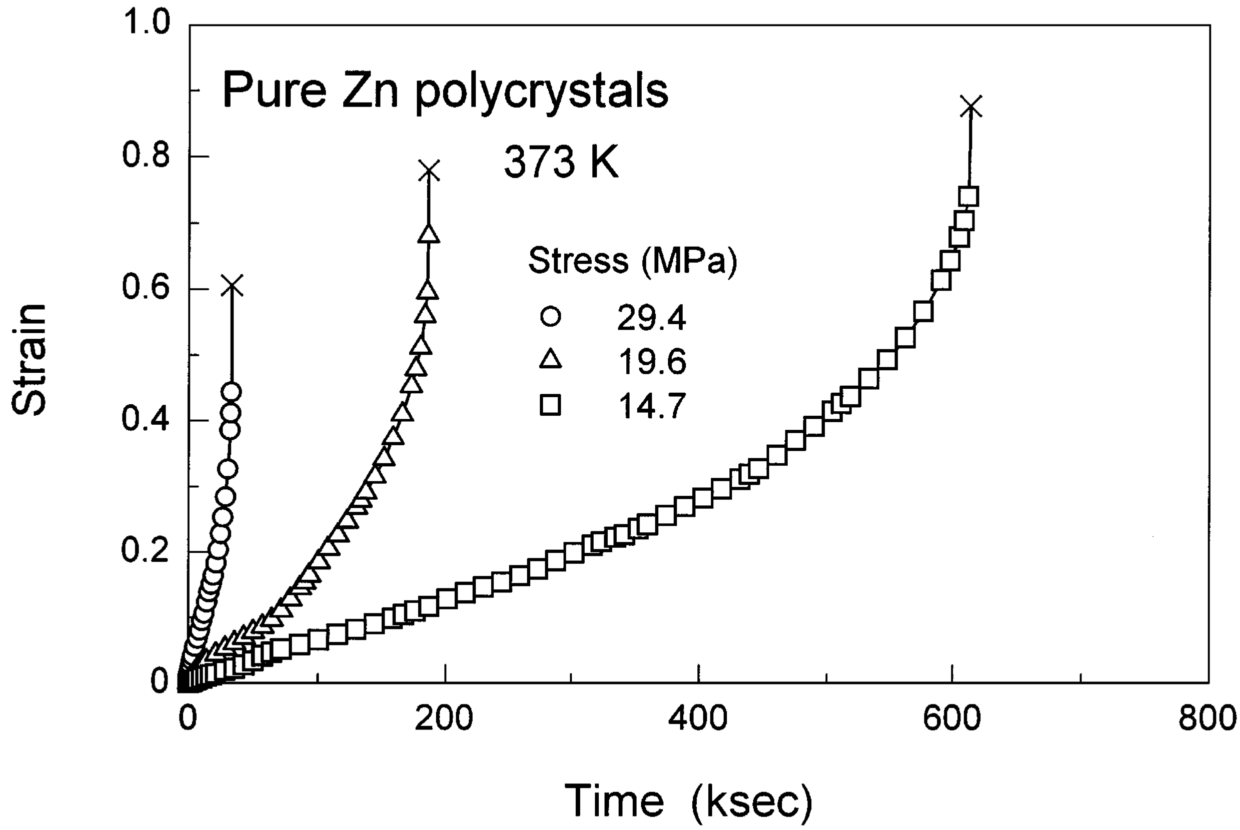

Figure 1 Creep curves in the specimens of pure Zn polycrystals at 373 K.

TABLE I The mean slip line spacing and the slip line spacing range in the specimens of pure Zn polycrystals crept at 373 K

Nominal creep strain \ Creep stress	14.7 MPa		19.6 MPa		29.4 MPa	
	Mean slip line spacing (10^{-6} m)	Slip line spacing range (10^{-6} m)	Mean slip line spacing (10^{-6} m)	Slip line spacing range (10^{-6} m)	Mean slip line spacing (10^{-6} m)	Slip line spacing range (10^{-6} m)
0.05	3.8	1.9–6.0	3.5	1.6–5.1	3.0	1.4–4.3
0.10	2.1	1.3–3.5	1.9	1.2–3.1	1.6	1.1–2.7
0.20	1.4	1.1–2.7	1.2	0.93–2.3	1.1	0.80–2.1
0.30	1.1	0.93–2.3	0.96	0.72–2.1	0.88	0.64–1.9
0.50	0.78	0.67–1.9	0.72	0.56–1.7	0.66	0.48–1.6
0.70	0.70	0.53–1.5	0.64	0.45–1.4	—	—
Rupture strain (ε_r)	0.64 ($\varepsilon_r = 0.877$)	0.40–1.3 ($\varepsilon_r = 0.877$)	0.60 ($\varepsilon_r = 0.817$)	0.40–1.1 ($\varepsilon_r = 0.817$)	0.59 ($\varepsilon_r = 0.606$)	0.43–1.3 ($\varepsilon_r = 0.606$)

ε_r, elongation

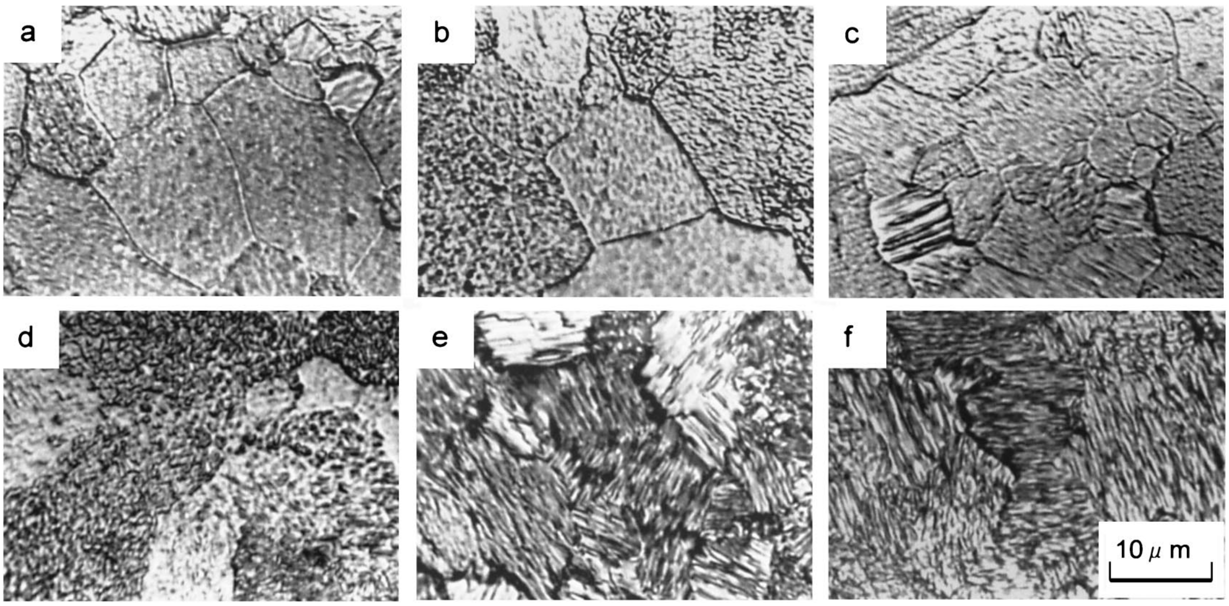

Figure 2 Optical micrographs in the specimens of pure Zn polycrystals crept under a stress of 29.4 MPa at 373 K. Creep strain is (a) 0, (b) 0.0514, (c) 0.100, (d) 0.205, (e) 0.519, and (f) 0.519. (a)–(e) Micrographs in the plane in parallel with the tensile axis. (f) Micrograph in the plane transverse to the tensile axis.

creep strain decrease as the creep stress increases. The slip line spacing in pure Zn polycrystals is in the range from 0.4×10^{-6} m to 6.0×10^{-6} m and is typically less than about 4.0×10^{-6} m. It has been reported that the scale range of the fractal analysis in which the grain boundaries exhibit a fractal nature is correlated with the slip line spacing [3, 15, 17]. In connection with this, the fractal dimension of the grain boundaries were estimated in the scale length (r) between 0.4×10^{-6} m and 4.0×10^{-6} m.

3.2. Effects of creep deformation on the fractal dimension of the grain boundaries

Fig. 3 shows a relationship between the length of a grain boundary (L) and the scale length of the fractal analysis (r) on the plane in parallel with the tensile axis in the crept specimens of pure Zn polycrystals. The slope of straight line gives the fractal dimension of a grain boundary D'. The value of D' increases with increasing the creep strain. Fig. 4 shows the change in the fractal dimension of the grain boundaries (the mean value of D' averaged over 20 grain boundaries) D, with the creep strain in the specimens crept under a stress of 29.4 MPa at 373 K. The value of D increases from about 1.08 to about 1.25 with increasing the creep strain. The value of D on the plane in parallel with the tensile axis is a little larger than that in the plane transverse to the tensile axis at the same creep strain [17]. The increase in the value of D with the creep strain is small when the creep strain exceeds about 0.30.

Fig. 5 shows the change in the fractal dimension of the grain boundaries D, with the creep strain in the specimens of pure Zn polycrystals crept at 373 K (on the plane in parallel with the tensile axis). The value of D increases with the creep strain and decreases a little with decreasing the creep stress. The increase in the value of D with the creep strain is small, especially under the lowest creep stress, when the creep strain is more than about 0.30. Fig. 6 shows schematically the grain boundary serration formed by slip within grains. As reported previously [15, 17], the ledges and steps are formed where slip lines meet grain boundaries, and the increase in the grain boundary roughness leads to the increase of the fractal dimension of the grain boundaries. The grain boundary serration during creep may also be affected by the grain boundary sliding [21–23]

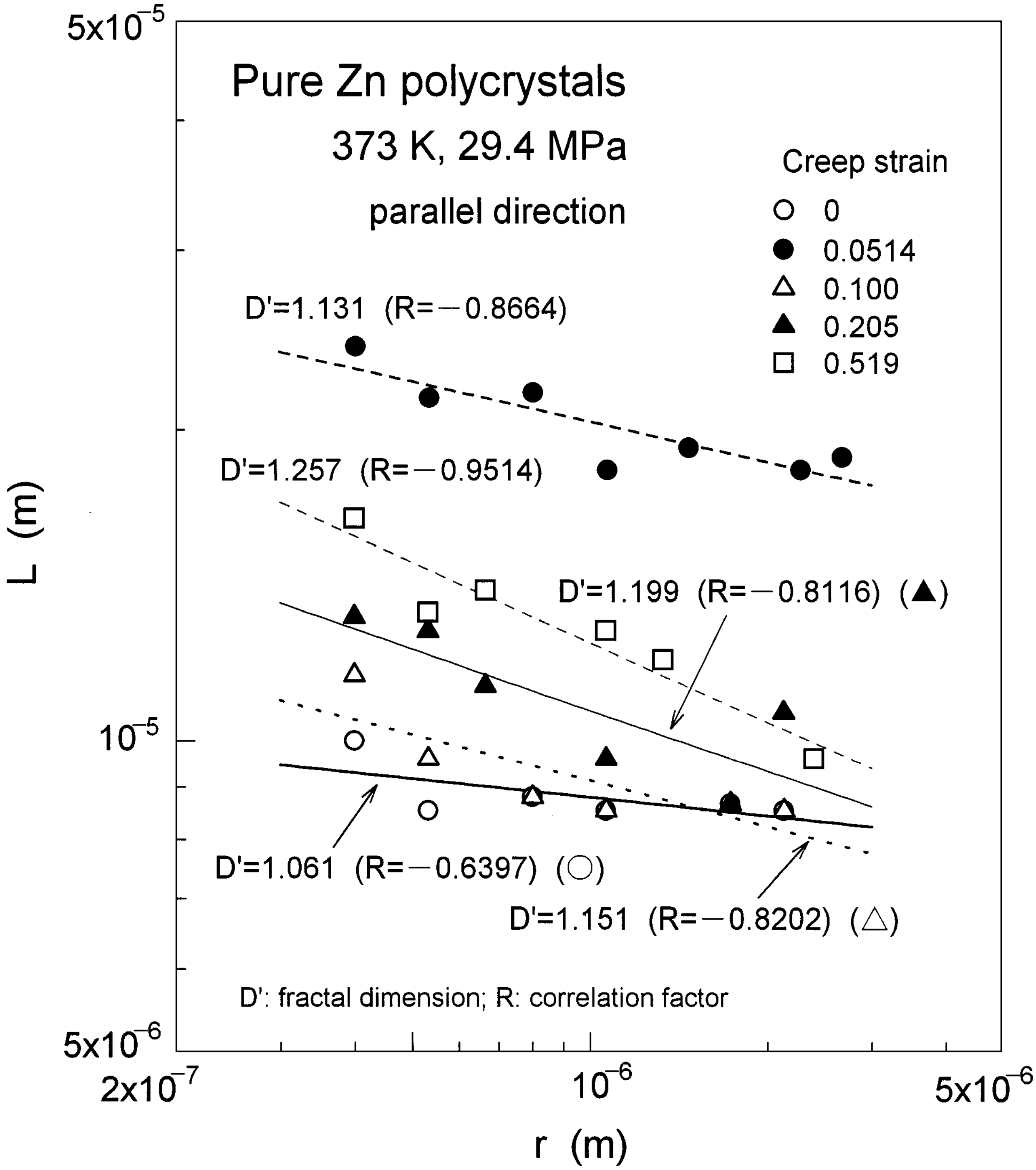

Figure 3 Relationship between the length of a grain boundary (L) and the length of the fractal analysis (r) on the plane in parallel with the tensile axis in the crept specimens of pure Zn polycrystals crept under a stress of 29.4 MPa at 373 K.

or by diffusion of atoms along or in the vicinity of grain boundaries [22–24]. Further, grain growth was observed in the specimens of pure Zn polycrystals during creep [18]. Fig. 7 shows the change in the number of grains in the specimens of pure Zn polycrystals crept at 373 K. The number of grains decreases with time of creep loading not only on the plane in parallel with the tensile axis but also on that transverse to the tensile axis in the crept specimens (29.4 MPa). This result cannot be explained by the change in the shape of grains caused by creep deformation [17] because the shape of grains is almost unchanged with the creep deformation (Fig. 2).

3.3. Variation in grain boundary roughness in the crept specimens

There is a certain variation in the roughness of grain boundaries in the crept specimens. Fig. 8 shows the fractal dimension of the grain boundaries D, and the standard deviation of D in the crept specimens of pure Zn polycrystals at 373 K (on the plane in parallel with the tensile axis). The standard deviation increases from about 0.047 in the undeformed specimen to more than about 0.090 in that of about 0.30 creep strain under both stresses of 14.7 and 29.4 MPa as the creep strain increases, but the standard deviation slightly decreases with the creep strain above about 0.50 creep strain.

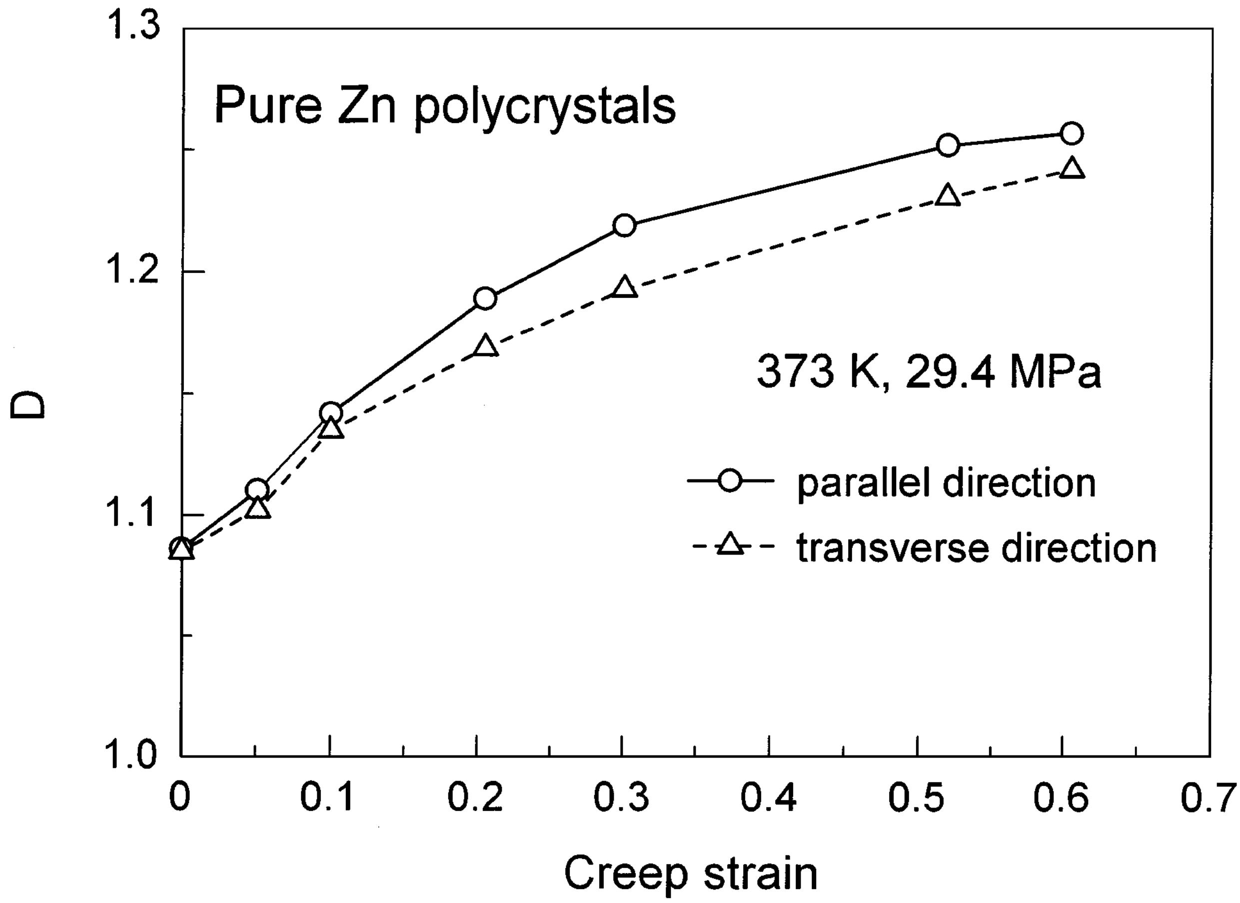

Figure 4 Change in the fractal dimension of the grain boundaries *D* with the creep strain in the specimens of pure Zn polycrystals crept under a stress of 29.4 MPa at 373 K.

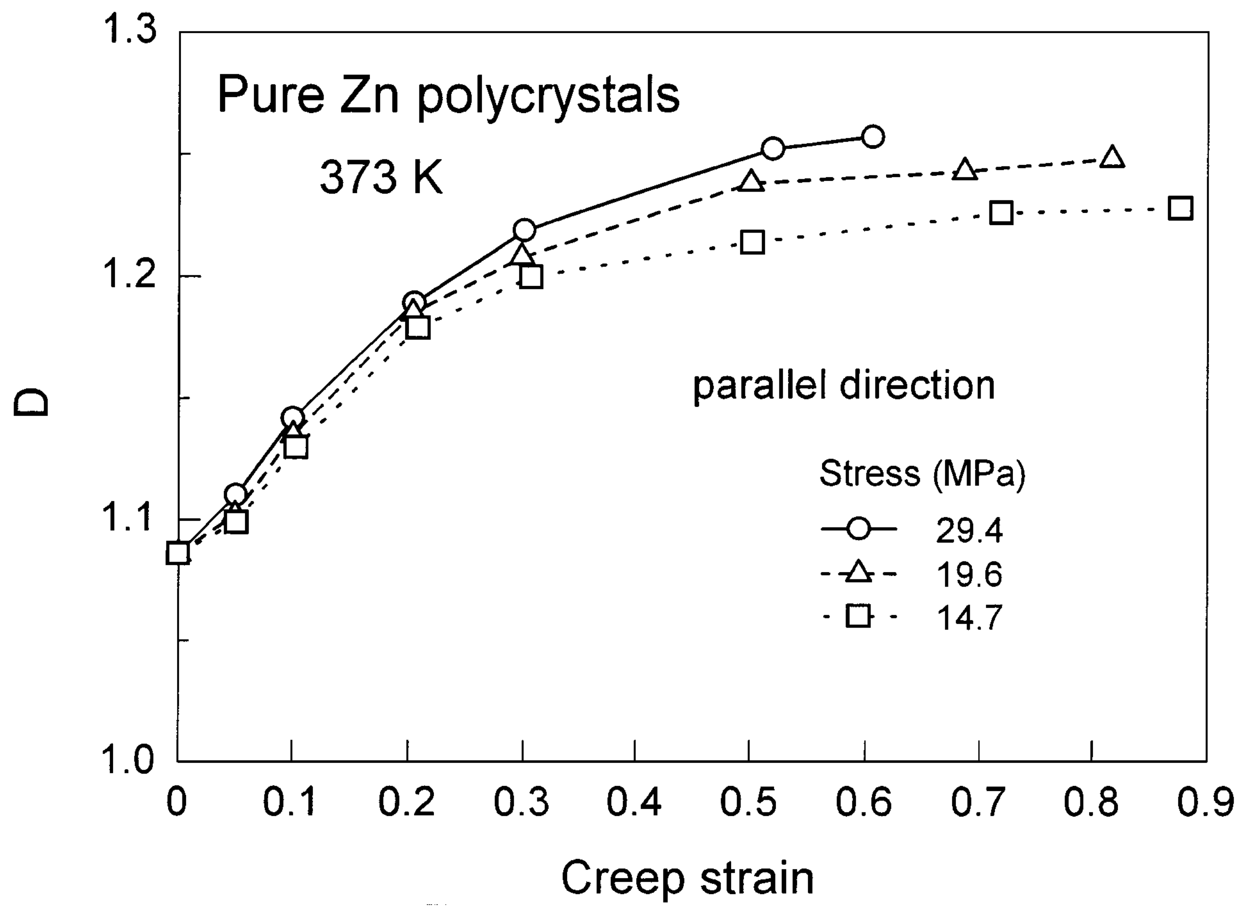

Figure 5 Change in the fractal dimension of the grain boundaries *D* with the creep strain in the specimens of pure Zn polycrystals crept at 373 K (on the plane in parallel with the tensile axis).

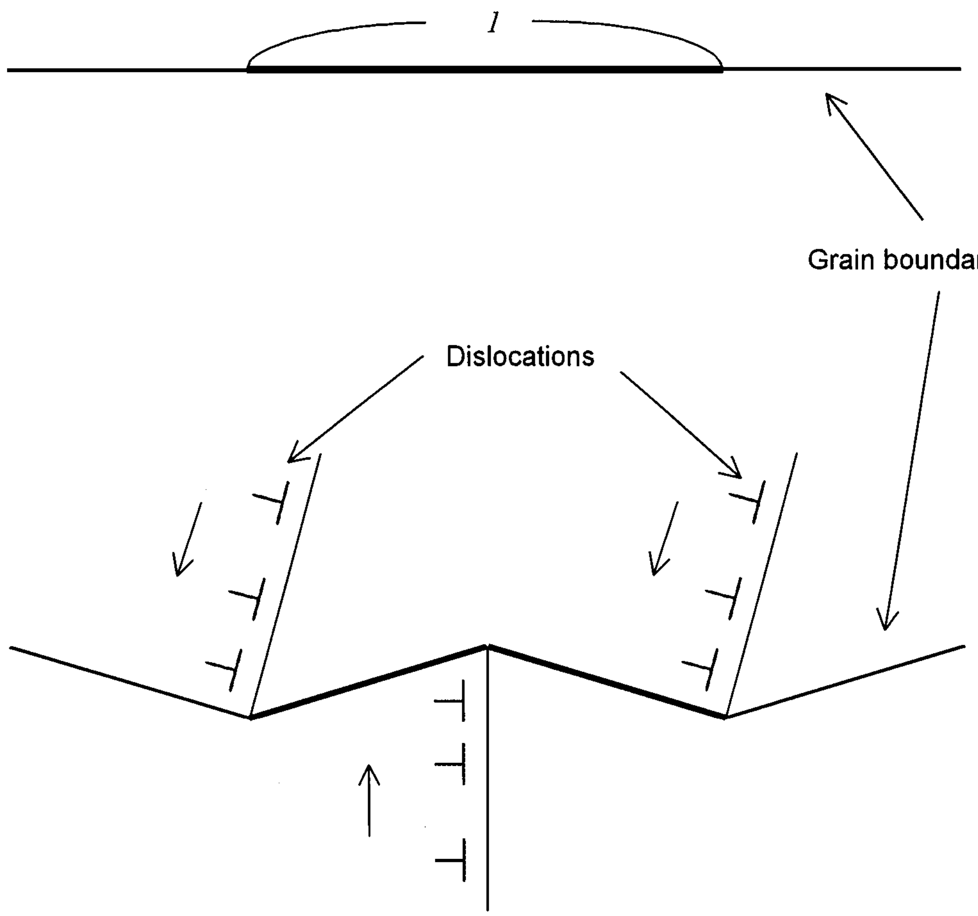

Figure 6 Schematic illustration of the grain-boundary serration formed by slip within grains.

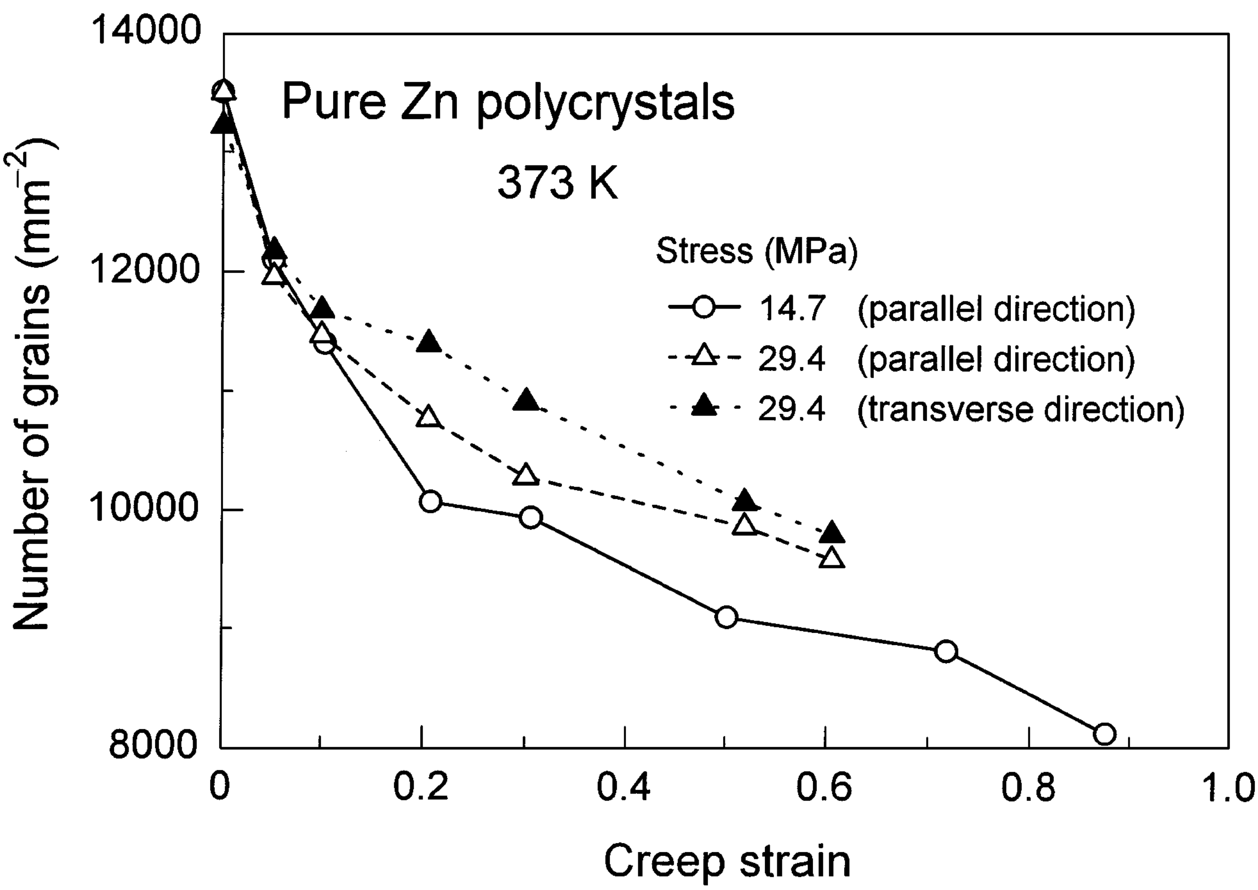

Figure 7 Change in the number of grains per unit area in the specimens of pure Zn polycrystals during creep at 373 K.

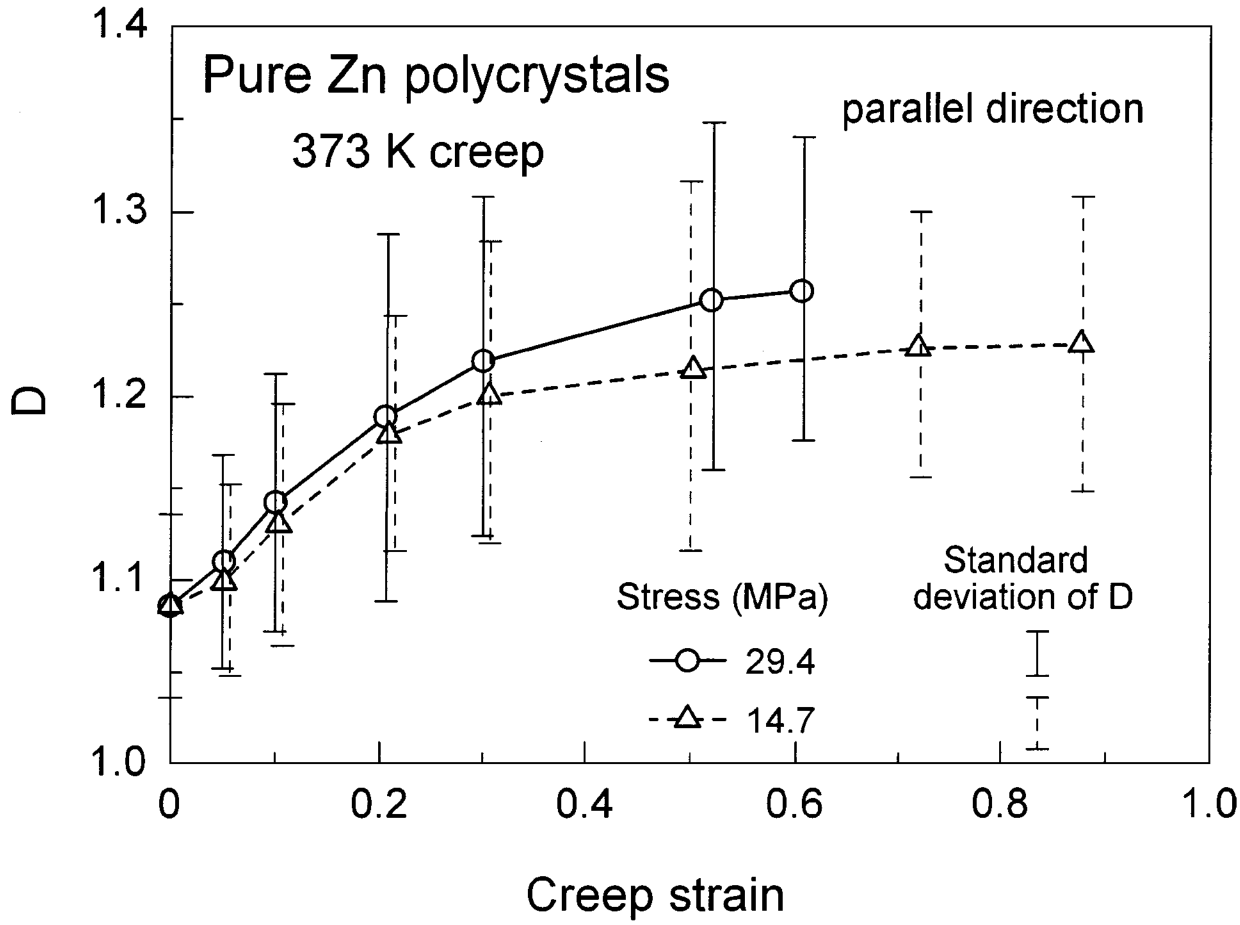

Figure 8 The fractal dimension of the grain boundaries *D* and the standard deviation of *D* in the crept specimens of pure Zn polycrystals at 373 K (on the plane in parallel with the tensile axis).

The results may indicate that some grains are deformed more extensively than others are [25] and that the difference in the creep deformation between these grains increases as the creep strain increases to about 0.30. The creep strain of about 0.30 corresponds to the strain above which the increase in the fractal dimension with the creep strain levels off in the specimens of pure Zn polycrystals. Rotation of crystallographic orientation may alter the relative ease of slip in each grain during creep [25, 26] and may reduce the difference in the creep strain between the grains. A similar result is obtained on the cold-worked specimens of pure iron polycrystals [15].

4. Discussion

4.1. Effects of microstructural change on grain boundary roughness

The fractal dimension of the grain boundaries (*D*) increases with increasing the creep strain but levels off above about 0.30 creep strain, and the fractal dimension decreases with decreasing the creep stress (see Figs 4 and 5). These results are associated with the change in the density of slip lines (see Table I). The grain boundary serration in the creep deformation can be correlated to the slip lines in the grains that form the ledges and steps on grain boundaries [15, 17]. As described in the previous section, grain growth occurred in the specimens of pure Zn polycrystals during creep at 373 K. Ramsey [18] has revealed that the grain boundary migration occurs in the high-temperature deformation of pure Zn polycrystals and that the grain boundaries show the ruggedness of the migrating front. Streitenberger *et al.* [14], however, have reported that in pure Zn polycrystals the fractal dimension of the grain boundaries increases with compressive deformation but decreases in the grain growth process caused by annealing after the deformation. The grain growth is generally accompanied by grain boundary migration [27], which may reduce the ruggedness of grain boundaries. Further, the grain boundary ruggedness may be decreased by grain boundary sliding [21, 22] or diffusion of atoms near grain boundaries [21, 23, 24]. Both effects of grain boundary sliding and diffusional recovery are remarkable under the lower stresses [21–24], as shown by the experimental results in this study (see Fig. 5) and SUS304 steel [17].

Ramsey [18] has also reported that the grain boundaries become rugged because of the slip and the cell formation in the grains during creep. Cell size in metallic materials is generally stress dependent and shows a constant value in the steady-state creep regime [28, 29]. Cell size seems to be at least more than a few micrometers in size in the specimens of pure Zn polycrystals deformed in the temperature range from 294 to 473 K [18], and this cell size lies close to the upper limit of the scale length of the fractal analysis (4.0×10^{-6} m) in this study. Therefore, the increase in the fractal dimension with the creep strain cannot be directly correlated to the cell formation. Dislocations that move to grain boundaries may be continuously supplied from the interior

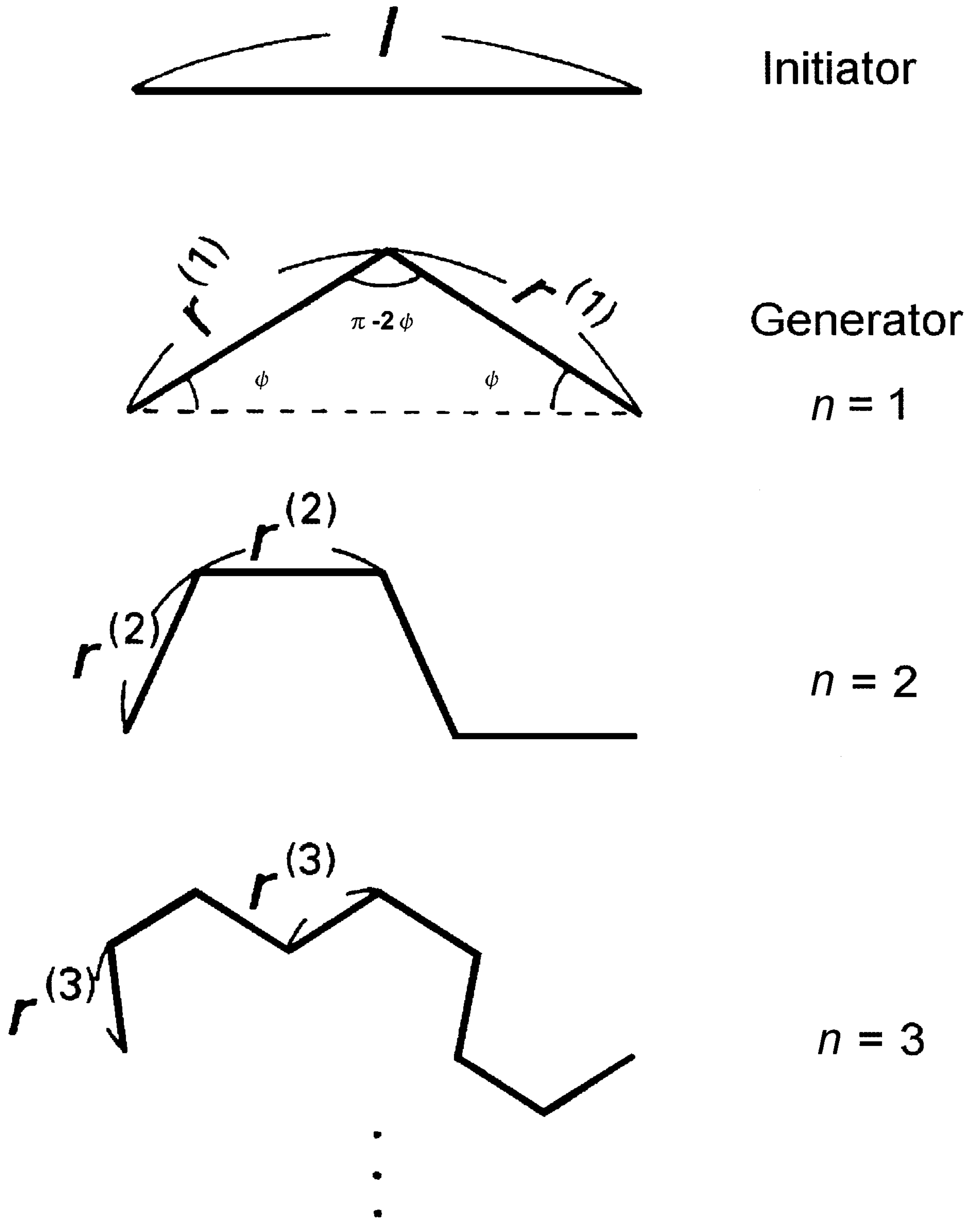

Figure 9 Schematic illustration of generating a serrated grain boundary.

of the grains, and the grain boundary roughening by slip may prevail the smoothing effects by grain boundary migration, grain boundary sliding, or diffusional recovery near grain boundaries during creep at least up to 0.30 creep strain (see Fig. 6).

4.2. Relations between the mean shear strain on grain boundaries and the creep or plastic strain

Let us consider a modelled grain boundary in a two-dimensional plane on the basis of the fractal geometry. Fig. 9 shows a schematic illustration of generating serrated grain boundaries. This model is similar to that of indentation crack [30]. For simplicity, it is assumed that the initiator is a straight line of the length l, and the generator is a concave line with an open angle $\pi - 2\phi$ in this model. The value of l can be correlated to the maximum value of slip line spacing or to the maximum scale length of the fractal analysis below which the grain boundaries exhibit a fractal nature. One can easily find the length of a line segment, $r^{(n)}$, at the nth generation ($n \geq 1$) from the geometrical consideration. At the first generation, the number of line segments is

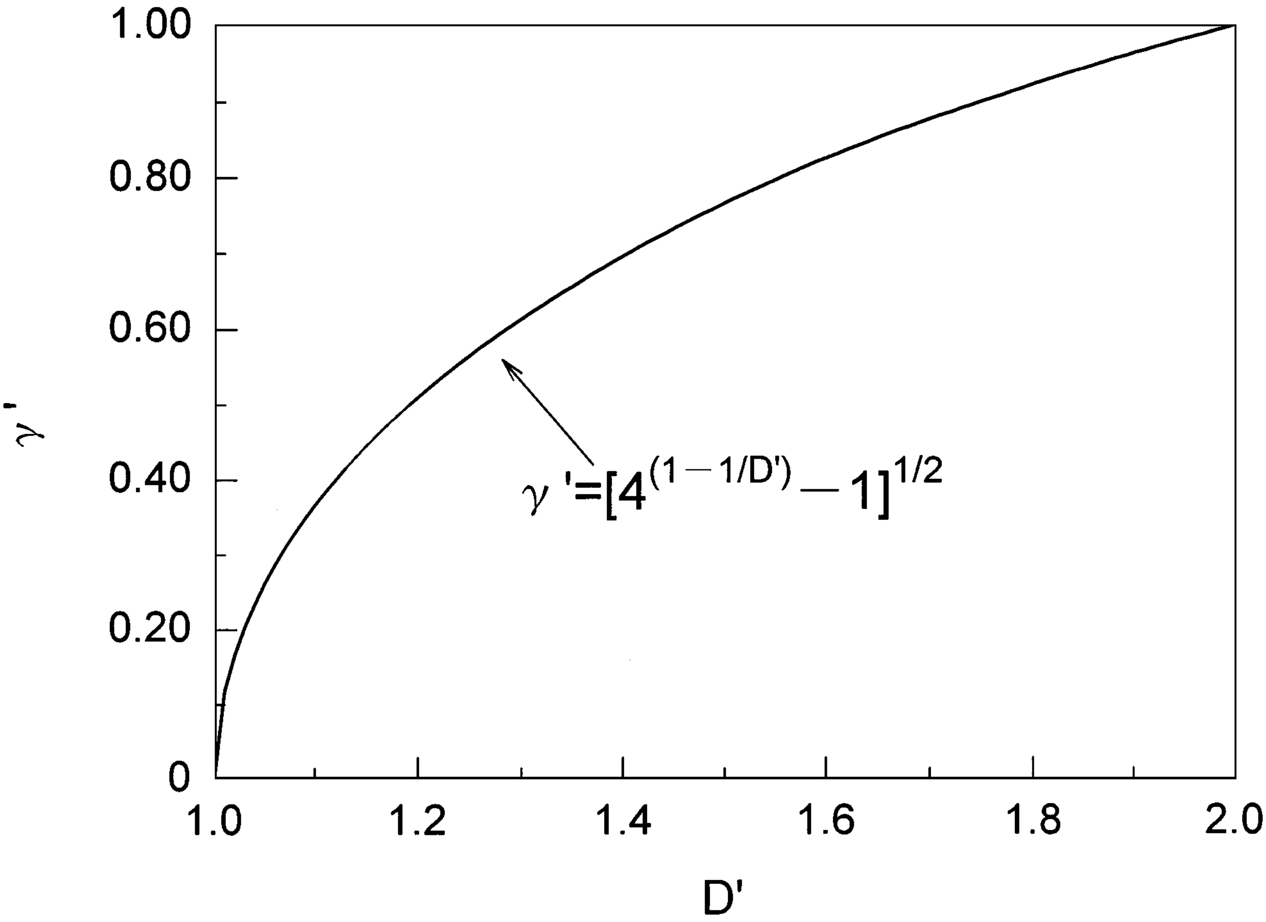

Figure 10 Relationship between the shear strain γ' and the fractal dimension D' of a modelled grain boundary.

two, and the length of a line segment, $r^{(1)}$, is given by

$$r^{(1)} = l/(2\cos\phi), \quad (2)$$

where $0 \leq \phi \leq \pi/4$. At the second generation, the number of line segments is 4 and the value of $r^{(2)}$ is expressed as

$$r^{(2)} = r^{(1)}/(2\cos\phi) = l/(2\cos\phi)^2 \quad (3)$$

At the mth generation, the number of line segments is 2^m and the value of $r^{(m)}$ is given by

$$r^{(m)} = l/(2\cos\phi)^m \quad (4)$$

Therefore, the fractal dimension of a modelled grain boundary (see Fig. 9) D' $(1 \leq D' \leq 2)$ is expressed as

$$D' = ln\, 2/ln(2\cos\phi) \quad (5)$$

and

$$\cos\phi = 2^{1/D'-1} \quad (6)$$

As illustrated in Fig. 6, the grain boundary serration is formed by the dislocation motion on slip planes. The shear strain, γ', on a grain boundary produced by slip can be correlated to the angle ϕ in Fig. 9 such that

$$\gamma' = \tan\phi \quad (7)$$

Using Equations 6 and 7, one can obtain the following equation:

$$\gamma' = \left[4^{(1-1/D')} - 1\right]^{1/2} \quad (8)$$

Fig. 10 shows the result of numerical calculation using Equation 8. The actual grain boundaries have a fractal nature in statistical meaning and the cascade (the generating process) of the actual serrated grain boundaries may be much more complex than the present model is. Equation 8, however, may give a rough estimation of the shear strain from the fractal dimension of a grain boundary. If D'_0 is the fractal dimension of a grain boundary before creep deformation, the initial value of γ', γ'_0, of this grain boundary is expressed as

$$\gamma'_0 = \left[4^{(1-1/D'_0)} - 1\right]^{1/2} \quad (9)$$

Therefore, the shear strain on this grain boundary produced by creep or plastic deformation, γ'_p, is given by

$$\gamma'_p = \left[4^{(1-1/D')} - 1\right]^{1/2} - \left[4^{(1-1/D'_0)} - 1\right]^{1/2}$$
$$= \left[4^{(1-1/D')} - 1\right]^{1/2} - \gamma'_0 \quad (10)$$

The value of γ'_p can be estimated on each grain boundary if the initial value of the fractal dimension, D'_0, is known. As described previously, the fractal dimension of the grain boundaries D in this study was

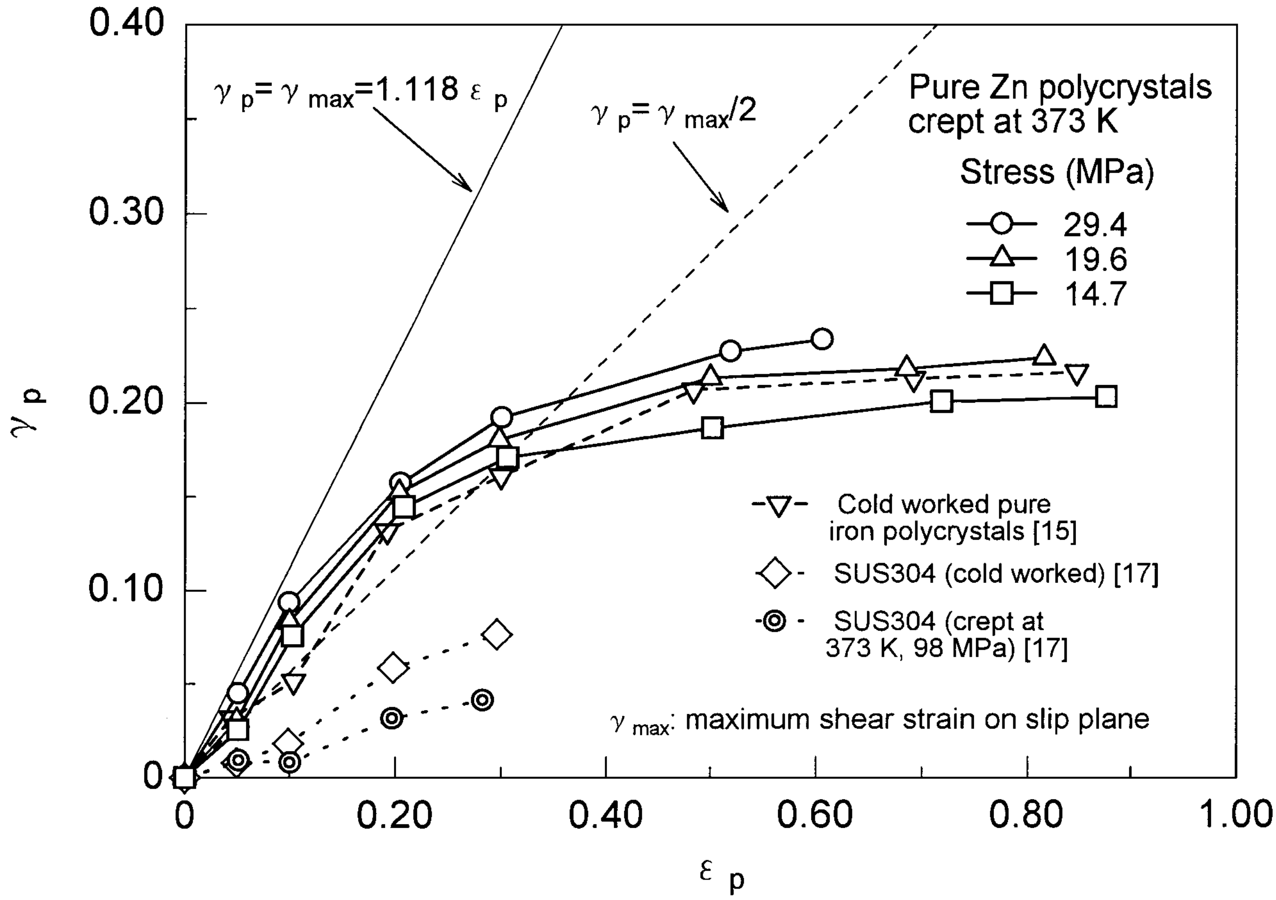

Figure 11 Relationship between the mean shear strain on grain boundaries γ_p, estimated from the fractal dimension of the grain boundaries (D) and the creep or plastic strain ε_p in the deformed specimens of metallic materials.

estimated as the mean value of the fractal dimension (D') over 20 grain boundaries in each specimen. Therefore, putting $D' = D$ and $D'_0 = D_0$ in Equation 10, one can estimate the mean value of the shear strain, γ_p, instead of γ'_p, from the value of D.

Fig. 11 shows the relationship between the mean shear strain on grain boundaries, γ_p, estimated from the fractal dimension of the grain boundaries (D) and the creep or plastic strain, ε_p, in the deformed specimens of metallic materials. If only a single slip system works on one slip plane inclined by about 19.5° to the tensile axis in the uniform creep or uniform plastic deformation, the maximum shear strain, γ_{max}, on that plane is about $1.118\varepsilon_p$. Most of the data points up to about 0.30 creep strain lie between a solid line ($\gamma_p = \gamma_{max} = 1.118\varepsilon_p$) and a broken line ($\gamma_p = \gamma_{max}/2$) in the crept specimens of pure Zn polycrystals. The value of γ_p slightly decreases with decreasing the creep stress, since the effects of grain boundary sliding and diffusional recovery is remarkable under the lower stresses (see section 4.1). The value of γ_p does not largely increase above about 0.30 creep strain (ε_p). As described in section 3.3, this may be explained by rotation of crystallographic orientation [25, 26]. A similar result is obtained on the cold-worked pure iron polycrystals [15].

The data points of pure Zn polycrystals and SUS304 steel in this figure are those obtained on the plane in parallel with the tensile axis. Almost the same tendency was observed on the data points obtained on the plane transverse to the tensile axis. Very small values of γ_p are observed in the crept specimens and in the cold-worked ones of SUS304 steel, however [17]. This may be explained by $Cr_{23}C_6$ carbide particles on grain boundaries that make the grain boundary serration difficult during creep or plastic deformation.

The values of γ_p estimated from the fractal dimension of the grain boundaries (D) are the mean shear strain averaged over more than 20 grain boundaries [15, 17]. Many slip systems generally work in the creep or plastic deformation of polycrystalline materials, and these slip systems have different orientations with respect to tensile or compressive stress axis [25]. Therefore, the rugged grain boundaries may actually be produced by dislocation motion on many slip systems [25], and this process may not be so simple as the model shown in Fig. 9. As Lim and Raj [31] have reported, dislocation reactions at grain boundaries may affect the grain boundary roughness also in the crept specimens. Nevertheless, it is interesting to note that there is a correlation between the mean shear strain γ_p, estimated from the fractal dimension (D) and the creep or plastic strain, ε_p, at least up to about 0.30 strain, in polycrystalline materials. As described in section 3.3, there is a boundary to boundary variation in the value of the fractal dimension in the crept specimens (see Fig. 8). If one monitors the change in the fractal dimension of each grain boundary (D') during creep or plastic deformation, one can know more precisely the shear strain on each grain boundary, γ'_p, and the difference in shear strain between grain boundaries.

5. Conclusions

The effects of creep deformation on the fractal dimension of the grain boundaries (D, $1 \leq D \leq 2$) were investigated using pure Zn polycrystals at 373 K. Discussion was then made on the relationship between the value of D, the microstructures such as slip lines, and the creep or plastic strain in the deformed specimens of metallic materials. The results obtained are summarized as follows.

1. The fractal dimension of the grain boundaries (D) increased with increasing the creep strain in pure Zn polycrystals, but the increase in the value of D with the creep strain levelled off above about 0.30 creep strain. The value of D decreased as the creep stress decreased.

2. The increase in the value of D with the creep strain was correlated with the slip lines in the grains that formed the ledges and steps on grain boundaries in pure Zn polycrystals during creep. The value of D examined on the plane in parallel with the tensile axis was slightly larger than that obtained on the plane transverse to the tensile axis.

3. On the basis of the fractal model of a serrated grain boundary, the mean shear strain γ_p on grain boundaries was estimated from the fractal dimension of the grain boundaries (D) on the crept or plastically deformed specimens of metallic materials. A correlation was found between the value of γ_p and the creep or plastic strain ε_p in these materials.

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