Transition of Deformation Behavior of Fe-2.1mol%Mo Alloy in the Power-law Creep Regime*

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Synopsis

The existence of a sharp transition of high-temperature creep behavior in bcc solid solutions has been confirmed by studying steady-state creep characteristics of an Fe-2.1mol% (3.56 mass%) Mo alloy in the temperature range T from 1 100 to 1 200 K under the tensile stress range σ from 4 to 50 MPa. In the lower stress region, the creep characteristics are those typical in creep controlled by viscously-gliding dislocations which drag the solute (Cottrell) atmosphere. Steady-state creep-rates $\dot{\epsilon}_s$ can be represented by the equation, $\dot{\epsilon}_s^M = 1.21 \times 10^{-2} (Gb/kT) D_{Mo} (\sigma/G)^{3.45}$ (G : the shear modulus, b : Burgers vector, D_{Mo} : the diffusion coefficient of Mo). In the higher stress region, the creep characteristics are different from those observed in the lower stress region and $\dot{\epsilon}_s^H = 5.7 \times 10^{-7} (Gb/kT) \cdot D_{Fe} (\sigma/G)^{5.3}$ (D_{Fe} : the diffusion coefficient of Fe). Results obtained in conventional tensile test at 1 100 K with strain-rates ranging from $3 \times 10^{-5} s^{-1}$ to $3 \times 10^{-3} s^{-1}$ coincide well with creep behavior. The solute atmosphere seems to be broken at some locations under high stresses and a part of dislocations can move as a bare dislocation in the matrix where immobile solute atoms are distributed randomly. The transition observed in this investigation is essentially the same as the upper transition, i.e., the transition between regions M and H, observed in Al-Mg (fcc) alloys and α -Ti-Al (hcp) alloys.

Key words: creep; iron-molybdenum alloy; solid solution; transition of deformation behavior; Cottrell atmosphere.

I. Introduction

Some concentrated alloys with solutes, atomic radii of which are greatly different from that of the solvent, show class I behavior in creep at high temperatures.¹⁾ Aluminum-magnesium solid solutions can be cited as one of typical examples, in which creep deformation shows characteristic behavior of class I. For examples, the stress exponent n of steady-state creep-rates is close to 3, no instantaneous strain occurs upon loading, primary creep is of inverted or sigmoidal types, no well-defined sub-boundaries are formed.²⁾

Class I behavior, however, can be observed over a limited condition: even in the power-law creep regime, n becomes about 5 (class II behavior) under the higher and the lower stresses.³⁻⁵⁾ The conditions of these transitions in creep behavior in Al-Mg alloys depend on temperature and the concentration of magnesium.⁵⁾

Alpha Fe-Mo alloy containing 6.3 mol% Mo has been known to behave as class I under conventional experimental conditions.⁶⁾ In dilute Fe-Mo alloys, however, some characteristic features of creep were reported to be different from those in dilute Al-Mg alloys. For examples, n decreases gradually from 5 to 3 with increasing the concentration of molybde-

num⁷⁾ and n increases monotonously with stress,⁸⁾ that is, no clear transition in creep behavior has been reported. Development of clear sub-boundaries has always been observed in Fe-Mo alloys⁶⁾ after creep straining, whereas no well-developed sub-boundaries are observed in Al-Mg alloys under creep conditions of typical class I behavior,⁹⁾ except under low stresses near the lower transition stress.¹⁰⁾

Therefore, the generality of the transitions in creep characteristics in power-law creep regime observed in Al-Mg alloys has not been confirmed yet. In the present investigation creep behavior, as well as conventional high-temperature tensile behavior, of a bcc Fe-Mo solid solution has been studied in detail to solve the question that creep characteristics of bcc Fe-Mo alloys are truly different from those of fcc Al-Mg alloys.

II. Experimental Procedure

1. Material

The alloy used in this study was an Fe-Mo solid solution containing 2.1 mol% (3.56 mass%) Mo. Main impurities (in mass ppm) were C: 150, Mn: 40, P: 40, S: 3, Cu: 10, Cr<30, Ni<40, Al: 50, O: 100 and N: 25. It was vacuum-melted and cast to a 50 mm-square billet, and then, hot-rolled at 1 370 ~ 1 230 K to bars of 20 mm diameter.

Threaded-end rod specimens were machined from the bars. The diameter and the length of the gage part were 3 or 6 mm and 15 or 20 mm, respectively. The specimens were annealed at 1 300 K for about 7 ks in an argon atmosphere to produce stable grains of 0.4 mm diameter.

2. Creep Test

Tensile creep tests under constant stress in an argon atmosphere were carried out with a simple inverted-type creep machine. Elongation of the specimen was detected from the relative displacement of the pull-rod and measured continuously with a linear differential transducer to 0.01 mm, which corresponds to about 5×10^{-4} true strain. Temperature was measured with a Pt/Pt-12.7 Rh thermocouple and controlled within ± 1 K, or in cases of long-term creep within ± 2 K.

The creep strain, strain-rate as well, is expressed as true strain ϵ throughout this report, i.e.,

$$\epsilon = \ln(1 + \Delta l/l_0)$$

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where, l_0 : the initial length of a specimen
 Δl : the elongation.

3. Tensile Test

Conventional tensile tests with constant nominal strain-rates were carried out in an argon atmosphere with a gear-driven testing machine. Temperature was measured with a Pt/Pt-12.7 Rh thermocouple and controlled within ± 1 K.

The stress is expressed as true stress σ at a constant true strain-rate by correcting raw data with the equation,

$$\sigma = \sigma_0(1 + \Delta l/l_0)^{1+1/n}$$

where, σ_0 : the engineering stress
 n : the stress exponent in the $\dot{\epsilon}$ - σ relationship.

III. Results

1. Creep Curves

Creep tests were done at 1 100, 1 155 and 1 200 K under 4~50 MPa. The total test duration ranged from 60 s to 1.8 Ms and the resulting steady-state creep-rate ranged from $1 \times 10^{-7} \text{ s}^{-1}$ to $1 \times 10^{-2} \text{ s}^{-1}$. Examples of some typical creep curves are shown in Fig. 1. All creep data are listed in Table 1.

Sigmoidal primary creep curves appear under a wide range of stress. The degree of sigmoidal, however, was relatively small: ratios of the maximum creep-rate at the inflection point $\dot{\epsilon}_{\text{max}}$ and the steady-state creep-rate, $\dot{\epsilon}_{\text{max}}/\dot{\epsilon}_s$, are 1.7 at most. Similar results were already reported on the alloys of this system.¹¹⁾

Normal primary creep curves appear under the higher stresses. The transition condition in types of primary creep is closely correlated with the transition in steady-state creep behavior described later.

Fig. 1. Typical creep curves of Fe-2.1mol%Mo alloy.

Table 1. Creep characteristics of Fe-2.1mol%Mo alloy.

<i>T</i> (K)	σ (MPa)	Inflection point			Steady-state stage					$\frac{\dot{\epsilon}_{\text{max}}}{\dot{\epsilon}_s}$
		$\dot{\epsilon}_{\text{max}}$ (10 ⁻²)	t_{max} (ks)	$\dot{\epsilon}_{\text{max}}$ (s ⁻¹)	$\dot{\epsilon}_s$ (10 ⁻²)	t_s (ks)	$\dot{\epsilon}_s$ (s ⁻¹)	ϵ_t (10 ⁻²)	t_t (ks)	
1 200	35				15	0.025	4.6×10^{-3}	26	0.05	
	30				12	0.09	1.23×10^{-3}	21	0.16	
	25	~3	0.02	1.20×10^{-3}	16	0.17	7.4×10^{-4}	28	0.34	1.63
	20	~4	0.06	5.6×10^{-4}	21	0.49	3.3×10^{-4}	29	0.74	1.73
	14.0	~4	0.30	1.71×10^{-4}	21	1.60	1.06×10^{-4}	29	2.3	1.60
	10.0	~5	0.90	4.9×10^{-5}	22	5.4	3.3×10^{-5}	28	7.4	1.50
	10.0	~5	0.80	5.3×10^{-5}	24	5.5	3.3×10^{-5}	29	7.2	1.60
	7.0	~4	2.0	1.43×10^{-5}	23	18.0	1.01×10^{-5}	32	28	1.42
	5.0	~5	10.0	4.4×10^{-6}	20	54	3.0×10^{-6}	28	78	1.44
1 155	4.0	~5	23	1.58×10^{-6}	20	140	1.32×10^{-6}	32	230	1.19
	40				15	0.06	1.75×10^{-3}	21	0.10	
	35				19	0.18	8.6×10^{-4}	28	0.28	
	30	~4	0.04	8.4×10^{-4}	19	0.29	5.4×10^{-4}	26	0.41	1.55
	20	~4	0.20	1.63×10^{-4}	20	1.60	1.04×10^{-4}	32	2.8	1.57
	14.0	~5	0.80	4.1×10^{-5}	24	7.0	2.8×10^{-5}	32	10.0	1.47
	10.0	~4	2.0	1.56×10^{-5}	24	18.0	1.08×10^{-5}	28	22	1.44
	5.0	~4	3.7	1.10×10^{-6}	21	200	9.3×10^{-7}	30	300	1.19
	50				15	0.09	1.24×10^{-3}	24	0.16	
1 100	45				13	0.15	6.6×10^{-4}	22	0.29	
	40				15	0.38	3.3×10^{-4}	24	0.66	
	35				17	0.75	1.87×10^{-4}	28	1.30	
	30	~4	0.20	1.81×10^{-4}	16	1.20	1.14×10^{-4}	29	2.3	1.59
	25	~5	0.40	9.2×10^{-5}	16	2.0	5.8×10^{-5}	32	4.8	1.58
	20	~3	1.00	3.1×10^{-5}	18	7.0	2.2×10^{-5}	28	12.0	1.44
	17.0	~5	2.2	1.77×10^{-5}	21	13.0	1.27×10^{-5}	30	20	1.39
	14.0	~4	3.0	1.08×10^{-5}	24	28	6.8×10^{-6}	32	40	1.60
	12.0	~4	6.0	5.4×10^{-6}	23	46	4.0×10^{-6}	32	70	1.33
	10.0	~3	10.0	2.6×10^{-6}	15	65	2.0×10^{-6}	26	120	1.29
	8.0	~4	20	1.02×10^{-6}	18	190	8.5×10^{-7}	29	330	1.19
	5.0	~5	190	1.98×10^{-7}	15	880	1.58×10^{-7}	24	1 400	1.25

2. Stress-Strain Curves

Conventional tensile tests were done at 1 100 K in the strain-rate ranging from $3 \times 10^{-5} \text{ s}^{-1}$ to $3 \times 10^{-3} \text{ s}^{-1}$. Two types of curves were obtained as seen in Fig. 2.

In lower strain-rate tests a yield drop, *i.e.*, so-called high-temperature yielding,¹²⁾ appeared before normal stress-increase occurs, though the amount of drop is not pronounced. In higher strain-rate tests no such yielding could be observed. The transition in types of stress-strain curves, *i.e.*, the existence of high-temperature yielding, is well correlated with types of the primary creep stage as discussed later.

A plateau region appeared in stress-strain curves under all conditions in the present experiments and data are listed in Table 2. When a steady-state stage appears, there is no difference in the deformation characteristics between creep and conventional test.¹³⁾ Therefore, the relationship between true strain-rate and true stress in steady-state stage will be treated without differentiation by testing methods, hereafter.

3. Stress Dependence of $\dot{\epsilon}_s$

Steady-state stage was observed under all experimental conditions and steady-state creep-rate (in true strain) $\dot{\epsilon}_s$ could be determined. Steady-state creep characteristics have been analyzed with the Dorn equation,

$$\dot{\epsilon}_s = AD \left[\frac{Gb}{kT} \right] \left[\frac{\sigma}{G} \right]^n \dots\dots\dots(1)$$

Fig. 2. Typical stress-strain curves of Fe-2.1mol%Mo alloy deformed at 1 100 K.

- where, D : the lattice diffusion coefficient
 G : the shear modulus
 b : the atomic diameter of the matrix
 k : Boltzmann constant
 T : temperature
 σ : the applied stress
 A, n : numerical constants.

In this report G 's are assumed to be independent of the addition of molybdenum and vary with the temperature: 49.2 GPa at 1 100 K, 46.9 GPa at 1 155 K, and 45.0 GPa at 1 200 K.¹⁴⁾

Steady-state strain-rate are shown in Fig. 3 as a function of stress, from which the existence of a transition in the gradient is obvious on each curve. The results are summarized in Table 3.

In the lower stress range, which will be called as region M hereafter, the gradient is small and the average value of n (the gradient) is obtained as 3.45. In the higher stress range, which will be called as region H hereafter, the gradient of curves is high and the average value of n is estimated as about 5.3. The value of n in each region is almost independent of temperature. The transition condition will be discussed later.

4. Temperature Dependence of $\dot{\epsilon}_s$

Creep tests were carried out at three temperatures

Solid marks: Sigmoidal primary or high-temperature yielding
Open marks: Normal primary or normal σ - ϵ curves

Fig. 3. Steady-state strain-rates as a function of stress in creep tests and steady-state stress as a function of strain rate in conventional tensile tests (inverted triangles).

Table 2. Deformation characteristics in tensile tests of Fe-2.1mol%Mo alloy at 1 100 K.

Strain rate (s ⁻¹)	Proof stress* (MPa)	Upper yield		Lower yield		Steady-state stage		Fracture ϵ_f
		σ_{uy} (MPa)	ϵ (10 ⁻²)	σ_{ly} (MPa)	ϵ (10 ⁻²)	σ_{max} (MPa)	ϵ	
2.2×10^{-3}	44.1					55.9	0.12~0.25	0.57
1.1×10^{-3}	42.0					48.8	0.16~0.31	0.62
4.1×10^{-4}	37.4					42.4	0.29~0.55	0.87
1.65×10^{-4}	29.5			32.2	3.7~8.3	35.3	0.32~0.52	0.97
3.3×10^{-5}	18.7	20.5	3.2	19.9	4.5~7.8	22.4	0.32~0.55	0.97

* Stress at the 0.2 % offset from the linearity in a load-elongation curve

Table 3. Steady-state characteristics in Fe-2.1 mol%Mo alloy.

Temp. (K)	Lower stress region		Transition stress σ_u (MPa) (σ_u/G)	Higher stress region	
	σ -range (MPa)	n		σ -range (MPa)	n
1 200	4~25	3.3 ₈	(~30)	—	—
1 155	5~20	3.3 ₇	31 (6.6×10^{-4})	35, 40	5.3
1 100	5~20	3.5 ₈	35 (7.2×10^{-4})	35~50	5.3 ₆
Average		3.45	(7.0×10^{-4})		5.3

over a range of stress as seen in Fig. 3. Temperature dependence of $\dot{\epsilon}_s$ is shown in Fig. 4 under selected stress levels.

It is obvious that the slopes are almost independent of the level of stress and the activation energy for creep Q_c can be evaluated with an equation of the Dorn type. The activation energy for creep in region M is evaluated as $Q_c^M = 281 \pm 5$ kJ/mol, which is close to that for diffusion of Mo in dilute α -Fe-Mo alloys, i.e., $Q_D^M = 273 \sim 250$ kJ/mol.^{15,16)}

The activation energy in region H, Q_c^H , could not be determined precisely, but it can be seen in Fig. 4 that Q_c^H takes a value similar to Q_c^M .

IV. Discussion

1. Generals

The creep characteristics of the lower stress range are those typical in creep controlled by the solute-atmosphere dragging²⁾; primary creep is of sigmoidal type, n is about 3, Q_c is close to that for Q_D of the solute. Values of $\dot{\epsilon}_s$ normalized with the diffusion coefficient of the solute D_{Mo} , the concentration of the solute N_{Mo} , and the shear modulus are plotted against the normalized stress in Fig. 5. Values of D_{Mo} were calculated by the equation,

$$D_{Mo}^*/m^2 s^{-1} = 2.63 \times 10^{-4} \exp \left[-\frac{244.8 \text{ kJ} \cdot \text{mol}^{-1}}{RT} \right]$$

.....(2)

which was reported by Nohara and Hirano¹⁵⁾ for $N_{Mo} = 0.0284$. Relationship between the steady-state strain-rate and the steady-state stress in region M is well represented by the following equation,

$$\dot{\epsilon}_s = 1.21 \times 10^{-2} \left[\frac{Gb}{kT} \right] D_{Mo} \left[\frac{\sigma}{G} \right]^{3.45}$$

.....(3)

or (see Fig. 5),

$$\dot{\epsilon}_s = 5.8 \times 10^{-1} \left[\frac{D_{Mo}}{N_{Mo}} \right] \left[\frac{Gb}{kT} \right] \left[\frac{\sigma}{G} \right]^{3.45}$$

.....(3')

In other words, creep characteristics, which are of typical for creep controlled by the viscous glide of dislocations dragging the solute atmosphere, appear in α -Fe-Mo solid solutions under a certain condition even in relatively dilute alloys.

It is also obvious in Fig. 5 that there is another region, in which n is about 5. This region, region H,

Fig. 4. Temperature dependence of steady-state creep rates at three stress levels.

Fig. 5. Steady-state creep rates normalized with the solute concentration, the solute diffusion coefficient and the shear modulus as a function of stress normalized with the shear modulus.

appears well below the condition of power-law breakdown,¹⁾ i.e., $\dot{\epsilon}/D \approx 10^{18} \text{ m}^{-2}$. The creep rates in region H depend on a simple power-law of the applied stress over a certain range of stress and can be expressed by the equation,

$$\dot{\epsilon}_s^H = 5.7 \times 10^7 \left[\frac{Gb}{kT} \right] D_{Fe} \left[\frac{\sigma}{G} \right]^{5.3}$$

.....(4)

Therefore, this region is the regime, in which a specific creep mechanism operates as in the case of region H in Al-Mg alloys.⁵⁾ It is not a region which is merely a transitional range from class I behavior to class II behavior under higher stresses¹⁷⁾ or the power-law breakdown at high creep rates.

2. Transition Condition

The breakaway of dislocation from the Cottrell atmosphere has been suggested to be responsible for the upper transition,^{3,5)} i.e., the transition between regions M and H in Al-Mg alloys. In the following the transition stresses in the Fe-Mo alloy will be

compared with the theoretical expectation.

The Cottrell atmosphere dragging model¹⁸⁾ gives the breakaway (shear) stress τ_u for a straight edge dislocation as

$$\tau_u = \frac{17}{2} \frac{WN}{\Omega b} \dots\dots\dots(5)$$

where, Ω : the atomic volume.

The factor W correlates with the elastic interaction energy:

$$W = \frac{4}{3} \frac{1+\nu}{1-\nu} G b e r^3 \dots\dots\dots(6)$$

where, ν : Poisson's ratio

e : the atom size factor

r : the radius of solvent atoms.

In the bcc Fe-Mo system W is $4.78 \times 10^{-30} \sim 4.34 \times 10^{-30} \text{ N}\cdot\text{m}^2$ at temperatures of interest and Ω is $11.8 \times 10^{-30} \text{ m}^3$, or $W/\Omega b = 1.64 \times 10^9 \sim 1.50 \times 10^9 \text{ N}\cdot\text{m}^{-2}$. The experimental value of the normalized (upper) transition stress σ_u/G is almost independent of temperature (see Table 3) and can be represented by the equation,

$$\sigma_u \doteq 1.0 \frac{W}{\Omega b} \cdot N_{\text{Mo}} \dots\dots\dots(7)$$

This experimental equation has a form similar to that of Eq. (5), except the numerical factor. This fact supports strongly that at least a part of dislocations can glide free from the atmosphere dragging in region H.

The calculated values of τ_u are an order of magnitude larger than the observed values of σ_u , that is $\sigma_u/2.7 \doteq \tau_u/22.5$. A similar quantitative difference between σ_u and τ_u was reported for the (upper) transition in creep of Al-Mg solid solutions.⁴⁾ Some possible reasons of this difference are curved dislocations in actual structures instead of long straight edge dislocations assumed in the theory, and the local fluctuation of effective stress in actual structures. Theoretical estimation of τ_u with a sophisticated model has been attempted¹⁹⁾ but the large difference in the absolute values between the experimental and the theoretical breakaway stresses is not solved yet.²⁰⁾

3. Comparison with Alloys of Other Systems

It becomes evident that a bcc Fe-Mo solid solution containing about 2 mol% Mo shows a distinct transition in steady-state deformation behavior in the power-law creep regime. In fcc Al-Mg solid solutions steady-state creep characteristics were thoroughly investigated^{4,5)} and two transitions were reported as shown in Fig. 6.²¹⁾ Recently deformation characteristics of α -Ti-Al solid solutions, the crystal system of which is hcp, have been studied in conventional tensile tests at high temperatures.²²⁾ Typical results on the relation between strain-rate and steady-state (or maximum) stress are shown in Fig. 7. In α -Ti-Al alloys, a transition similar to the present work can also be observed.

All these solid solutions which contain roughly 1 mol% of solutes show a transition in high-tempera-

Fig. 6. Steady-state creep-rates in Al-1.1mol%Mg alloy as a function of stress.²¹⁾

Fig. 7. Steady-state creep-rates normalized by the Dorn equation in α -Ti-5.6mol%Al alloy as a function of normalized stress.²²⁾

ture deformation behavior independent of their crystal system. The transition condition can well be correlated with the breakaway of the Cottrell (solute) atmosphere. The numerical constants in Eq. (7) are similar in these three kinds of alloys; about 1.0 in the bcc Fe-Mo alloy, about 1.0 in fcc Al-Mg alloys⁴⁾ and about 1.1 in hcp Ti-Al alloys.²²⁾ Therefore, the (upper) transition in high-temperature deformation behavior is a common phenomenon in solid solutions containing a moderate amount of the solute. The condition for occurrence of this transition does not depend on the crystal system of solid solutions.

The lower transition has been reported in Al-Mg alloys, though the deformation mechanism in the lower stress region (region L) has not been fully understood. In alloys of the other systems this lower transition has not been observed, but it is not clear at present that this missing is only apparent simply because of the limitation in the stress range examined, or it is inherent in these alloys.

V. Conclusions

(1) Creep behavior of a bcc Fe-Mo solid solution in the power-law creep regime can be divided at least into two regions according to the stress depen-

dence of steady-state creep-rates.

(2) Steady-state deformation behavior in the conventional tensile test is in good agreement with that in creep test.

(3) A transition in deformation behavior occurs at a stress level, σ_u/G , which is almost independent of temperature. In the Fe-2.1mol%Mo alloy, σ_u/G is about 7×10^{-4} .

(4) Creep characteristics in the lower stress region are similar to those observed in region M of Al-Mg alloys: the creep rates depend on $D_{\text{solute}} \sigma^{3.5}$. Creep in this region is controlled by viscous gliding of dislocations dragging the solute atmosphere.

(5) Creep characteristics in the higher stress region are similar to those observed in region H of Al-Mg alloys: the creep rates depend on $\sigma^{5.3}$. Creep in this high stress region seems to be controlled by recovery as in pure metals.

(6) The transition in high-temperature creep behavior, other than the power-law breakdown, is a common phenomenon in solid solutions containing a moderate amount of solutes, independent of the crystal system.

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