Stress Relaxation of Ti-N Alloys at 300 and 400°K

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Stress relaxation tests were conducted at 300 and 400°K to ascertain the deformation kinetics for Ti-N(0.035~0.615 at %N) alloys. An alternative method in addition to that of Li and Gupta was used to evaluate the dislocation velocity-stress exponent m^* , since the Li-Gupta method leads to difficulties for materials having large values of m^* such as Ti and Ti alloys. The present method consists of first evaluating m from $\log\sigma$ - $\log t$ curves, which are dependent on both grain size and strain, and secondly of double extrapolations of m to $\varepsilon \rightarrow 0$ and $d \rightarrow \infty$ to deduce m^* , i.e.

$$m^* = \lim_{1/\sqrt{d} \rightarrow 0} [\lim_{\sqrt{\varepsilon} \rightarrow 0} (m)].$$

The thermal component of the flow stress, σ^* , was then deduced from $\sigma^* = (m^*/m)\sigma_0$, where σ_0 is the stress from which the relaxation initiates. With a knowledge of σ^* and m^* from the stress relaxation tests, various thermal activation parameters such as the activation volume and the force-activation distance curve for the dislocation-obstacle interaction were evaluated and found to be in good accord with those previously obtained from the conventional analysis of the temperature dependence of the flow stress and strain rate cycling tests. It is concluded that the thermally activated overcoming of interstitial nitrogen atoms is the rate controlling mechanism in the plastic flow of Ti-N alloys at low temperatures ($T < 0.3T_m$).

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I. Introduction

The stress relaxation technique has been utilized by several investigators^{(1)~(8)} to evaluate the deformation kinetics of metals. A common method of analyzing the stress relaxation data is based on the determination of the dislocation velocity-stress exponent m^* from the relaxation curves⁽⁴⁾⁽⁷⁾⁽⁹⁾, the theoretical background being the following.

The imposed strain rate during a constant strain test can be divided into two components, elastic and plastic,

$$\dot{\varepsilon}_t = \dot{\varepsilon}_e + \dot{\varepsilon}_p. \quad (1)$$

When the crosshead is stopped, $\dot{\varepsilon}_t = 0$, and so

$$\dot{\varepsilon}_p = -\dot{\varepsilon}_e = (-1/E_c)\dot{\sigma} \quad (2)$$

where E_c is the effective modulus of the specimen-machine system and $\dot{\sigma}$ the rate of stress relaxation. The plastic strain rate $\dot{\varepsilon}_p$ is also given by the Orowan equation⁽¹⁰⁾

$$\dot{\varepsilon}_p = \alpha \rho b \bar{v} \quad (3)$$

where α is a geometric factor, ρ the density of mobile dislocations, b their Burgers vector and $\bar{v}$ their average velocity. The relation between stress and average

velocity is taken from the suggestion of Johnston and Gilman⁽¹¹⁾

$$\bar{v} = B(\sigma - \sigma_i)^{m^*} = B(\sigma^*)^{m^*} \quad (4)$$

where σ_i is the long-range internal stress and B is a constant. Equations (1) ~ (4) are combined to yield

$$\dot{\sigma} = -E_c \alpha \rho b B (\sigma^*)^{m^*} = -K' (\sigma^*)^{m^*} \quad (5)$$

where $K' = E_c \alpha \rho b B$. Integration of eq. (5) under the assumption of constant K' , σ_i and m^* yields

$$\sigma^* = \sigma - \sigma_i = K(t + a)^{-n} \quad (6)$$

where $n = 1/(m^* - 1)$, $K = [K'(m^* - 1)]^{-n}$ and a is an integration constant.

Li⁽⁷⁾ and Gupta and Li⁽⁹⁾ have analyzed the stress relaxation curves for some *bcc* metals and alloys according to eqs. (5) and (6); m^* was calculated from the slope of the linear portion of a log-log plot of the negative of the stress rate versus relaxation time, i.e.

$$m^* = S'/(1 + S') \quad (7)$$

where S' is the slope of the long-time, linear part. They have had some success with *bcc* metals and ionic crystals. Recently Law and Beshers⁽¹²⁾ have shown that the Li-Gupta method breaks down on *fcc* metals and *hcp* α -Ti; the absolute values of S' are less than unity for *fcc* and *hcp* materials to yield negative m^* . However, they have not provided any clue to an alternative method of analyzing the data for *fcc* and *hcp* materials.

Noble and Hull⁽⁴⁾ have shown an alternative method for the analysis. Instead of eq. (4), they used the dislocation velocity-stress relation

$$\bar{v} = (\sigma/\sigma_f)^m \quad (8)$$

where σ_f is a constant at constant temperature, and so eq. (5) is now written as

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$$-\dot{\sigma} = A(\sigma)^m \quad (9)$$

where A is given by $(\alpha b \rho / \sigma_0^m)$. Finally the stress-time relation ends up with the equation

$$\ln \sigma = \frac{1}{1-m} \ln \left(1 + \frac{B'}{C} t \right) + \ln \sigma_0 \quad (10)$$

where B' and C are constants and σ_0 the stress at time = 0. Accordingly m is obtained from

$$S = 1/(1-m) \quad (11)$$

where S is the slope of a log-log plot of the stress versus time. The actual value of m varied with the strain at which the stress relaxation was made in Fe-3.25 at %Si⁽⁴⁾ and Noble and Hull attributed the difference between m^* and m thus obtained to the existence of internal stress.

A variation of m with strain and grain size has been observed by the present authors employing strain rate cycling tests in zone-refined titanium⁽¹³⁾ as well as in a series of Ti-N alloys⁽¹⁴⁾. They have shown that the dislocation velocity-stress exponent m^* could be derived through elimination of the effects of work hardening and grain size on m .

The objectives of this paper are to apply the stress relaxation tests to a series of Ti-N alloys for which deformation parameters have already been evaluated using conventional thermal activation analysis⁽¹⁵⁾ and also from strain rate cycling tests⁽¹⁴⁾ and to propose an alternative method for analyzing the stress relaxation data of alpha phase Ti alloys.

II. Experimental Procedures

Five Ti-N alloys were prepared from MARZ grade zone-refined titanium and 99.8% TiN powder from the Material Research Corporation, by melting them in a vacuum of less than 10^{-6} torr with an electron beam. The zone-melted ingots were first cold-rolled to $6 \times 6 \times 150$ mm bars and then homogenized in vacuum for 48 hr at 800°C. No increase in oxygen was found, the analysis being within the range of as-received MARZ grade titanium. The nitrogen content was analyzed by the Kjeldahl technique (Table 1). Homogenized bars were swaged at room temperature to 1.2 mm diameter wires without any intermediate annealing, representing a reduction in area of approximately 96%.

Table 1 Chemical analyses of as-received MARZ Ti and Ti alloys.

MARZ Ti	O	N	C	H	Fe	O (at%) eq.
	63	6	78	4	50	0.05
N Content (at%)	A	B	C	D	E	
	0.035	0.094	0.175	0.355	0.614	

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Table 2 Heat treatments to obtain various grain sizes.

Alloy	A	B	C	D	E
Grain size (microns)	Annealing conditions, temperature (°C) and time (min)				
2	520° 48	520° 54	520° 58	520° 60	520° 63
4	692° 23	692° 30	692° 34	692° 38	692° 43
15	792° 50	792° 70	792° 80	792° 90	792° 110

Grain sizes were measured by the linear intercept method.

The swaged wires were chemically polished in a solution of HNO₃ and HF, and then annealed in vacuum at several temperatures and for certain periods to obtain the grain sizes of 2, 4 and 15 microns, on referring to the grain growth data for Ti-N alloys by the present authors⁽¹⁶⁾. Table 2 shows the heat treatments to prepare the grain size. Grain sizes were measured under an optical microscope using the conventional linear intercept method. The total length of the tensile specimens was 50 mm and the effective gauge length was about 25 mm.

Stress relaxation tests were conducted with an Instron-type Shimadzu tensile testing machine IS-500 at 300 and 400°K. Specimens were first strained in tension to a predetermined load (or plastic strain) at the strain rate of 3.3×10^{-4} sec⁻¹ and the crosshead was stopped to allow the load to relax for 5 min. These operations were repeated at the plastic strains of approximately 1, 2, 4, 8 and 16% unless otherwise the specimen fractured.

III. Experimental Results

Typical stress relaxation curves at 300°K are given in Fig. 1 for 0.035 at %N-Ti alloy with the grain sizes of 2, 4 and 15 microns. All the materials exhibited a linear stress-log time relation behavior. The change in stress due to the relaxation for 5 min, $\Delta\sigma$ increased with increasing nitrogen content and test temperature, but was independent of the grain size and plastic strain where the stress relaxation was made. This fact implies that work hardening does not affect the stress relaxation.

Let us first analyze the stress relaxation curves by means of the Li-Gupta method according to eqs. (5) and (6). The logarithm of the stress rate is plotted against the logarithm of relaxation time in Fig. 2 for 0.035 at %N-Ti alloy as an example. In the figure the coordinates are properly shifted in order to include all the experimental data for three grain sizes. It is seen from the figure that a linear relationship holds at long times to yield a limiting slope of $S' = m^*/(1-m^*)$ according to eq. (7), though deviations from a straight line occur in the early period of the stress relaxation.

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Fig. 1 Stress relaxation curves at 300°K for Ti-0.035 at%N alloys with the grain sizes of 2, 4 and 15 microns.

Fig. 2 Log-log plots of the stress rate versus relaxation time at 300°K for Ti-0.035 at%N alloy with the grain sizes of 2, 4 and 15 microns.

The experimental data show approximately the same slope regardless of the grain size and plastic strain. It should be noted that in all cases the absolute value of the slope S' is larger than unity to yield positive m^* , although Law and Beshers⁽¹²⁾ have observed negative m^* for *hcp* titanium because the absolute value of S' was less than unity.

Fig. 3 m^* deduced at 300°K from the Li-Gupta analysis (eq. 7) versus plastic strain for Ti-N alloys.

Fig. 4 m^* deduced at 400°K from the Li-Gupta analysis (eq. 7) versus plastic strain for Ti-N alloys.

m^* calculated from the slope S' is depicted in Figs. 3 and 4 as a function of the plastic strain where the stress relaxation was made for the five Ti-N alloys at 300 and 400°K, respectively. Special precaution had to be taken to evaluate carefully the values of S' for the present alloys, since they ranged from -1.0962 to -1.0377 , for the resultant values of m^* varied so rapidly with a small variation of S' . From Figs. 3 and 4 it is seen that m^* increases with decreasing temperature and with increasing nitrogen content at 300°K. m^* , however, is independent of nitrogen content at 400°K, the value of m^* being approximately 11.6 for

all alloys. This trend is in accord with the results of strain rate cycling tests on the same alloys⁽¹⁴⁾. The values of m^* deduced from the Li-Gupta method will be listed later in Table 5 for comparison.

In the Li-Gupta method, one has to evaluate the stress relaxation rate with extreme care from the relaxation curve, whose curvature becomes quite small at long times so that the resultant error in the stress rate becomes large. Again, especially for the materials whose m^* is large, the slope in a log-log plot of the stress rate versus time becomes too small to evaluate m^* exactly.

Let us now consider the alternative method, eq. (10), for analyzing the stress relaxation curves.

Depicted in Fig. 5 are log-log plots of the relaxed load versus time for Ti-0.035 at %N alloy of 2 microns grain size. The experimental data points are well represented by straight lines whose slope varies with the plastic strain where the relaxation is made. m calculated from the slope S according to eq. (11) increases with increasing plastic strain. The values of m as a function of strain are listed in Tables 3 and 4 for the stress relaxations at 300 and 400°K respectively.

Analogous to the strain dependence of m obtained from the strain rate cycling tests⁽¹³⁾⁽¹⁴⁾, m is now plotted against the square root of plastic strain, $\epsilon^{1/2}$ in Fig. 6 for Ti-0.035 at %N alloy as an example. Straight lines can be drawn through the experimental

Table 3 Values of m and σ_0/m as a function of strain and of m^* and σ^* obtained from the stress relaxation tests at 300°K for Ti-N alloys.

Alloy	Temp. (°K)	G.S. (μ)							m_0	m^*	σ_0/m	σ^*
A	300	2	$\epsilon(\%)$ m σ_0/m	0.48 45.3 0.62	1.55 51.5 0.61	4.00 61.0 0.62	7.98 69.5 0.61	15.97 78.9 0.63	38.0	15.4	0.62	9.6
		4	$\epsilon(\%)$ m σ_0/m	0.66 38.0 0.61	2.20 42.5 0.63	4.00 47.9 0.63	7.50 54.0 0.63	15.70 65.0 0.62	31.0			
		15	$\epsilon(\%)$ m σ_0/m	0.82 30.1 0.62	1.66 34.3 0.62	3.60 38.0 0.61	7.50 47.5 0.61	15.90 54.0 0.64	23.5			
B	300	2	$\epsilon(\%)$ m σ_0/m	0.40 41.3 0.85	1.40 45.5 0.84	3.20 49.8 0.82	6.10 53.5 0.84	15.10 60.7 0.86	39.0	19.2	0.84	15.8
		4	$\epsilon(\%)$ m σ_0/m	0.95 37.4 0.84	2.05 40.3 0.82	5.35 44.8 0.86	7.95 48.3 0.85	16.29 53.9 0.86	33.0			
		15	$\epsilon(\%)$ m σ_0/m	0.60 29.7 0.83	1.40 33.2 0.82	3.26 36.3 0.85	7.17 41.0 0.83	15.30 47.5 0.83	26.5			
C	300	2	$\epsilon(\%)$ m σ_0/m	0.26 43.2 0.96	1.44 46.7 0.93	3.37 52.5 0.93	7.32 56.7 0.93	15.50 63.1 0.95	39.5	22.1	0.95	21.0
		4	$\epsilon(\%)$ m σ_0/m	0.48 39.0 0.94	1.53 40.4 0.95	3.12 45.5 0.95	7.26 49.8 0.96	15.30 57.4 0.94	34.2			
		15	$\epsilon(\%)$ m σ_0/m	0.59 31.4 0.94	1.41 36.0 0.94	3.28 39.0 0.96	7.20 44.5 0.96	15.10 50.5 0.95	28.2			
D	300	2	$\epsilon(\%)$ m σ_0/m	0.30 40.3 1.22	1.44 45.8 1.20	3.30 47.3 1.19	7.10 52.2 1.19	14.90 58.4 1.19	40.5	24.8	1.20	29.8
		4	$\epsilon(\%)$ m σ_0/m	0.56 38.8 1.19	1.48 41.4 1.20	3.36 44.5 1.20	7.34 48.8 1.18	15.50 54.0 1.20	35.8			
		15	$\epsilon(\%)$ m σ_0/m	0.65 34.0 1.21	1.51 34.9 1.21	3.38 38.2 1.19	7.30 41.5 1.22	15.47 48.0 1.22	30.5			
E	300	2	$\epsilon(\%)$ m σ_0/m	0.32 42.5 1.62	1.34 43.2 1.60	3.31 46.5 1.59	6.93 48.5 1.60	15.13 52.0 1.62	41.0	27.0	1.60	43.2
		4	$\epsilon(\%)$ m σ_0/m	0.42 39.5 1.63	1.45 40.2 1.59	3.40 42.0 1.60	7.50 45.5 1.62	15.80 48.3 1.58	37.0			
		15	$\epsilon(\%)$ m σ_0/m	0.08 32.2 1.65	1.37 35.6 1.58	3.36 36.9 1.62	7.85 40.0 1.62	16.00 43.1 1.58	32.5			

data points for each grain size at each temperature. To exclude the effect of strain on m , straight lines are now extrapolated to zero strain to obtain m_0 . The intercept value of m_0 shows a grain size dependence, which is depicted in Fig. 7, where m_0 is plotted against the reciprocal of the square root of grain size. Therefore, the grain size dependence is excluded by extrapolating straight lines to infinite grain size ($d^{-1/2} = 0$) and the extrapolated m_0 is now designated m^* . Namely, the dislocation velocity-stress exponent m^* is obtained from the stress relaxation tests by the double extrapolations

$$m^* = \lim_{d^{-1/2} \rightarrow 0} [\lim_{\epsilon^{1/2} \rightarrow 0} (m)] \quad (12)$$

The derived values of m^* from the stress relaxation tests using both the Li-Gupta method $(m^*)_L$ and the extrapolation method $(m^*)_{ext.}$ and those from the strain rate cycling tests are given in Table 5. There occurs good agreement in the values for the various techniques.

Stress relaxation curves at 400°K, as an example, are shown in Fig. 8 on a semi-log plot. Straight lines can be drawn through the data points for the three grain sizes in each alloy. For the present alloys the total load relaxed for 5 min, $\Delta\sigma$ was independent of strain and grain size. It has been shown by Feltham⁽²⁾ and Sargent⁽⁵⁾ that the slope S'' of such plots is related to the activation volume $V^* (= kT \partial \ln \dot{\nu} / \partial \sigma^* |_T)$ for

Table 4 Values of m and σ_0/m as a function of strain and of m^* and σ^* obtained from the stress relaxation tests at 400°K for Ti-N alloys.

Alloy	Temp. (°K)	G.S. (μ)							m_0	m^*	σ_0/m	σ^*
A	400	2	$\epsilon(\%)$ m σ_0/m	0.85 43.6 0.56	1.79 47.0 0.55	3.65 51.5 0.55	7.52 59.0 0.58	15.50 68.0 0.57	34.5	11.2	0.56	6.2
		4	$\epsilon(\%)$ m σ_0/m	0.95 32.0 0.55	1.80 36.2 0.54	3.83 40.8 0.57	7.68 42.8 0.58	15.75 49.4 0.59	27.4			
		15	$\epsilon(\%)$ m σ_0/m	1.01 24.5 0.57	1.43 28.6 0.55	2.08 29.0 0.58	7.43 31.1 0.57	12.20 34.4 0.59	19.5			
B	400	2	$\epsilon(\%)$ m σ_0/m	0.43 30.60 0.96	1.35 34.0 0.95	3.21 37.4 0.96	7.10 42.6 0.94	15.13 47.3 0.95	26.7	11.2	0.95	10.6
		4	$\epsilon(\%)$ m σ_0/m	0.78 24.0 0.96	1.66 26.0 0.97	4.49 29.8 0.95	7.45 32.7 0.94	15.73 36.1 0.96	21.5			
		15	$\epsilon(\%)$ m σ_0/m	0.76 19.3 0.94	1.86 21.2 0.95	3.87 23.7 0.93	7.93 29.7 0.94	14.07 31.8 0.93	17.0			
C	400	2	$\epsilon(\%)$ m σ_0/m	0.40 22.5 1.27	1.46 25.6 1.27	3.34 28.5 1.28	7.23 32.9 1.26	15.24 37.8 1.26	20.0	10.8	1.26	13.6
		4	$\epsilon(\%)$ m σ_0/m	0.80 19.2 1.26	1.80 22.7 1.23	3.50 25.4 1.25	7.50 27.0 1.28	15.70 31.4 1.25	17.0			
		15	$\epsilon(\%)$ m σ_0/m	0.85 16.6 1.6	2.68 19.4 1.26	3.44 20.0 1.27	7.41 22.3 1.26	15.57 24.7 1.26	14.0			
D	400	2	$\epsilon(\%)$ m σ_0/m	0.67 20.80 1.77	1.89 22.0 1.76	3.76 24.6 1.76	8.67 27.3 1.78	15.71 30.1 1.75	17.5	10.8	1.75	18.8
		4	$\epsilon(\%)$ m σ_0/m	1.10 18.4 1.74	1.80 20.8 1.75	3.66 22.4 1.74	7.60 25.4 1.74	15.90 28.1 1.76	15.5			
		15	$\epsilon(\%)$ m σ_0/m	0.78 17.2 1.74	1.69 16.8 1.75	3.62 20.2 1.74	8.06 21.8 1.74	16.27 24.5 1.75	13.0			
E	400	2	$\epsilon(\%)$ m σ_0/m	0.63 16.0 2.48	3.22 19.5 2.50		7.93 23.5 2.55	15.99 25.3 2.53	13.5	11.0	2.50	27.4
		4	$\epsilon(\%)$ m σ_0/m	1.10 15.5 2.57	2.20 16.8 2.48	3.30 17.8 2.51	8.50 21.2 2.52		12.5			
		15	$\epsilon(\%)$ m σ_0/m	0.98 14.0 2.48	1.90 15.2 2.54	3.90 17.3 2.48	8.00 18.3 2.51		11.5

Fig. 5 Log-log plots of the relaxed load versus time for Ti-0.035 at%N of 2 microns grain size.

Fig. 6 Strain dependence of m at 300 and 400°K for Ti-0.035 at%N alloy of 2, 4 and 15 microns grain sizes.

plastic flow at temperature T by

$$S'' = d(\Delta\sigma)/d[\ln(t + c)] = kT/V^* \quad (13)$$

where k is Boltzmann's constant and c a constant. The derived values of the activation volume in terms of the Burgers vector for prism slip in titanium are given in Table 6, where the values of the activation volume obtained from the strain rate cycling tests for the same materials⁽¹⁴⁾ are also given for comparison. Agreement is found to be reasonable between the results of the stress relaxation tests and those of the strain rate cycling tests.

Fig. 7 Grain size dependence of m_0 at 300 and 400°K for Ti-N alloys.

Table 5 Values of m^* obtained from the stress relaxation tests and the strain rate cycling test at 300 and 400°K for Ti-N alloys.

Alloy	Temp. (°K)	Stress relaxation test		Strain rate cycling test
		$(m^*)_0$	$(m^*)_{ext.}$	$(m^*)_{ext.}$
A	300	15.3	15.5	36
B		19.4	19.2	19
C		22.1	22.1	22
D		24.5	24.8	25
E		27.5	27.0	28
A	400	11.7	11.2	10
B		11.7	11.2	—
C		11.8	10.8	12
D		11.4	10.8	—
E		11.4	11.0	12

Fig. 8 Semi-log plots of stress relaxation curve at 400°K for Ti-N alloys.

IV. Discussion

It has been shown in the previous section that the value of m^* obtained by the Li-Gupta analysis is in good accordance with that obtained by the double-

Table 6 Values of the activation volume obtained from the stress relaxation tests and the strain rate cycling tests at 300 and 400°K for Ti-N alloys.

Alloy	Temp. (°K)	Stress relaxation test		Strain rate cycling test
		V^*/b^3 ($=kT/S''$)	V^*/b^3 ($=kTm^*/\sigma^*$)	V^*/b^3 ($=kT(\partial \ln \bar{v}/\partial \ln \sigma^*)$)
A	300	125.7†	134.9††	133.3
B		96.5	101.4	98.5
C		83.3	88.0	85.0
D		66.5	69.6	70.5
E		51.0	52.4	60.0
A	400	191.0	198.2	175.8
B		118.5	114.2	—
C		89.0	85.8	89.3
D		66.8	61.8	—
E		48.3	42.6	47.8

†Equation (13) is used. ††(m^*)_{ext.} and σ^* of eq. (19) are used.

extrapolation method and also with that obtained from the strain rate cycling test. With a knowledge of m^* , the effective stress (thermal component of the flow stress) σ^* can be evaluated in the following way.

If a single thermally activation mechanism is rate controlling, the strain rate $\dot{\epsilon}$ can be written by (assuming that the entropy of activation is zero)

$$\dot{\epsilon} = \alpha \rho b \bar{v} = \alpha (\rho/l^*) A b v^* \exp(-H(\sigma^*)/kT) \quad (14)$$

where l^* is the dislocation segment length involved in the thermal activated process, A the area of the slip plane swept out per successful fluctuation, v^* the frequency of vibration of the segment l^* and H the activation enthalpy which is a decreasing function of σ^* . Therefore the activation volume V^* can be deduced from eq. (14), yielding

$$V^* = -dH/d\sigma^* = kT(\partial \ln \bar{v}/\partial \ln \sigma^*)_T \\ = kT(\partial \ln \bar{v}/\partial \ln \sigma^*)_T/\sigma^*. \quad (15)$$

On the other hand, m^* is defined from eq. (4) to be

$$m^* = (\partial \ln \bar{v}/\partial \ln \sigma^*). \quad (16)$$

From eqs. (15) and (16), σ^* is interrelated with m^* and V^* as

$$\sigma^* = kTm^*/V^*. \quad (17)$$

The calculated values of σ^* using eq. (17) are given in the first two columns of Table 7, one for (m^*)_d obtained from the Li-Gupta analysis and another for (m^*)_{ext.} obtained from the double extrapolation method (Okazaki-Conrad analysis).

An alternative way to correlate m^* with the effective stress σ^* is as follows. By assuming that both the athermal stress σ_μ and the mobile dislocation density ρ remain unchanged during the stress relaxation, m defined as $(\partial \ln \bar{v}/\partial \ln \sigma)$ from eq. (8) is further transformed to be

$$m = (\partial \ln \bar{v}/\partial \ln \sigma) = \sigma(\partial \ln \bar{v}/\partial \ln \sigma^*) \\ = (\sigma/\sigma^*)(\partial \ln \bar{v}/\partial \ln \sigma^*) = (\sigma/\sigma^*)m^*. \quad (18)$$

Therefore,

$$\sigma^* = (\sigma/m)m^* \quad (19)$$

Figure 9 shows the plots of (σ_0/m) versus plastic strain ϵ for the present alloys stress-relaxed at 300°K. It is seen from this figure that the value of (σ_0/m) remains constant independent of both strain and grain size, but depends strongly on the nitrogen concentration. This is also shown in Tables 3 and 4. The constancy of

Fig. 9 (σ_0/m) versus plastic strain at 300°K for Ti-N alloys.

Table 7 Thermal component of the flow stress in kg/mm² evaluated from several methods in Ti-N alloys.

Alloy	Temp. (°K)	Stress relaxation test		Strain rate cycling test	Conventional test
				(Ref. 14)	(Ref. 15)
		$\sigma^*(=kTm^*/V^*)$	$\sigma^*(=\sigma m^*/m)$	$\sigma^*(kTm^*/V^*)$	σ^*
A	300	10.2†	10.4††	10.0	8.9
B		16.8	16.6	15.9	14.8
C		22.8	22.8	21.4	19.0
D		30.8	31.2	29.2	27.6
E		43.6	44.4	38.2	39.0
A	400	6.6	6.2	6.2	4.4
B		10.8	10.6	—	—
C		14.8	13.6	14.8	12.7
D		18.4	18.8	—	—
E		25.2	24.2	27.6	25.5

†(m^*)_d is used. ††(m^*)_{ext.} is used. †††(m^*)_{ext.} is used.

(σ_0/m) is easily understood from eq. (19) since (σ_0/m) is equal to (σ^*/m^*), which should be independent of both strain and grain size but dependent on the impurity content if the rate-controlling process is overcoming interstitial solute obstacles. The calculated values of σ^* using eq. (19) are given in the third column of Table 6. For comparison, the fourth and fifth columns give σ^* obtained from the strain rate cycling tests⁽¹⁴⁾ and also from the conventional analysis of the temperature dependence of the flow stress⁽¹⁵⁾ for the same alloys as the present. Agreement of σ^* among the results from the stress relaxation and strain rate cycling tests is very good. Moreover, they are only slightly larger (1 ~ 2 kg/mm²) larger than those obtained from the conventional analysis.

The dependence of σ^* on the nitrogen concentration is depicted in Fig. 10 in which an approximately linear relation is observed between σ^* and the square root of the atomic fraction of interstitial nitrogen atoms. The slopes of the best fitting lines through the experimental points at 300 and 400°K are 0.148μ and 0.107μ respectively where μ is the shear modulus C_{66} at each temperature, giving

$$\sigma^* = 0.148\mu\sqrt{C_N} \text{ at } 300^\circ\text{K}$$

and

$$\sigma^* = 0.107\mu\sqrt{C_N} \text{ at } 400^\circ\text{K} \quad (20)$$

A similar relation as eq. (20) was reported by Conrad⁽¹⁷⁾⁽¹⁸⁾ for materials of various interstitial contents.

Okazaki et al.⁽¹⁵⁾ have obtained for Ti-N alloys from the conventional analysis of the temperature dependence of the flow stress

$$\sigma_0^* = 0.34\mu_0\sqrt{C_N} \quad (21)$$

where σ_0^* and μ_0 are thermal component of the flow stress and shear modulus C_{66} extrapolated to 0°K. An identical relation with eq. (21) may be obtained from the present results if the thermal component of the flow stress at 0°K is deduced by extrapolating the $\sigma^*-T^{1/2}$ relation to 0°K, which is shown in Fig. 11. Although the data points were obtained only at 300

Fig. 10 Concentration dependence of the thermal component of the flow stress σ^* at 300 and 400°K for Ti-N alloys.

Fig. 11 $\sigma^*-T^{1/2}$ plots for Ti-N alloys.

Fig. 12 Concentration dependence of the activation volume at 300 and 400°K for Ti-N alloys.

and 400°K, they fall on the same straight lines of the σ^* versus $T^{1/2}$ plots which were obtained for these Ti-N alloys⁽¹⁵⁾. The strong effect of the nitrogen content on σ^* given by eq. (21) is a case of "rapid hardening" according to the nomenclature of Fleischer⁽¹⁹⁾.

Depicted in Fig. 12 are plots of $1/V^*$ versus $C^{1/2}$ for the present alloys at 300 and 400°K, where V^* is derived from the relation $V^* = kTm^*/\sigma^*$ from the Li-Gupta and Okazaki-Conrad analysis. This dependence of $1/V^*$ on $C^{1/2}$ is explained as follows. Considering the obstacles to be individual interstitial solute atoms distributed at random, their spacing λ on the slip plane is given by

$$\lambda \simeq b/C. \quad (22)$$

Taking into account the dislocation line tension, an average spacing l^* between the obstacles along the dislocation is^{(20)~(22)}

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(17) H. Conrad: *Acta Met.*, **14** (1966), 1634.

(18) H. Conrad: *Can. J. Phys.*, **45** (1967), 581.

$$l^* = \left(\frac{2\mu b}{\tau^*}\right)^{1/3} = b\left(\frac{2\mu}{\tau^* C}\right)^{1/3}. \quad (23)$$

The force acting on each contact point is

$$F = \tau^* b l^* = b^2 (\tau^*)^{2/3} (2\mu/C)^{1/3}. \quad (24)$$

Finally, the activation volume is given by

$$V^* = d^* (dF/d\tau^*) = (2/3)(2\mu)/(\tau^* C)^{1/3} b^2 d^* \quad (25)$$

where d^* is the activation distance and V^* is proportional to $C^{-1/2}$ (by taking $\tau^* = a\mu\sqrt{C}$), in accord with experimental observations.

The force-activation distance curve derived from the present data (by taking the average values of σ^* and V^* from all stress relaxation methods), using eqs. (23) to (25), is given in Fig. 13. The present data are not sufficient to cover the entire curve, however, they are still within the experimental errors to support the

Fig. 13 Force-activation distance curve for Ti-N alloys.

previous results from the different type of experiment. To be noted is that a rapid rise in the force occurs at $d^* \simeq 1.5b$, which implies the interaction between dislocations and interstitial nitrogen atoms is a short-range interference by nitrogen atoms with atomic motions at the core of moving dislocations, as suggested by Tyson⁽²³⁾.

V. Summary

An alternative method in addition to that of Li and Gupta was used to analyze the stress relaxation curves of Ti-N alloys at 300 and 400°K. The dislocation velocity-stress exponent m^* was evaluated by eliminating the effects of grain size and strain on the exponent m , which had been deduced from the slope of $\log\sigma$ - $\log t$ plots. The resultant m^* increased with increasing nitrogen content and decreasing temperature. The thermal component σ^* was then evaluated from the equation, $\sigma^* = (m^*/m)\sigma_0$, where σ_0 is the stress from which the relaxation initiates. The values of σ^* thus evaluated vary linearly with the square root of nitrogen content, implying that nitrogen atoms are the rate-controlling obstacles to dislocation motion. Supporting this conclusion, the activation volume V^* obtained from the stress relaxation curves varies as the inverse square root of the nitrogen content.

The various parameters pertaining to the deformation kinetics obtained from the present stress relaxation tests compare well with those previously obtained from strain rate cycling tests and from the conventional analysis of the temperature dependence of the flow stress of Ti-N alloys.

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