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EFFECT OF SUBSTRUCTURE ON FRACTURE OF TUNGSTEN AND MOLYBDENUM

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Since 1959, a comprehensive investigation[1,2,3] of substructure and mechanical properties of refractory metals sponsored by ASD has been carried out by ManLabs, Inc. in collaboration with M.I.T. and Rutgers in this country and with the universities of Liverpool and Cambridge in England. Some of the results obtained by Rutgers, Liverpool and M.I.T. have been presented during this Symposium. I would like to discuss some of the results obtained by ManLabs, which has the objective of elucidating the role of substructure with respect to the ductile-brittle transition in tungsten and molybdenum.

One of the main questions has been to decide whether primary grain size or subgrain size plays the more important role in fracture. Based on the Griffith-Orowan relation, the magnitude of the critical crack length was utilized as a criterion for answering this question. Calculations of critical crack length (2c) were made on the assumption that the effective surface energy for crack propagation (γ') as used in the Griffith-Orowan relation is approximately the same as in the Cottrell-Petch relation. It was also assumed that the fracture stress based on the Griffith-Orowan relation is equal to (or slightly less than) the fracture stress based on the Cottrell-Petch relation. This second assumption is at least partly justified on the basis that no evidence of the occurrence of microcracks prior to fracture of the materials studied has been found.

Under these conditions, it is believed that a crack forms when the Cottrell-Petch relation is satisfied and almost simultaneously attains the critical size required for propagation in accordance with the Griffith-Orowan relation. Propagation of this crack should involve the same γ' value as for the Cottrell-Petch relation at least until the first barrier is encountered. Even though the crack propagation energy (γ_1') associated with crossing such a barrier is presumably greater than for moving between barriers, the crack should cross the barrier provided that

$$\frac{\gamma_1'}{\gamma'} \leq \frac{c}{d} \tag{1}$$

where 2d is the distance between barriers and 2c is the critical crack length. Equation (1) presumably holds since no microcracks were observed prior to fracture of the materials studied.

Assuming that no necking occurs prior to fracture, the fracture stress (σ_F) is given in terms of the shear modulus (G) and the yield stress parameter (k_y) in the following relations:

Cottrell-Petch:
$$\sigma_F = \frac{2G\gamma'}{k_y} d^{-1/2} \tag{2}$$

Griffith-Orowan:
$$\sigma_F = (1.7\, G\gamma')^{1/2}\, c^{-1/2} \tag{3}$$

Rosenfield[4] has reported that the yield parameter k_y based on primary grain size is given by the following approximate relation:

$$k_y = 5 \times 10^{-5} G \tag{4}$$

Combining equations (2), (3) and (4) gives

$$\frac{c}{d} = 1.0 \times 10^{-9} \frac{G}{\gamma'} \tag{5}$$

Assuming that the ratio of subgrain size (d_s) to primary grain size (d) is approximately constant, the yield stress parameter based on subgrain size (k_{ys}) is related to k_y as follows:

$$\frac{k_{ys}}{k_y} = \left(\frac{d_s}{d}\right)^{1/2} \tag{6}$$

The corresponding equation to (5) for the ratio of critical crack length ($2c_s$) to subgrain size ($2d_s$), which involves the effective surface energy (γ_s') based on subboundaries acting as dislocation obstacles, is as follows:

$$\frac{c_s}{d_s} = 10^{-9}\left(\frac{d_s}{d}\right)\left(\frac{G}{\gamma_s'}\right) \tag{7}$$

Knowing the measured values of σ_F, d and d_s, the above equations enable calculations of γ', γ_s', c/d and c_s/d_s to be made. Such calculations are valid provided that a σ_F value is used that corresponds to a test temperature below the tensile ductility transition (T_d) at which essentially no necking occurs prior to fracture. For this situation, plastic constraint does not have to be taken into account, i.e. Cottrell's β-value = 1.

For the case of severely drawn tungsten wire (about 75 ppm total interstitial content) recovery annealed at five temperatures between 20°C and 1400°C, the increases in fiber width (d) and subgrain size (d_s) with annealing temperature were determined by microexamination using both light and electron microscopy. It was found that $d_s/d \approx 0.1$. From the measured values of σ_F corresponding to temperatures just below T_d, where fracture occurred with relatively little reduction in area, the following calculations were made:

Calculated on Basis of Primary Grain Size*	Calculated on Basis of Subgrain Size
$\gamma' = 7000$ ergs/cm^2	$\gamma_s' = 200$ ergs/cm^2
$c/d = 0.2$	$c_s/d_s = 0.01$

*Corrected for the effect of preferred orientation, i.e. slip occurs in a direction that is about 35° with respect to the fiber direction. Therefore, the effective grain size is about 1.8 times the fiber width.

The calculation based on primary grain size indicates that γ' is about 7000 ergs/cm^2, which is a more reasonable value than the value of 200 ergs based on subgrain size because surface energy measurements give values of 2000-3000 ergs. Assuming that the calculations based on primary grain size are more valid than based on subgrain size, it appears that the critical crack length is approximately equal to the subgrain size ($c = 0.2\ d$ and $d_s \cong 0.1d$).

Similar calculations were carried out for molybdenum strip (about 70 ppm total interstitial content) given about 5, 45 and 90% reduction by rolling starting with the recrystallized condition. In this case a correction was made for the decrease in primary grain size, due to the occurrence of appreciable reduction in area (about 15%) prior to fracture. In addition, the flow stress parameter (k_f) was substituted for k_y, and k_f was taken as approximately equal to $0.3\ k_y$[2].

Calculated on Basis of Primary Grain Size*	Calculated on Basis of Subgrain Size**
$\gamma' = 3000$ ergs/cm^2	$\gamma'_s = 100$ ergs/cm^2
$c/d = 0.04$	$c_s/d_s = 0.04$

*Corrected for preferred orientation and reduction in area: The correction factor = $1.8\ d\ (\frac{1-\Delta A}{2A})$ where $\frac{\Delta A}{A}$ is the fractional reduction in area.

**Corrected for reduction in area only. *By measurement, d_s was found $\cong 0.04d$.

The calculated value of γ' based on primary grain size agrees with accepted surface energy values (2000 - 3000 ergs/cm^2) and is at the low end of the range (3000 to 12,000 ergs/cm^2) previously reported by other investigators[5, 6, 7]; but the subgrain value of 100 ergs/cm^2 appears too low. Again it is found that the critical crack length based on primary grain size is approximately equal to the subgrain size ($c = 0.04\ d$ and $d_s \cong 0.04\ d$).

To summarize the results for the tungsten and molybdenum materials studied, the primary grain boundaries appear to constitute the main barriers to crack propagation since the calculated values of effective surface energy obtained on this basis are more reasonable than on the basis of subboundary obstacles. On the other hand, the fact that the calculated critical crack size was found to be approximately equal to the subgrain size suggests that subboundaries may also influence fracture behavior.

For the moderately worked molybdenum strip (about 45% reduction in area), the tensile ductility transition temperature (T_d) does not appear to change appreciably on annealing in the recovery range. However, annealing at 1000°C results in a significant increase in T_d of about 30°C. This correlates with the onset of recrystallization as manifest by the formationof substructure-free regions. These appear to be considerably larger than the polygonized subgrains representing the unrecrystallized matrix, but definitely smaller than the primary grain size. Since no appreciable change in either the polygonized subgrain size or the primary grain size was found to occur as a result of the 1000°C anneal, the increase in transition temperature is ascribed to the formation of the recrystallized regions. Within these substructure-free regions, larger cracks can presumably form as compared to the polygonized subgrains. Therefore, a decrease in fracture stress and a corresponding increase in T_d would be expected.

References

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