Constant structure creep tests and the influence of subgrain size on creep

It is commonly observed in high temperature isostress creep tests of polycrystalline solids that the steady state creep rate ($\dot{\epsilon}_S$)-creep stress (σ) relationship may be described by $\dot{\epsilon}_S \mid_T = K\sigma^n$ where n, the stress exponent, is approximately 4 to 5. Such creep tests, however, are not constant structure tests since the subgrain size developed during steady state creep is observed to be inversely proportional to the applied stress. Thus, the subgrain size is not constant when creep rates are compared at varying stresses. In a previous analysis of high temperature creep in bcc tungsten [1], two of the authors proposed an equation which incorporated the subgrain size into the creep rate equation. The equation proposed was

$$\dot{\epsilon}_S = SD\lambda^2(\sigma/E)^7 \qquad (1)$$

where S is a constant, D the lattice diffusion coefficient, λ the subgrain size or grain size, and E is the elastic modulus. Equation 1 offers several interpretations of the creep process which are potentially rewarding.

The essential characteristic of the equation is that when subgrains form during creep and the relationship $\lambda \propto E/\sigma$ is observed, i.e. a normal subgrain size to stress relationship [2], then $\dot{\epsilon}_S \propto \sigma^5$ emerges from Equation 1. If the subgrain or grain size is stable, that is, invariant with stress, then a relation $\dot{\epsilon}_S \propto \sigma^7$ emerges, as was observed in tungsten. The ability of the equation to predict both of the observed stress dependencies is due to the λ^2 $(\sigma/E)^7$ term, which is unique in two regards. First, the subgrain size, λ, was explicitly included in the creep rate equation, apparently for the first time, and secondly, while pure metal creep studies [2] usually produce stress exponents of 4 to 5, an unusually high fundamental stress exponent of 7 was postulated. The introduction of these terms into the creep rate equation then marked a significant change in the approach to the formulation of creep models and to an understanding of the mechanisms of high temperature creep.

We propose to give evidence for the validity of Equation 1 from creep data available for pure polycrystalline fcc aluminium. Our relationship predicts that a high stress dependence of about 7 should result from "constant structure" creep tests where $\lambda \neq f(\sigma)$. Such a test could be a stress drop test [3] or a stress relaxation test [4]. In analysing the data from a stress drop test, one takes the creep rate just prior to the stress reduction, and the rate after the stress reduction before significant structural changes can occur.

In Fig. 1, data on aluminium [3, 5-8] are summarized in the form of $\dot{\epsilon}(\sigma_R) \mid_{\lambda,T}$ where σ_R is the stress *after* stress reduction or the original stress before stress reduction. In most of these tests, the specimens are first pre-strained to a steady state condition, and then the stress is reduced. From Fig. 1, we find that $\dot{\epsilon} \mid_{\lambda,T} \propto \sigma^7$ when the subgrain size is held constant during creep.* The exponent of 7 is to be compared with the 4.5 observed for aluminium in normal isostress creep testing [9]. These results are in general agreement with our proposal. In Fig. 2 the creep rate is normalized to a common temperature of 257°C by using the known activation energy for high temperature creep of aluminium, i.e. the self diffusion activation energy [10], and the stress is normalized for modulus variation with temperature, i.e. σ_T (E_{257}/E_T). We are next able to determine the creep rate-subgrain size relationship by using the λ versus σ data available for aluminium from electron microscopy [12, 13] and etch pit studies [8]; the subgrain size for each pre-straining condition was thus readily determined. The result is shown in Fig. 3 for the creep stress value of 600 psi (4.14 MPa). The data appear to be fit best by a straight line with a slope of approximately 3 as shown†, which suggests that $\dot{\epsilon} \mid_{\sigma,T} \propto \lambda^3$. Thus, for the aluminium constant structure tests considered, it has been found that $\dot{\epsilon} \mid_T \propto \lambda^3 \sigma^7$, which agrees quite well with $\lambda^2 \sigma^7$ of Equation 1, from bcc tungsten studies.

---

*The results of Mitra and McLean [7] do not agree with the interpretation presented, having a much higher dependence of the creep rate upon stress ($n \sim 11$). Their data at 250°C are given as the dashed line in Fig. 1. Mitra and McLean performed sequential stress reductions upon single specimens, creep rates being measured after several per cent strain had occurred subsequent to a stress reduction. By such a method, one would expect that their results would be comparable to steady state rates and structures. Thus the stress sensitivity obtained should be like that of steady creep, i.e. $\dot{\epsilon} \propto \sigma^{4,5}$ rather than the $\dot{\epsilon} \propto \sigma^{11}$ which was obtained by Mitra and McLean. It would appear that the Mitra-McLean results must be viewed as anomalous in light of the current results.

†We also include in Fig. 3 the steady state creep rate predicted at 600 psi (4.14 MPa) from the extensive creep data obtained by Servi and Grant [9].

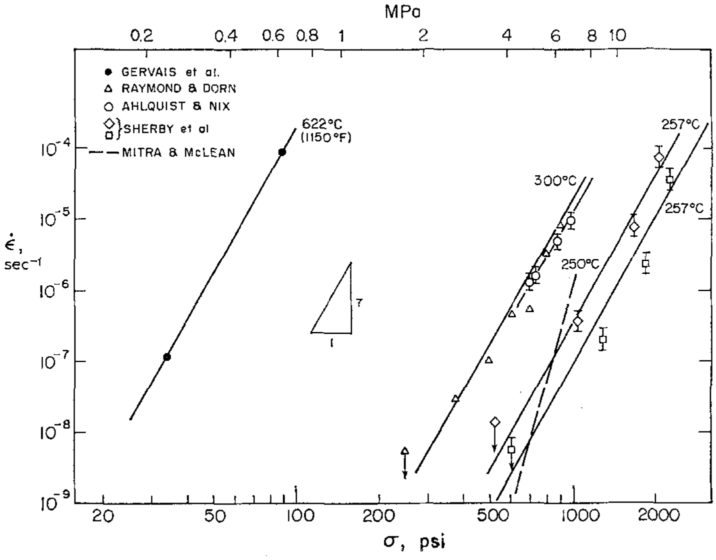

Figure 1 Stress dependence of the creep rate in high purity aluminium (99.987+) at constant subgrain size as assessed by stress-drop tests. A relation $\dot{\epsilon}\ |_{\lambda,T} \propto \sigma^7$ is obtained.

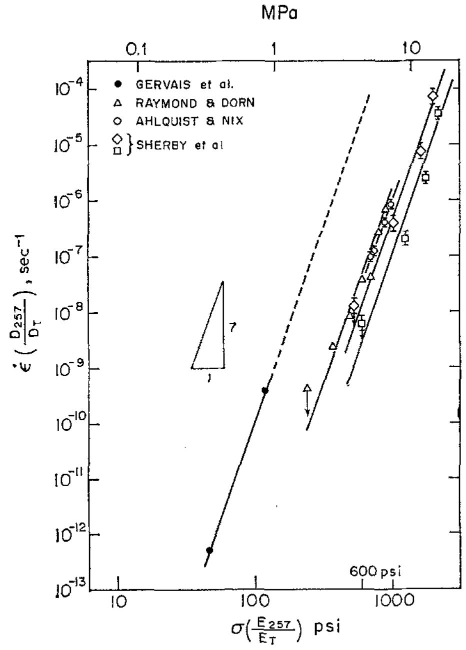

Figure 2 Creep rates in Fig. 1 corrected to a common temperature of 257°C using $\dot{\epsilon}_T\ (D_{257}/D_T)$ and the parameter $\sigma_T\ (E_{257}/E_T)$. Diffusivity data from [10] and modulus data from [11].

The above results suggest that Equation 1 fairly accurately predicts both the stress dependence of the creep rate at constant structure, and

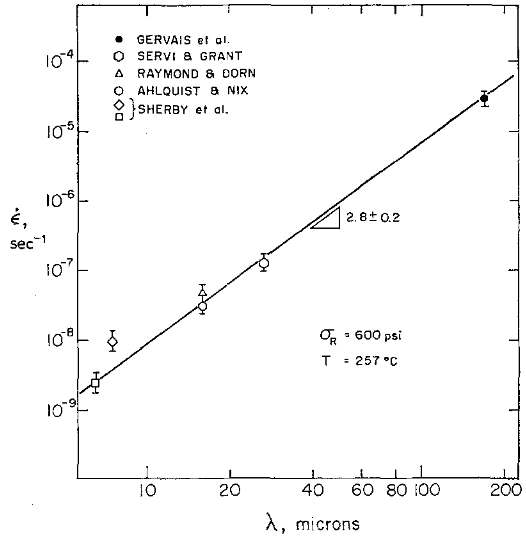

Figure 3 The creep rate dependence upon subgrain size in high purity aluminium as assessed by stress drop tests (from Fig. 1). A relation $\dot{\epsilon}\ |_{\sigma,T} \propto \lambda^3$ is obtained.

the influence of the subgrain size on the creep rate. We note that virtually no contemporary theories of creep consider the subgrain size as an important variable [2].

As noted by Weertman [14], most high temperature pure metal creep models, using the most reasonable assumptions, obtain $\dot{\epsilon}_S \propto \sigma^3$ whereas $\dot{\epsilon}_S \propto \sigma^{4.5}$ is obtained in isostress steady state creep tests on pure aluminium. The proposal of an even higher constant subgrain size stress sensitivity, $\dot{\epsilon}_s \propto \sigma^7$, places a burden upon theories of creep, requiring attention to the question of stress concentrations as a means of obtaining high stress exponents.

The role of the λ term (λ^2 or λ^3) may be related to the area swept out from generation to annihilation by a dislocation in gliding across a subgrain or some part of a subgrain. An alternative interpretation may be to treat the λ term as a subgrain surface area, controlling dislocation generation and annihilation processes. The former interpretation is partially embodied in an existing creep model [14], but the latter has not yet been considered. We note that Weertman has predicted a λ^3 relation by using subgrain boundaries as dislocation pile-up barriers.

From a practical viewpoint, our relation quantitatively predicts the strengthening expected by subgrain size refinement; a factor of 10 reduction in subgrain size results in the reduction of the creep rate by 1000. Retention of fine subgrains for high creep resistance at elevated temperature would be possible by pinning boundaries with fine precipitates or dispersed particles.

Acknowledgement

The authors wish to acknowledge financial support by the Army Materials and Mechanics Research Center, Watertown, Massachusetts of a programme on the influence of subgrain boundaries on mechanical behaviour of materials.

References

1. S. L. ROBINSON and O. D. SHERBY, *Acta Metallurgica* **17** (1969) 109.
2. J. E. BIRD, A. K. MUKHERJEE and J. E. DORN, in "Quantitative Relations Between Properties and Microstructure (Israel Universities Press, 1969) p. 255.
3. O. D. SHERBY, R. FRENKEL, J. NADEAU and J. E. DORN, *Trans. Met. Soc. AIME* **200** (1954) 275.
4. E. W. HART and D. LEE, *Met. Trans.* **2** (1971) 1245; S. L. ROBINSON, Ph.D. Dissertation, Stanford University, Stanford, Ca., 1972.
5. L. RAYMOND and J. E. DORN, *Trans. Met. Soc. AIME* **230** (1964) 560.
6. C. N. AHLQUIST and W. D. NIX, *Acta Metallurgica* **19** (1971) 373.
7. S. K. MITRA and D. MCLEAN, *Met. Sci. J.* **1** (1967) 192.
8. A. M. GERVAIS, J. T. NORTON and N. J. GRANT, *Trans. Met. Soc. AIME* **197** (1953) 1166 (stress drop test described on p. 1173).
9. I. S. SERVI and N. J. GRANT, *ibid* **191** (1951) 909.
10. S. L. ROBINSON and O. D. SHERBY, *Phys. Stat. Sol. (a)* **K119** (1970) 1.
11. M. E. FINE, *Rev. Sci. Instrum.* **28** (1957) 643.
12. J. J. JONAS, D. R. AXELRAD and J. L. UVIRA, *Acta Metallurgica* **19** (1971) 1053.
13. H. J. MCQUEEN and J. E. HOCKETT, *Met. Trans.* **1** (1970) 2997.
14. J. WEERTMAN, "High Temperature Creep Produced by Dislocation Motion", presented at John Dorn Memorial Symposium, Cleveland, Ohio, October (1972).

Received 10 September
and accepted 1 October 1973

STEVEN L. ROBINSON
CONRAD M. YOUNG
OLEG D. SHERBY
Department of Materials Science and Engineering,
Stanford University, Stanford,
California, USA

Crystal data for layer compounds in the series HfS_xSe_{2-x}

Hafnium disulphide and hafnium diselenide are members of a group of compounds which crystallize with the characteristic cadmium iodide layer structure. Although Greenaway and Nitsche [1] and McTaggart and Waddsley [2] quote X-ray data for these compounds no examination appears to have been made of their solid solutions. During work on the growth of a number of systems of cadmium-iodide-type layer compounds by iodine vapour transport we have grown and examined a number of these.

Work by Franzen and Graham [3], and by Stocks *et al* [4] has established that, for the lower sulphides, the system Hf-S is almost totally isomorphous with the system Zr-S. This is borne out by our present work on the dichalcogenides.

Single crystals of hafnium dichalcogenides and their solid solutions were grown in closed ampoules under the influence of a temperature gradient, using iodine as transporting agent. The