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.

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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).

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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

TABLE I Growth conditions used to grow hafnium disulphide, hafnium diselenide and their solid solutions by iodine vapour transport

Compound	Reaction temperature (°C)	Growth temperature (°C)	Crystal size (mm	× mm	× mm)	Growth time (h)
HfS_2	1010	1000	5	5	0.1	1000
but compare Conroy and Parks [7]						
	900	800	15	12	0.05	70
Nitsche [8]						
	900	800	5	5	0.2	48-72
Greenaway and Nitsche [1]						
	900	800	4	4	—	72
$HfS_{1.5}Se_{0.5}$	1010	1000	5	5	0.1	1000
$HfS_{1.0}Se_{1.0}$	1000	950	3	3	0.1	800
$HfS_{0.5}Se_{1.5}$	950	900	3	3	0.1	700
$HfSe_2$	900	850	5	5	0.1	700
but compare Greenaway and Nitsche [1]						
	900	800	4	4	—	72
Nitsche [8]						
	900	800	4	4	0.1	48-72

TABLE II Unit cell dimensions of trigonal solid solutions in the series HfS_xSe_{2-x}

Compound	a (Å)	c (Å)
HfS_2	3.623 ± 0.003	5.841 ± 0.006
but compare Nitsche [8]		
	3.62	5.88
Greenaway and Nitsche [1]		
	3.623 ± 0.003	5.88 ± 0.03
McTaggart and Waddsley [2]		
	3.635	5.837
$HfS_{1.5}Se_{0.5}$	3.653 ± 0.003	5.927 ± 0.008
$HfS_{1.0}Se_{1.0}$	3.682 ± 0.004	6.006 ± 0.007
$HfS_{0.5}Se_{1.5}$	3.711 ± 0.004	6.076 ± 0.008
$HfSe_2$	3.741 ± 0.003	6.143 ± 0.006
but compare Nitsche [8]		
	3.73	6.15
Greenaway and Nitsche [1]		
	3.733 ± 0.005	6.141 ± 0.005
McTaggart and Waddsley [2]		
	3.748	6.159

experimental method used has been described previously [5, 6]. Growth conditions used are given in Table I, although these are not the optimum for growth, being near the upper operating limits of the furnaces available. The compositions of the growth products were monitored from their change in weight upon oxidation to the oxide, which is assumed to be stoichiometric HfO_2.

By varying the ratio of S:Hf in the initial charge during growth of hafnium disulphide, the phase limits of the disulphide were found to be between 1.98 ± 0.02 and 1.91 ± 0.02. The single crystals obtained were of an orange colour and only the basal faces of the crystals were well-formed and with few growth features; the usual trigonal habit was poorly developed. To achieve even minimal transport along the ampoule the reaction temperature had to be raised above 1000° C for a growth time of 1000 h. The distance over which transport then occurred was never greater than 3 cm. At lower growth temperatures transport did not occur, but a mass of small intergrown crystals formed on the untransported charge. The slow rates of transport achieved are thought to be caused by a low reaction rate between the iodine transporter and the hafnium used, which was in the form of wire.

Small crystals of hafnium diselenide were grown similarly. By varying the Se:Hf ratio in the charge, diselenide crystals varying in composition from Se:Hf :: 1.98 ± 0.03 to Se:Hf :: 1.92 ± 0.03 were obtained. The growth products were metallic bronze in colour, with well-developed faces and edges. Crystals were transported a distance of 5 cm from the charge under the conditions quoted, but were often intergrown.

Single crystals of three compositions of solid solutions in the series HfS_xSe_{2-x} $(0 \leqslant x \leqslant 2)$ were grown. These varied in colour from transparent orange-red (sulphur-rich) to metallic bronze (selenium-rich). All solutions formed a single phase trigonal compound isomorphous with the disulphide and diselenide. X-ray powder data have been used to derive the unit cell dimensions listed in Table II. Cell axial lengths are almost linear functions of the composition variable, x, but slight quadratic deviations from linearity appear in the "c" dimension.

The oxide reduction technique showed the single crystal growth products to be close in composition to that of the mix of elements forming the original charge. Results of analyses are given in Table III.

TABLE III Compositions of solid solutions as determined by the oxide reduction technique

Compound	Sulphur fraction	Selenium fraction
$HfS_{1.5}Se_{0.5}$	1.52 ± 0.03	0.48 ± 0.03
$HfS_{1.0}Se_{1.0}$	1.03 ± 0.03	0.97 ± 0.03
$HfS_{0.5}Se_{1.5}$	0.51 ± 0.03	1.49 ± 0.03

References

1. D. GREENAWAY and R. NITSCHE, *J. Phys. Chem. Solids* **26** (1965) 9.
2. F. MCTAGGART and A. WADDSLEY, *Australian J. Chem.* **11** (1958) 445.
3. H. FRANZEN and I. GRAHAM, *J. Inorg. Nuclear Chem.* **28** (1966) 377.
4. K. STOCKS, G. EULENBERGER and H. HAHN, *Z. Anorg. allg. Chem.* **374** (1970) 318.
5. H. P. B. RIMMINGTON and A. A. BALCHIN, *Phys. Stat. Sol.* (*a*) **6** (1971) K47.
6. C. R. WHITEHOUSE, H. P. B. RIMMINGTON and A. A. BALCHIN, *ibid* (*a*) **18** (1973) 623.
7. L. CONROY and K. PARKS, *Inorg. Chem.* **7** (1968) 459.
8. R. NITSCHE, "Crystal Growth" (supplement to *J. Phys. Chem. Solids*), Proc. Intern. Conf. Boston (1966), edited by H. S. Peiser (Pergamon Press, New York, 1967) p. 215.

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On the methods for determining trap depth from glow curves

In a recent paper, Shalgaonkar and Narlikar [1] review some of the important methods used in the determination of trap depth from glow curves. The purpose of the present note is threefold:

1. suggesting the possibility of using the mentioned methods in their more modern versions which enable more accurate evaluation of the activation energies by simpler and more direct calculation;
2. pointing out the differences between thermally stimulated conductivity (TSC) and thermally stimulated luminescence (TSL) (usually called thermoluminescence, TL). In addition, a few more phenomena can be included in this category, for which the determination of activation energies is both relevant and important;
3. a few possible errors in the paper of Shalgaonkar and Narlikar are to be corrected.

The method of various heating rates due to Hoogenstraaten [2] mentioned in [1] has been generalized [3] in two ways. First, it has been shown that the method is applicable when non-linear heating rates are used, namely, that a