from 1 to 100 g: of the three faces indented, i.e. $(1\,1\,1)$, $(1\,\bar{1}\,0)$ and $(1\,1\,\bar{2})$, the second was found to be somewhat softer than the others, but the anisotropy of the VHN was not sufficiently pronounced to warrant discussion of details here.

The indenter was held under load for 10 sec in all cases; an investigation of the effect of holding-time on the VHN, using a 100 g load, showed that transient "indentation creep" [9] was insignificant except above 120° C, when the hardness dropped a few percent if the load was applied for 1 to 2 min. With a 100 g load the VHN was found to be effectively constant below 0° C at a value of 280, falling to about 250 at 20° C, 165 at 60° C, 110 at 110° C and to about 35 at 200° C.

Definite evidence of "ductility", i.e. slip bands near the indentation, was found at and above room temperature (Fig. 1), but not at significantly lower temperatures. With the present method of deformation the ductile-to-brittle transition appears to begin at about 0° C, as may also be inferred from the foregoing hardness data.

Fig. 2 shows the relation between the load and d, the length of the indentation diagonal as projected onto the crystal surface, at four temperatures. Each point is based on at least three measurements, made by optical and scanning electron-microscopy. In the "brittle" range, i.e. line (a) referring to −65° C, one has $L \propto d^{3.5}$ up to $L \simeq 30$g, which implies that the $\text{VHN} \propto L^{0.57}$, in reasonable agreement with the relation $\text{VHN} \propto L^{0.50}$ valid to about the same load level in germanium at room temperature [1]. Again, as in germanium, at higher loads the dependence of the VHN on load becomes less pronounced.

The VHN, evaluated as the ratio $2L/d^2$ kg mm^{-2}, for the points on the line (a), i.e. for loads of 1, 3, 10 and 30 g, is 64, 102, 163 and 265 respectively. With the 100 g load the measured VHN (275) is substantially less than the corresponding value obtained by extrapolation of the power-law relation (450). The lines (b), (c), (d), referring to 60, 110 and 200° C, in the order given, comply with the relation $L \propto d^2$, i.e. at these temperatures the VHN is no longer discernably load-dependent in the L-range investigated. This "conventional" behaviour can almost certainly be ascribed to the relative ease of slip and the concomitant, reduced, propensity to cracking.

References

1. R. K. BANERJEE and P. FELTHAM, *J. Mater. Sci.* 9 (1974) 1478.
2. M. J. HILL and D. J. ROWCLIFFE, *ibid* 9 (1974) 1569.
3. M. V. SWAIN, B. R. LAWN and S. J. BURNS, *ibid* 9 (1974) 175.
4. R. K. BANERJEE and P. FELTHAM, *Rev. Deform. Beh. Mater.* 1 (4) (1975) in press.
5. B. J. HOCKEY and B. R. LAWN, *J. Mater. Sci.* **10** (1975) 1275.
6. B. R. LAWN and M. V. SWAIN, *ibid* **10** (1975) 113.
7. B. R. LAWN and E. R. FULLER, *ibid* **10** (1975) 2016.
8. K. SUMINO and H. HASEGAWA, *Trans. Jap. Inst. Met. (Suppl)* 9 (1968) 749.
9. C. A. BROOKES, R. P. BURNARDS and J. E. MORGAN, *J. Mater. Sci.* **10** (1975) 2171.

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The effect of thermal cycling on the microstructure of the Pb–Ag eutectic

An important problem associated with the use of *in situ* composite materials at high temperatures is the prevention of microstructural coarsening. The stability of eutectic composites at uniform high temperature has been studied extensively (see [1]). In contrast, the thermal stability of eutectic alloys under more realistic conditions (e.g. in fluctuating temperatures [2–4], or in temperature gradients [1, 5–7]) has been comparatively neglected.

The present communication describes the results of an investigation of the thermal stability, under conditions of cyclically fluctuating temperature, of the fibrous Pb–Ag eutectic composite. This alloy has been shown [5] to exhibit unusual morphological changes after heating in a temperature gradient; and it was considered pertinent to investigate the effects on Pb–Ag of other non-isothermal heat-treatments.

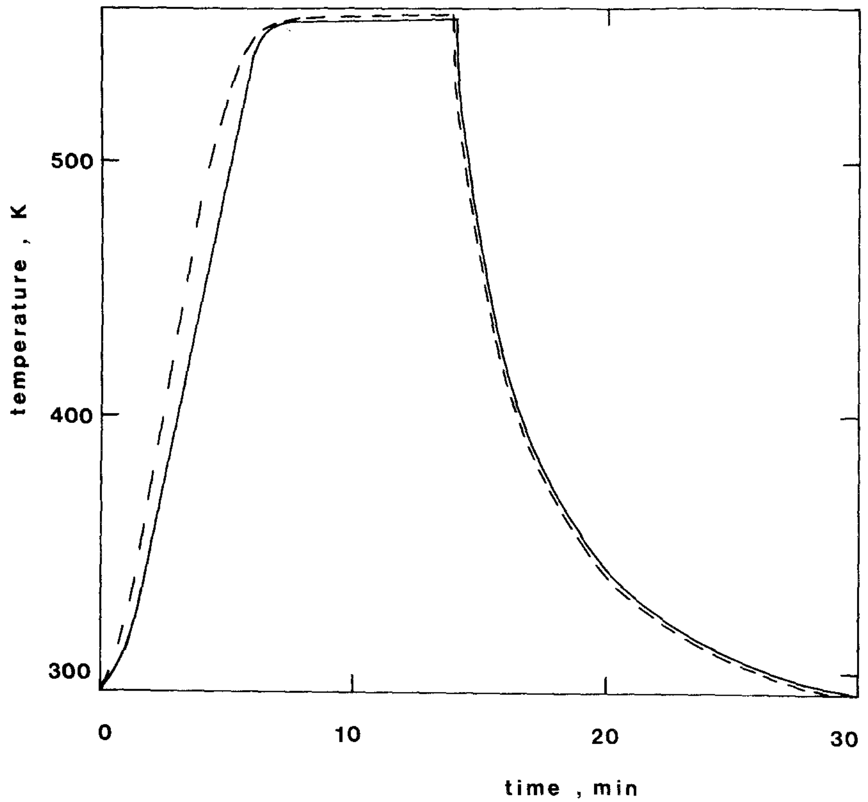

Figure 1 The thermal cycle. The solid line is the cycle measured at the centre of the dummy specimen; the dashed line refers to temperatures registered by the control thermocouple.

The Pb–Ag specimens employed in the work were taken from a unidirectionally solidified bar prepared as indicated previously [5]. Samples measuring about 5 mm × 7 mm × 7 mm were removed carefully from the bar using a fine jeweller's saw. In addition to selecting specimens for thermal cycling treatment, control specimens, and samples intended for isothermal annealing at 558 K, were extracted from the bar at regular intervals.

Thermal cycling of specimens (in the range 50 to 520 cycles) was accomplished using a 1 kW dual elliptical radiant furnace equipped with a Thermac programmable temperature controller. Specimens were placed in a tubular stainless steel jacket supported inside the furnace. A dummy specimen, containing a thermocouple for temperature measurement, was also included in the specimen jacket. The furnace-control thermocouple was attached to the exterior of the jacket, and the whole was surrounded by a tubular tantalum radiation shield. Oxidation of the specimens was prevented by an atmosphere of high-purity argon. The thermal cycle employed, as measured inside the dummy specimen, is shown in Fig. 1. Also included in this figure is a plot of time against the temperature of the control thermocouple. The accuracy of temperature measurement was ±2 K for both thermocouples.

The heat-treated specimens and the control specimens were sectioned transverse to the growth direction, and mounted in cold-setting plastic. The sections were prepared for optical metallography by conventional mechanical means, followed by etching with a cold solution of 1 volume of nitric acid to 5 volumes of water. Quantitative metallographic measurements were made at many positions on each transverse section. Specimens were subsequently prepared for examination in the scanning electron microscope as described previously [5].

Examination of the transverse sections showed that, although the thermally cycled samples exhibited a coarser morphology than the control

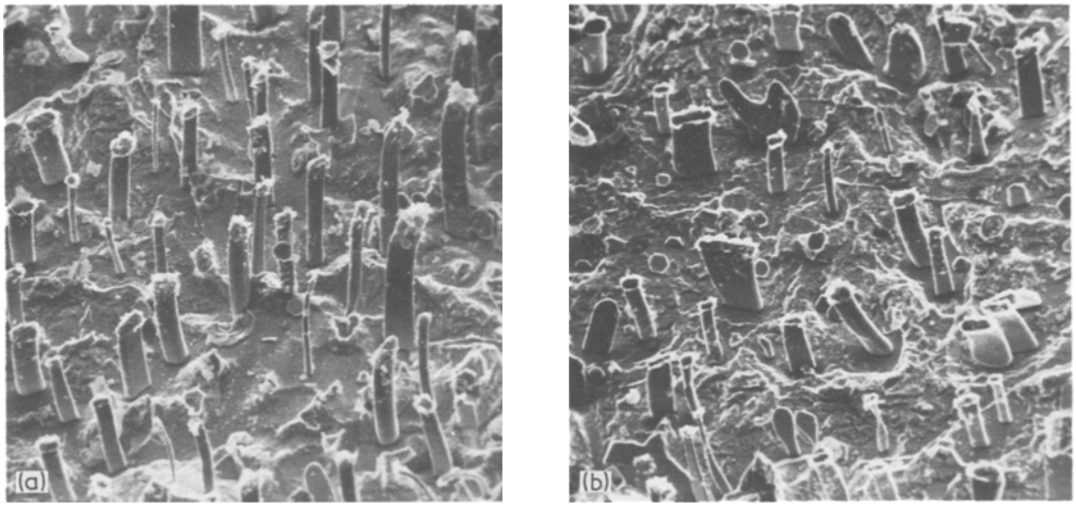

Figure 2 Scanning electron micrographs of typical regions of a specimen cycled 520 times, × 1375.

specimens, they generally retained an essentially rod-like structure (Fig. 2). Although unusual microstructural features (e.g. extended platelets, etc) were occasionally observed, their occurrence was not reproducible; and such features were not apparently similar to those observed after temperature-gradient annealing [5].

The microstructures observed after thermal cycling were not significantly different in appearance from those produced by the present isothermal annealing treatment. Finally, comparison of the rod densities of the thermally cycled and the isothermally annealed eutectic (after making appropriate corrections for the limited time spent at high temperature during the thermal cycle) failed to reveal any significant difference.

Of the possible phenomena that might have been expected to lead to thermal-cycling damage, two may be singled out as relevant to the Pb–Ag alloy. First, under the heading of differential thermal expansion, periodic strain differences between the two alloy phases of up to 0.3% might be expected during the above cycling treatment. This figure is roughly four times the strain to yielding of Pb or Ag at room temperature. Secondly, in connection with periodic fluctuations in alloy composition during the thermal cycle, the Ag phase exhibits a significant variation in composition within the temperature range used (0.75 mol% Pb at 573 K, 0.35 mol% Pb at 523 K). The conclusion to be drawn from the present study is that, significant though the above effects may appear, they are not of the right magnitude to lead to noticeable coarsening in Pb–Ag. Neither does there appear to be any obvious parallel between the behaviour of Pb–Ag in a temperature gradient and in a thermal cycle. In connection with the latter type of comparison, a cautionary note should be sounded: this is that, although not the case in our experiments, thermal cycling may often entail the generation of appreciable temperature gradients. In particular, materials of low thermal conductivity, and experiments where thermal cycles are generated by self-resistive heating come to mind. Care should be taken in such cases not to confuse temperature-gradient effects with those due to thermal cycling as such.

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References

1. D. R. H. JONES and G. J. MAY, *Acta Metallurgica* **23** (1975) 29.
2. G. GARMONG and C. G. RHODES, "Proceedings of the Conference on *In-Situ* Composites", Lakeville, USA (1972) Vol. 1, p. 251.
3. E. M. BREINAN, E. R. THOMPSON and F. D. LEMKEY, *ibid*, Vol. 2, p. 201.
4. H. BIBRING, *ibid*, Vol. 2, p. 57.

5. D. R. H. JONES, *Mat. Sci. Eng.* **15** (1974) 203.
6. D. R. H. JONES, *Metal Sci.* 8 (1974) 37.
7. M. McLEAN, *Scripta Met.* 9 (1975) 439.

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Electron microscopy and electron diffraction study of ordering in Ni_4W

The intermediate phase Ni_4W in the nickel–tungsten system undergoes an order–disorder transformation at 970 ± 10° C. The disordered phase is fcc while the ordered phase is bct with $c/a = 0.980$. Extensive electron microscopic and field-ion microscopic investigations have been carried out on the nature of ordering and the transformation from short range order (SRO) to long range order (LRO) in the structurally similar Ni_4Mo [1–6], but only one X-ray analysis [7] and one field-ion microscopic investigation on growth of Ni_4W from the disordered phase is reported in the literature [8]. The present communication reports some transmission electron microscopic observations of SRO and the development of LRO in Ni_4W.

Samples were taken in the form of thin sheets,

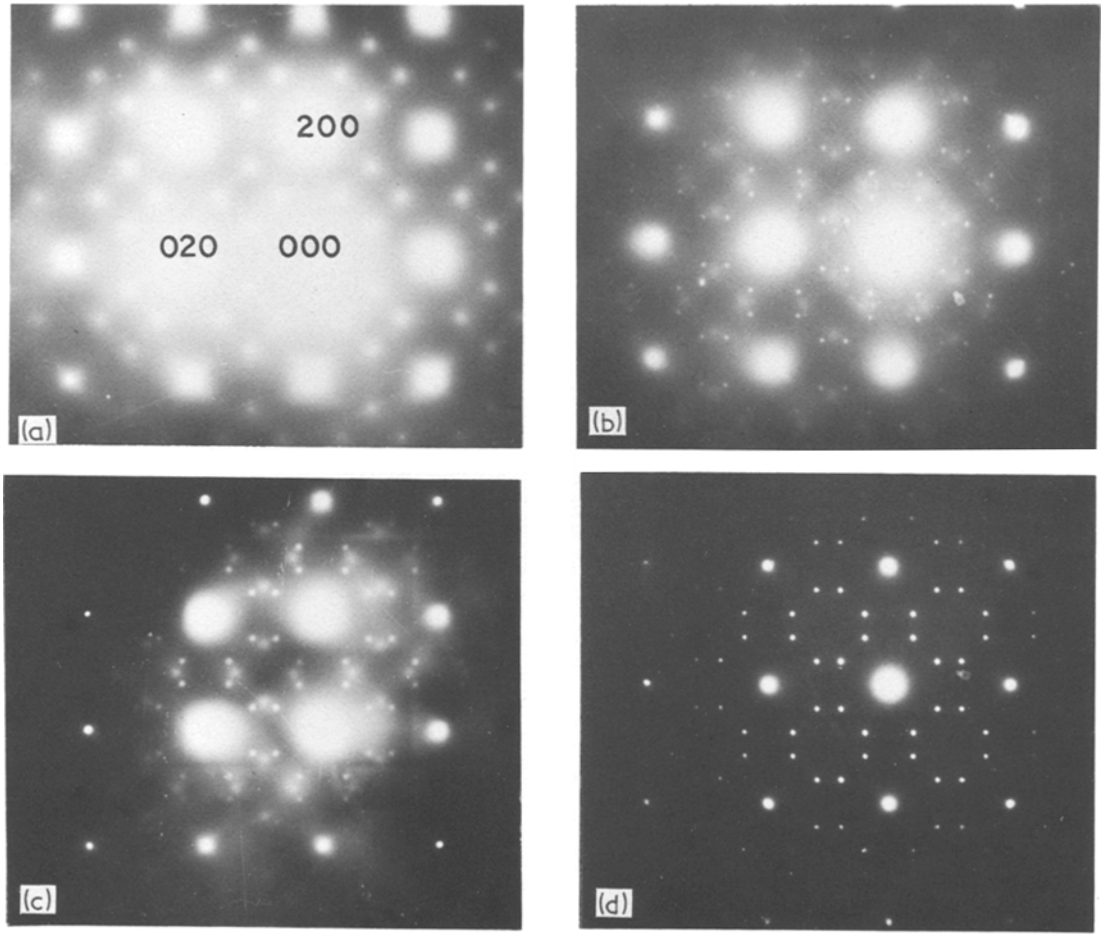

Figure 1 Electron diffraction patterns corresponding to the [0 0 1]* reciprocal lattice section of Ni_4W quenched from 1125° C and aged at 830° C for (a) 0 min (b) 15 min (c) 20 min (d) 30 min.