

# Room-temperature deformation of PdIn

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Room temperature tensile testing of the B2-structured compound Pd–52 at % In produced, a yield strength of 50 MPa, a fracture strength of 130 MPa, and limited tensile ductility ( $\sim 3\%$ ). Examination, in the transmission electron microscope, of the deformed microstructure indicated that it deforms by  $\langle 001 \rangle \{100\}$  slip. Extra reflections and streaking were observed in selected area electron diffraction patterns, which appear to be a thin foil effect.

## 1. Introduction

The intermetallic compound PdIn is being evaluated as a potential material for both dental prosthetics and jewellery because of its colour, which ranges from copper-coloured near the stoichiometric composition to gold-coloured when palladium-rich. It is B2-structured, or ordered body-centred cubic, and exists over a range of compositions on both sides of stoichiometry (39–52 at % In) [1]; however, little is known of either its mechanical properties or modes of deformation.

B2-structured intermetallic compounds generally deform by either  $\langle 001 \rangle$  or  $\langle 111 \rangle$  slip depending upon the composition and temperature of deformation [2].  $\langle 001 \rangle$  slip provides only three independent slip systems, which is insufficient for generalized plastic flow in polycrystals. Some tensile ductility has been observed in B2-structured intermetallics where only  $\langle 001 \rangle$  slip was observed [4–7], but the reasons for this ductility are, as yet, not clear. In contrast,  $\langle 111 \rangle$  slip provides five independent slip systems, although this alone does not guarantee ductility [3].

This letter reports the microstructure, room-temperature tensile properties and deformation modes of cast Pd–52 at % In, and relates these to other isostructural compounds.

## 2. Experimental procedure

An alloy of composition Pd–52 at % In was cast into two dumb-bell-shaped tensile specimens (gauge length 25 mm, gauge diameter 3 mm). The microstructure of the tensile samples was examined using both optical microscopy, of samples etched in aqua regia, and transmission electron microscopy (TEM), using a Jeol 2000FX operated at 200 kV, of thin foils made from ion-milled discs. The crystal structure was also determined from Debye–Scherrer X-ray diffraction patterns using a Straumanis camera and nickel-filtered  $\text{CuK}\alpha$  radiation, of powders filed from the tensile specimens after testing and annealed at 873 K in flowing argon for 1 h. Estimated errors in lattice parameter measurement (see [8]) were  $\pm 2 \times 10^{-3}$  nm. The tensile samples were strained to fracture under

tension at room temperature and the fracture surfaces were examined in a scanning electron microscope (SEM). In addition, thin foils from a cylinder of Pd–52 at % In, lightly compressed at room temperature, were examined for analysis of the dislocation structures. Thin foils were also examined after both cooling to  $\sim 100$  K using a Gatan cryostage and heating to 723 K in a Gatan heating stage.

## 3. Results and discussion

### 3.1. Microstructure

Optical microscopy revealed that the cast Pd–52 at % In was single phase with a grain size of about 100  $\mu\text{m}$  (Fig. 1). A number of voids, presumably casting defects, were observed. Examination of the X-ray data (Table I) revealed that the reflections corresponded to the fundamental reflections for a B2 structure. The lattice parameter was determined from a linear regression analysis of the measured lattice parameter against the Nelson–Riley function,  $N-R = 1/2 (\cos^2\theta/\sin\theta + \cos\theta^2/\theta)$ , extrapolated to  $N-R = 0$ , to be 0.3238 nm, which is consistent with previous data on this particular composition [9, 10]. No superlattice

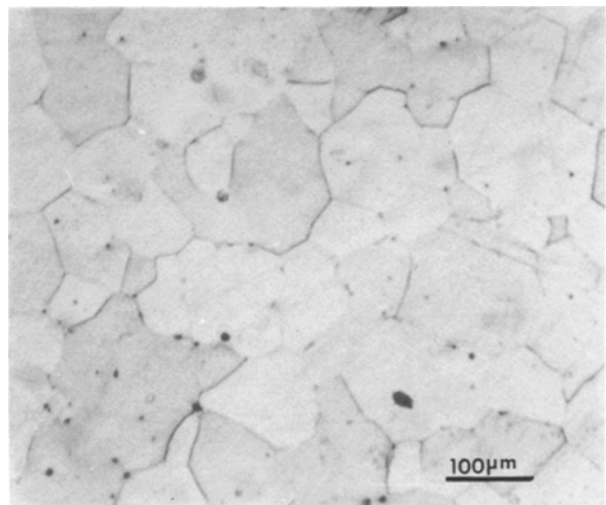


Figure 1 Optical micrograph of as-cast Pd–52 at % In.

TABLE I Observed  $d$ -spacings and relative intensities from Pd-52 at % In

| $d$ -spacing (nm) | Relative intensity | $hkl$ values for PdIn |
|-------------------|--------------------|-----------------------|
| 0.229             | vs                 | 110                   |
| 0.162             | m                  | 200                   |
| 0.133             | m                  | 211                   |
| 0.115             | m                  | 220                   |
| 0.103             | m                  | 310                   |
| 0.0939            | w                  | $222 K_{\alpha_1}$    |
| 0.0938            | w                  | $222 K_{\alpha_2}$    |
| 0.8535            | m                  | $321 K_{\alpha_1}$    |
| 0.0853            | m                  | $321 K_{\alpha_2}$    |
| 0.0812            | vw                 | $400 K_{\alpha_1}$    |
| 0.0810            | vw                 | $400 K_{\alpha_2}$    |

vs = very strong, m = medium, w = weak, vw = very weak.

reflections were observed. This is not surprising because the X-ray scattering factors for palladium ( $f_{Pd}$ ) and indium ( $f_{In}$ ) are very close [11] and the intensities of superlattice reflections are proportional to  $(f_{Pd} - f_{In})^2$ .

Examination of the microstructure by TEM revealed that the composition was single phase in agreement with earlier studies [10]. Selected-area diffraction (SAD) patterns obtained in the TEM from thin foils of PdIn also indicated a B2 structure, very weak superlattice reflections were observed (Fig. 2). Streaking was observed in the SAD patterns around fundamental reflections parallel to the  $\langle 112 \rangle$  direction. Extra reflections, indexed as  $a/12\langle 112 \rangle$ , associated with the streaking can also be seen within the SAD patterns. However, because the extra reflections were not observed by X-ray diffraction the indication is that they are a thin-foil effect. Dark-field imaging with the  $a/12\langle 112 \rangle$  reflections did not produce any significant mottled contrast consistent with possible martensitic microstructures [12], although some streaking was observed in the bright-field images (see Fig. 4a).

To investigate further the origin of the extra reflections and streaking in the SAD patterns, two ex-

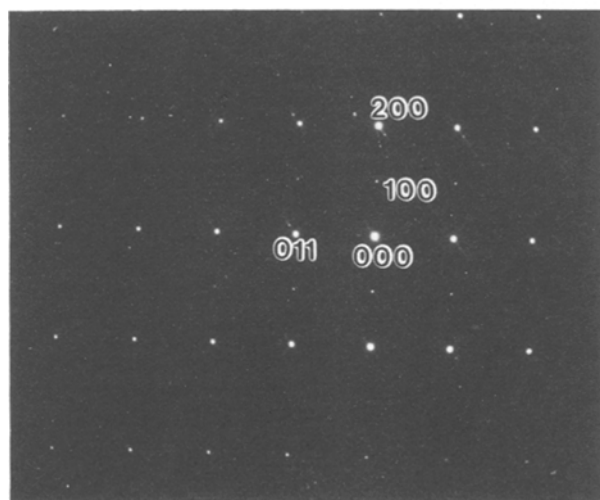


Figure 2 Selected-area diffraction pattern from  $\langle 011 \rangle$  zone axis, shows streaking parallel to  $\langle 112 \rangle$  together with  $a/12\langle 112 \rangle$  extra reflections.

periments were performed. First, a thin foil of PdIn was cooled to  $\sim 100$  K in a TEM cryostage. Cooling had no effect upon either the streaking or extra reflections, suggesting that this phenomenon is not associated with pre-martensitic effects which are commonly observed in non-stoichiometric B2 alloys such as nickel-rich NiAl [13]. Second, the foil was heated to 723 K. Similarly, this had no observable effect on either the extra reflections or the streaking. If these features were attributable to tweed microstructures, commonly observed in B2 compounds [14], it might be expected that the intensity of the streaking would increase as the temperature increased, due to the increase in phonon scattering at higher temperatures. The origin of these extra reflections is, at present, unknown. It is pertinent to note that Harris *et al.* [9] observed extra  $(401)/(322)$  reflections when they examined PdIn alloys in the range 49–54 at % In by X-ray diffraction. The origin of this reflection was also not understood, but it was suggested to occur due to further ordering within the B2 structure. No such reflections were observed here.

### 3.2. Deformation behaviour

The tensile samples exhibited yield strengths of 46 and 50 MPa, elongations of 2.0% and 3.6% and fracture strengths of 95 and 132 MPa, respectively, the variation presumably indicating the heterogeneity of the cast structure. The fracture surfaces were predominantly intergranular with a few grains exhibiting transgranular cleavage (Fig. 3). Numerous small voids (1–10  $\mu\text{m}$  diameter) were observed, consistent with the optical metallography, often at triple points. These may have contributed to failure. Presumably, suitable thermo-mechanical processing after casting which decreased both the porosity and the grain size would improve the ductility of this alloy.

Thin foil examination of the compressed alloy revealed a single-phase microstructure which contained a high density of dislocations. The Burgers' vector of

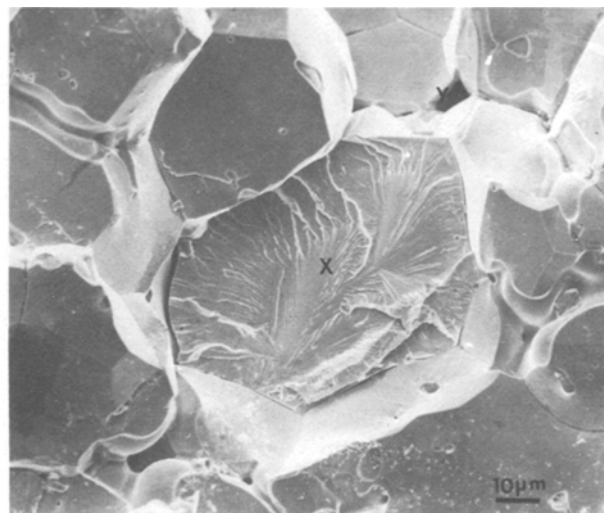


Figure 3 Scanning electron micrograph of fracture surface from Pd-52 at % In. Failure principally by intergranular fracture. Note transgranular facet marked X and void marked Y.

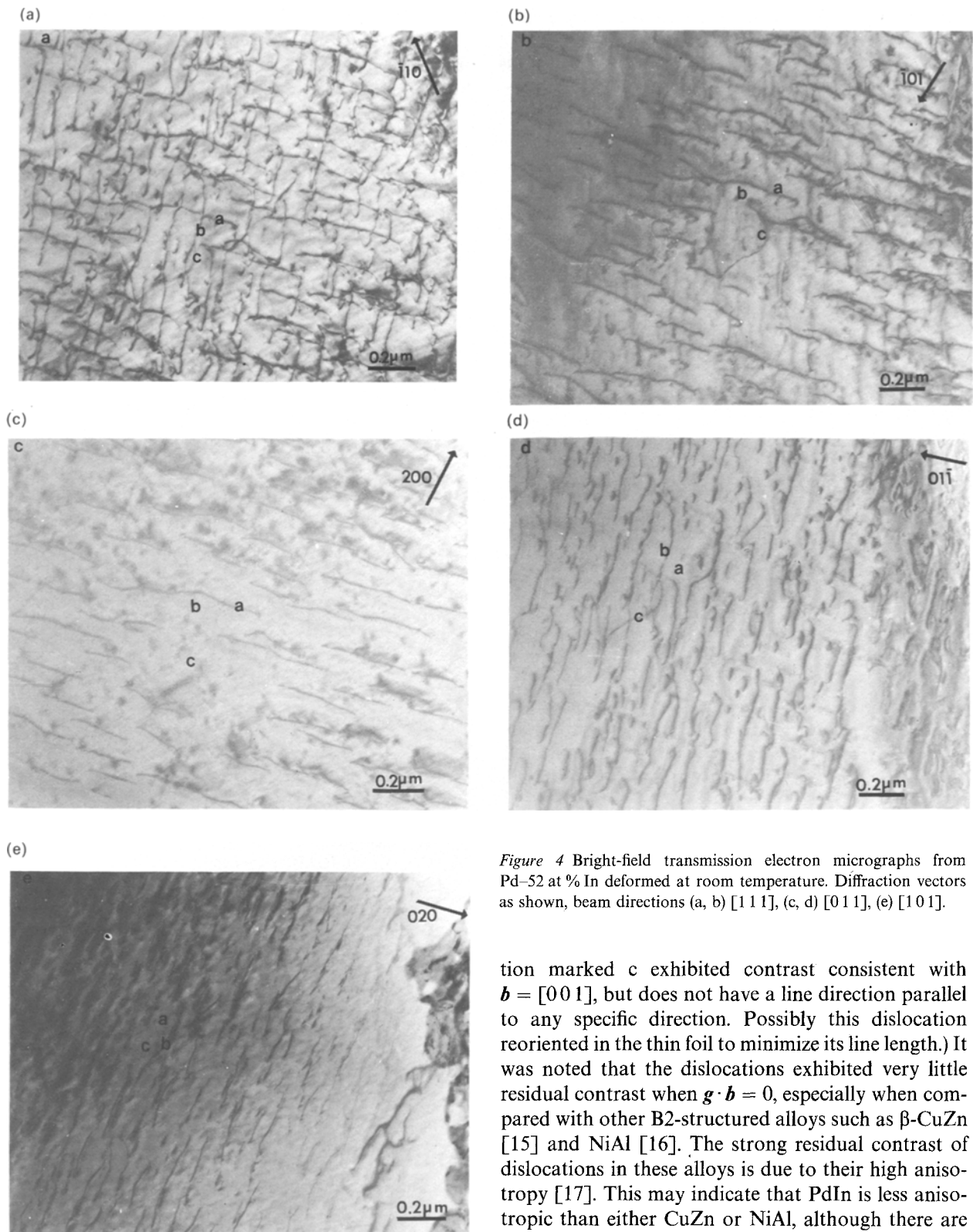


Figure 4 Bright-field transmission electron micrographs from Pd-52 at % In deformed at room temperature. Diffraction vectors as shown, beam directions (a, b)  $[111]$ , (c, d)  $[011]$ , (e)  $[101]$ .

these dislocations was found using the  $\mathbf{g} \cdot \mathbf{b} = 0$  invisibility criterion. For example in Fig. 4, the dislocations marked a are invisible when imaged with  $\mathbf{g} = 020$  and  $01\bar{1}$  reflections, indicating  $\mathbf{b} = [100]$ . Similarly, the dislocations labelled b, show invisibility when  $\mathbf{g} = 200$  and  $\bar{1}01$ , indicating that  $\mathbf{b} = [010]$ . The line directions of the dislocations were found by trace analysis. Dislocations marked a have a line direction of  $[010]$ , and those marked b a line direction of  $[100]$ . Thus the dislocations are edge dislocations with  $\mathbf{b} = \langle 001 \rangle$  and  $\mathbf{u} = \langle 010 \rangle$ , therefore glide is on  $\{100\}$  planes. (Note that the disloca-

tion marked c exhibited contrast consistent with  $\mathbf{b} = [001]$ , but does not have a line direction parallel to any specific direction. Possibly this dislocation reoriented in the thin foil to minimize its line length.) It was noted that the dislocations exhibited very little residual contrast when  $\mathbf{g} \cdot \mathbf{b} = 0$ , especially when compared with other B2-structured alloys such as  $\beta$ -CuZn [15] and NiAl [16]. The strong residual contrast of dislocations in these alloys is due to their high anisotropy [17]. This may indicate that PdIn is less anisotropic than either CuZn or NiAl, although there are no data available on the elastic constants of PdIn. In accordance with the calculations for dislocation line energy of anisotropic B2-structured materials, dislocations of the type  $\mathbf{b} = \langle 001 \rangle$  on  $\{100\}$  planes should be stable about their edge orientation [15, 17] and are thus expected. That the dislocations are observed to be edge in character suggests that the non-edge components are more mobile than the edge dislocations.

Because  $\langle 001 \rangle$  slip provides only three independent slip systems, which are insufficient for general, volume-conserving plastic flow in polycrystalline structures, the tensile elongation exhibited by PdIn is rather surprising, especially as porosity appears to be one of the causes of premature failure. However, as

noted earlier, room-temperature ductility has also been observed in other B2-structured intermetallic compounds which exhibited  $\langle 001 \rangle$  slip; both stoichiometric NiAl [4, 5] and Ni-30Al-20Fe [6] have shown  $\sim 2.5\%$  plastic elongation and AuZn has exhibited  $\sim 40\%$  tensile elongation before failure at room temperature [7]. The source of the ductility in these alloys is at present unclear, the origins of ductility in B2 alloys exhibiting  $\langle 001 \rangle$  slip have been discussed elsewhere [2, 6]. B2-structured compounds such as NiTi [4] and Co(Zr, Ni) [18] exhibit  $\langle 001 \rangle$  slip, but are ductile through stress-induced pseudoelasticity associated with a reversible martensitic transformation. There was no evidence of any martensitic structures in PdIn, which would suggest that stress-induced pseudoelasticity was not the cause of this ductility.

#### 4. Conclusions

In summary, microstructural examination and tensile testing of Pd-52 at % In at room temperature found:

1. limited tensile ductility at room temperature, the presence of voids and casting defects may have reduced its ductility;
2. it deforms by  $\langle 001 \rangle \{100\}$  slip at room temperature;
3. extra reflections and streaking were observed by transmission electron microscopy, these remained unaffected by both *in situ* heating and cooling experiments. It appears therefore that they were thin-foil effects.

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#### References

1. T. B. MASSALSKI (ed.), "Binary Phase Alloy Diagrams" (ASM, Metals Park, Ohio) p. 1390.
2. I. BAKER and P. R. MUNROE, "Properties of B2 Compounds", in *High Temperature Aluminides and Intermetallics*, edited by S. H. Whang, C. T. Liu, D. P. Pope and J. O. Stiegler, TMS (1990) p. 425.
3. C. C. LAW and M. J. BLACKBURN, Report FR-18674-4, Pratt and Whitney Group (1985).
4. A. G. ROZNER and R. J. WASILEWSKI, *J. Inst. Metals* **94** (1966) 169.
5. K. H. HAHN and K. VEDULA, *Scripta Metall.* **23** (1989) 7.
6. S. GUHA, P. R. MUNROE and I. BAKER, *ibid.* **23** (1989) 897.
7. A. R. CAUSEY and E. TEGHTSOONIAN, *Met. Trans. A* **1** (1970) 1177.
8. I. BAKER, B. HUANG and E. M. SCHULSON, *Acta Metall.* **36** (1988) 493.
9. I. R. HARRIS, M. NORMAN and A. W. BRYANT, *J. Less-Common Metals* **16** (1968) 427.
10. D. FORT, I. R. HARRIS and R. E. SMALLMAN, *ibid.* **16** (1973) 263.
11. B. D. CULLITY, in "Elements of X-Ray Diffraction" (Addison-Wesley, Reading, MA, 1967) p. 475.
12. I. M. ROBERTSON and C. M. WAYMAN, *Met. Trans. A* **15A** (1984) 1353.
13. *Idem.*, *Phil. Mag.* **48** (1983) 421.
14. *Idem.*, *ibid.* **48** (1983) 629.
15. A. K. HEAD, *Phys. Status Solidi* **19** (1967) 185.
16. N. RUSOVIC and H. WARLIMONT, *ibid.* **44** (1977) 609.
17. D. L. YANEY, A. R. PELTON and W. D. NIX, *J. Mater. Sci.* **21** (1986) 2083.
18. C. LALL, M. H. LORETTO and I. R. HARRIS, *Acta Metall.* **26** (1978) 1631.

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