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Small-punch technique for measurement of material degradation of irradiated ferritic alloys

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The yield stress and fracture strain of irradiated ferritic alloys were measured using a small-punch test. The characteristics of hardening and intergranular embrittlement induced during neutron irradiation (0.94×10^{23} n m^{-2}) at 395 °C in ferritic alloys doped with Cu, P and/or C were investigated. The effect of neutron irradiation on hardening was found to be greater in alloys doped with Cu and/or P than in C-containing alloys. The neutron irradiation produced a more substantial increase in the ductile–brittle transition temperature in the Cu-doped alloy compared with the other alloys and cause a significant decrease in the ductility of the Cu-doped alloy. Intergranular and transgranular fracture occurred in the alloys doped without and with C, respectively. The neutron irradiation did not alter the fracture mode.

1. Introduction

Neutron irradiation gives rise to an increase in the ductile–brittle transition temperature (DBTT) by inducing hardening in ferritic steels. Neutron irradiation-induced hardening is attributed to the formation of defect clusters and precipitates which hinder the dislocation motion. The characteristics of hardening and DBTT shift caused by neutron irradiation are related to the alloy composition and the impurity content in ferritic steels [1, 2]. It has been shown that the presence of Cu strongly promotes hardening and embrittlement. Atom probe-field ion microscopy studies [3] have demonstrated that extremely fine Cu- and/or P-rich precipitates play an important role in radiation damage in ferritic alloys, although the mechanism is not fully understood. Moreover, during high-temperature neutron irradiation, solute segregation to defect sinks such as grain boundaries and free surfaces proceeds because of inverse Kirkendall effects and/or the formation of defect–solute complexes arising from the dynamic interaction between defect and solute fluxes [4]. It is possible that the intergranular segregation of metalloid impurities occurring under thermal or irradiation environments facilitates embrittlement by weakening grain-boundary cohesion [5]. Therefore, it is interesting to investigate the combined effect of hardening and solute segregation induced during neutron irradiation on intergranular embrittlement in Fe-base alloys.

The present paper reports is a study on the effect of neutron irradiation on intergranular solute segregation and embrittlement in ferritic alloys. The effect of P, Cu and/or C on changes in the yield strength, ductility and the DBTT induced during neutron irradiation is examined. Owing to size limitations in the irradiation facility it was impossible to use larger, conventional specimens. The small-punch test, as a new specimen technique, was therefore used for measurement of mechanical properties of irradiated and non-irradiated materials.

2. Experimental procedure

Several iron-base alloys doped with P, Cu and/or C were made for this study by an electron beam-melting method. The chemical composition of the alloys, designated Heats I–V, is shown in Table I. The electron beam-melted alloys were cold rolled to plates which were first heat treated at various temperatures for 1 h and then at 600 °C for 1 h. The initial heating temperatures were 875, 918 and 972 °C for Heat I, Heats II and III and Heats IV and V, respectively. The heat treatments were followed by water quenching. The resulting microstructure was ferrite for the alloys without carbon and martensite and bainite mixture for the alloys with carbon. The grain size of ferrite or prior austenite was varied in the range 75–150 μm. Small punch (SP) specimens of dimensions 10 mm × 10 mm × 0.5 mm were machined from the heat-treated alloys and mechanically polished using 600 grit emery paper. SP specimens were irradiated to 0.94×10^{23} n m^{-2} ($E > 0.1$ MeV) in the low-temperature neutron irradiation facility at Oak Ridge National Laboratory. The neutron irradiation experiment was performed at 395 °C for 126.5 h. The irradiated specimens were tested after the activation of specimens had decayed sufficiently. In order to extract the effect of neutron irradiation on the mechanical properties, non-irradiated SP specimens were aged at 395 °C for 126.5 h under vacuum (10^{-2} Pa).

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TABLE 1 Chemical composition of Fe-based alloys (wt %)

	C	Cu	P	S	O	N
Heat I (Cu-doped)	0.0007	0.2957	0.0039	0.0020	0.0035	0.0025
Heat II (P-doped)	0.0012	0.0011	0.0392	0.0016	0.0047	0.0016
Heat III (P–Cu-doped)	0.0008	0.2971	0.0520	0.0019	0.0072	0.0020
Heat IV (P–C-doped)	0.0927	0.0016	0.0541	0.0019	0.0016	0.0009
Heat V (P–Cu–C-doped)	0.0959	0.2958	0.0444	0.0013	0.0014	0.0007

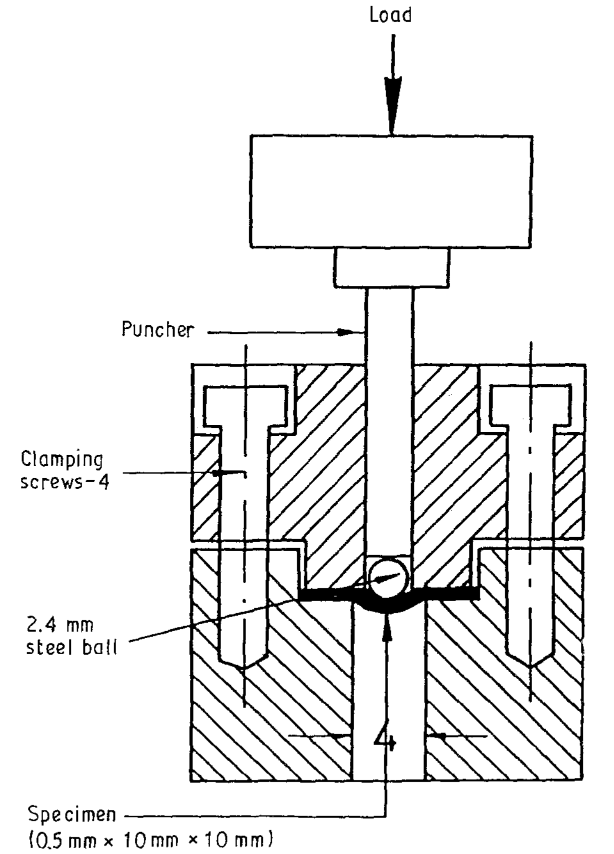

Figure 1 Loading and specimen support configuration for the small punch test.

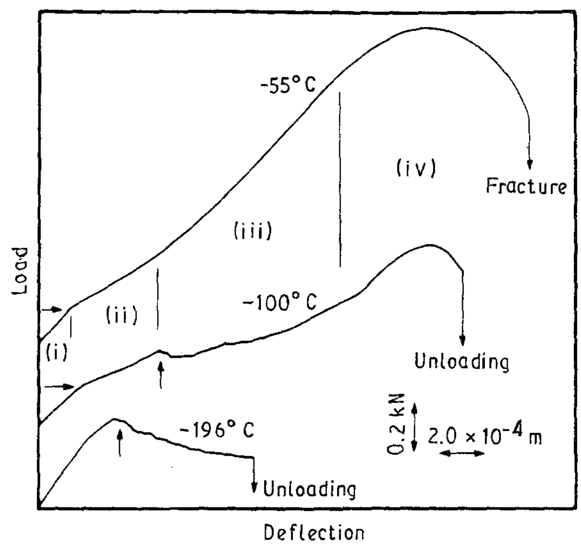

Figure 2 Load–deflection curves obtained from SP tests on non-irradiated specimens of P–Cu-doped alloy (Heat III) at various temperatures.

SP tests were performed at various temperatures by punching non-irradiated and irradiated specimens in a specially designed holder using an Instron testing machine. The SP experimental configuration [6–8] consists of a clamped centre-loaded specimen disc. The SP specimen holder consists of an upper and lower die, and four clamping screws. Using this specimen holder, the specimens are prevented from cupping upward during punching; therefore plastic deformation is concentrated in the region below the punch (steel ball), as shown schematically in Fig. 1. The SP specimens were deformed using a cross head speed of 2.1×10^{-5} m s^{-1}. The load–deflection curve was plotted on an X–Y recorder. The deformation and fracture properties were determined from the load–deflection curve. The fracture mode in the non-irradiated and irradiated SP specimens of the various alloys was examined using scanning electron microscopy.

3. Results and discussion

Fig. 2 shows load–deflection curves obtained from the SP test on the non-irradiated specimen of the P–Cu-doped alloy (Heat III) at various temperatures. As shown in Fig. 1, the load–deflection curve obtained from the SP test is divided into (i) an elastic bending regime, (ii) a plastic bending regime, (iii) a plastic membrane stretching regime, and (iv) a plastic instability regime [6, 8]. The arrows pointing to the right indicate the yielding load P_y (kN) corresponding to the boundary between regimes (i) and (ii). Mao and Takahashi [9] have established an empirical correlation between the yield strength σ_y (MPa) and P_y (kN) using materials of various strengths and specimen geometries, i.e. $\sigma_y = 360\ P_y/t^2$ where t (mm) is the specimen thickness. The yield strength was estimated using this equation. Fig. 3 shows temperature dependence of the yield stress of non-irradiated material predicted by the small punch test. The results have good agreement with that obtained from simple tension tests [10]. The temperature dependence of the yield strength estimated from the SP results for the non-irradiated and irradiated alloys is shown in Fig. 4. Three groups, Cu-doped alloy (I), P-doped and P–Cu-doped alloys (II and III), and P–C-doped and P–Cu–C-doped alloys (IV and V), exhibited the same temperature dependence of the yield strength. It is clear that the effect of neutron irradiation on hardening depends strongly on the composition of the alloys. The addition of Cu and/or P greatly facilitated neutron irradiation-induced hardening. However, the hardening effect was mitigated in the presence of carbon.

The strengthening effect induced during neutron irradiation is probably caused by the creation of defect

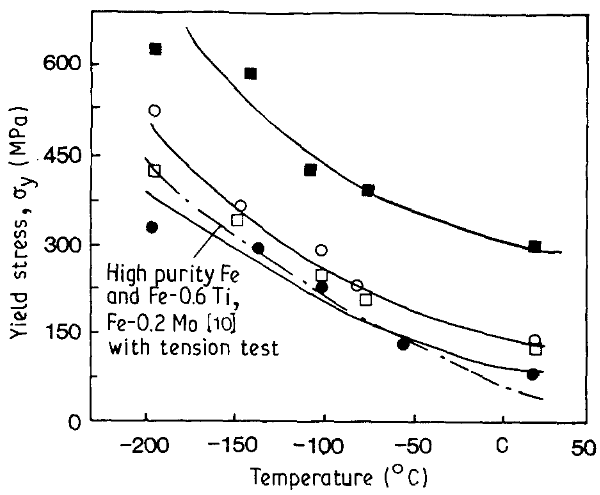

Figure 3 Temperature dependence of the yield stress predicted by small punch test and simple tension test. (■) Fe + C, (○, ●, □) Fe without C.

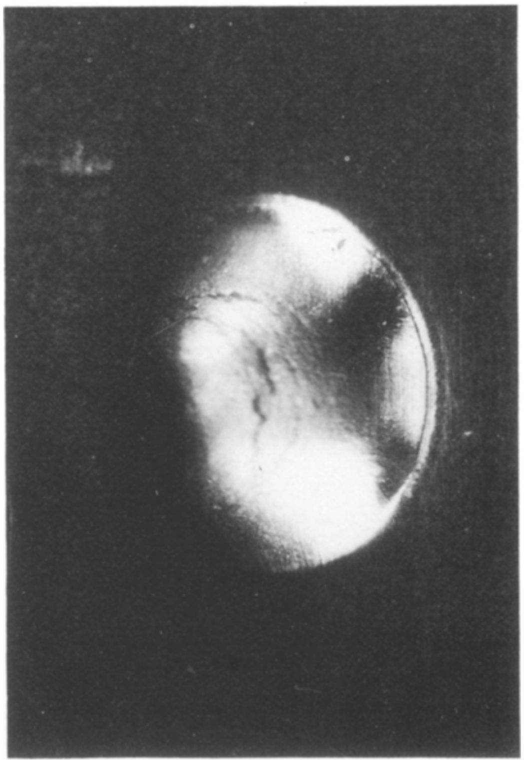

Figure 5 Fractured SP specimen (at room temperature).

clusters and the formation of fine Cu- and/or P-rich precipitates which increase the athermal and thermal stress components, respectively, by dragging the dislocation motion. The different characteristics of irradiation-induced hardening between the Cu-doped alloy (I) and the P-doped (II) or P–Cu-doped (III) alloy are mainly related to the magnitude of the athermal stress change. In the P-containing alloys (II and III), defect clusters are likely to be less densely distributed in the grain matrix because defect fluxes are expended to promote greatly intergranular P segregation [7]. Thus the athermal stress component in the alloys with P (II and III) might not be increased as greatly as in the Cu-doped alloy (I). Furthermore, the weak effect of irradiation on hardening observed for the alloys doped with C (IV and V) is probably ascribed to the depletion of irradiation-induced C- and/or P-rich precipitates and defect cluster formation arising from stronger binding of defects with C than with Cu or P [11]. Fig. 5 shows the SP specimen fractured at room temperature. The circumferential crack can be seen in Fig. 5. The fracture strain of the SP specimen was determined from the load–deflection curve [8]

$$\varepsilon_f = \beta\,(\delta^*/t)^{3/2} \qquad (1)$$

where, δ^* is the deflection at the load dropping point, and β is dependent on specimen size ($\beta = 0.15$, in this case). Fig. 6 shows the variation of the fracture strain with temperature obtained from the SP tests on the non-irradiated and irradiated specimen of the various alloys. In the non-irradiated specimen tests the P-doped and P–Cu-doped alloys (II and III) exhibited a higher DBTT than the copper-doped and carbon-

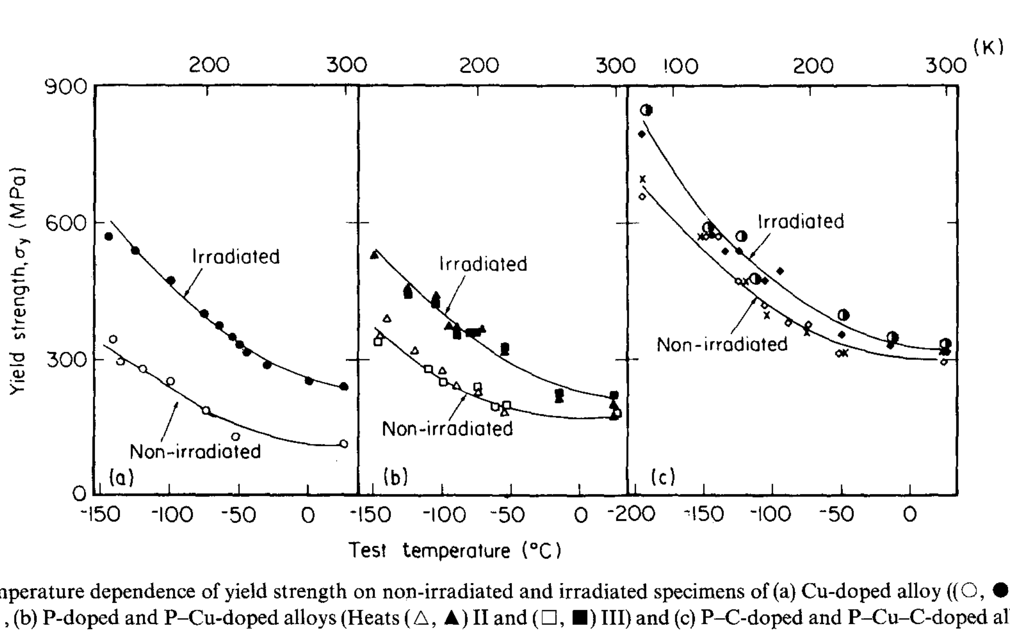

Figure 4 Temperature dependence of yield strength on non-irradiated and irradiated specimens of (a) Cu-doped alloy ((○, ●) Heat I; (–––) Pure Fe [10], (b) P-doped and P–Cu-doped alloys (Heats (△, ▲) II and (□, ■) III) and (c) P–C-doped and P–Cu–C-doped alloys (Heats (◇, ◆) IV and (×, ◑) V).

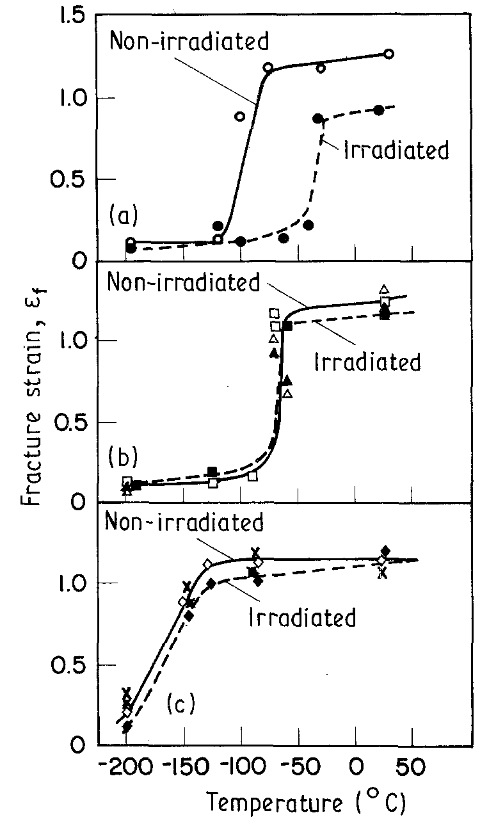

Figure 6 Variation of fracture strain with temperature obtained from SP tests on non-irradiated and irradiated specimens of (a) Cu-doped alloy ((○) Heat I), (b) P-doped and P–Cu-doped alloys (Heats (△) II and (□) III), and (c) P–C-doped and P–Cu–C-doped alloys (Heats (◇) IV and (×) V).

containing alloys (I, IV and V). The neutron irradiation produced a greater shift of the DBTT to higher temperatures in the Cu-doped alloy (I) than in the other alloys. The DBTT was almost unchanged by the neutron irradiation in the P-doped and P–Cu-doped alloy (II and III) and it was slightly increased in C-doped alloys (IV and V). It should be noted that the effect of neutron irradiation on the DBTT shift is not necessarily consistent with that on hardening. The fracture strain decrease of Cu-doped alloy with temperature is shown in Fig. 7. As shown, the temperature for maximum strain change is the DBTT. Scanning electron micrographs on the fracture surfaces in the irradiated specimens tested at $-196\,^{\circ}C$ are shown in Fig. 8. The SP specimens of the Cu- and/or P-doped alloys (I–III) fractured along grain boundaries, while those of the C-containing alloys (IV) exhibited transgranular fracture. It was found that the fracture surface morphology remained the same in the non-irradiated and irradiated specimens. Abiko *et al.* [12] have suggested that intergranular segregation of C produces a toughening effect and thereby the occurrence of intergranular fracture becomes difficult in the C-containing alloys.

The susceptibility of the ferritic alloys to irradiation-induced intergranular embrittlement is now briefly discussed in the light of the result of intergranular solute segregation, although the details are presented in [7]. The neutron irradiation intensified intergranular segregation of S in the Cu-doped alloy (I) where intergranular P segregation is not identified, while it reduced the amount of segregated S in the alloys (II and III) with P segregation. The intergranular segregation of P in the alloys (I–III) was facilitated during neutron irradiation. It is clear that the enrichment of intergranular S segregation as well as the great hardening effect can be attributed to the large increase in the DBTT induced during irradiation in the Cu-doped alloy (I). On the other hand, in the P-containing alloys (II and III) the effect of solute segregation on the DBTT is small. The enhancement of P segregation during neutron irradiation gives rise to grain-boundary weakening. However, the embrittlement effect is offset by the grain-boundary strengthening effect caused by the mitigation of S segregation. Thus the alloys (II and III) almost do not exhibit a small change in the DBTT during irradiation.

4. Conclusions

Small-specimen test has been carried out to measure the effect of neutron irradiation of Fe-based alloys doped with P, Cu and/or C. Irradiation-induced hardening was observed more prominently for Cu- and/or P-doped alloys (I–III) than for C-containing alloys (IV and V). It appears that the great irradiation effect on hardening in the Cu- and/or P-doped alloys is related to an increase in both the thermal and athermal stress components. SP tests have demonstrated that the neutron irradiation gives rise to a significant increase in the DBTT in the Cu-doped alloy (I), while it does not strongly affect the DBTT in the alloys doped with P or C (II–V). Neutron irradiation causes a significant

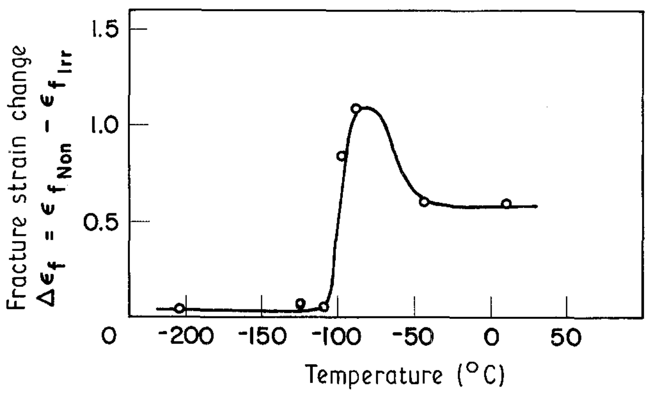

Figure 7 Fracture strain change of Cu-doped alloy (Heat I) with temperature.

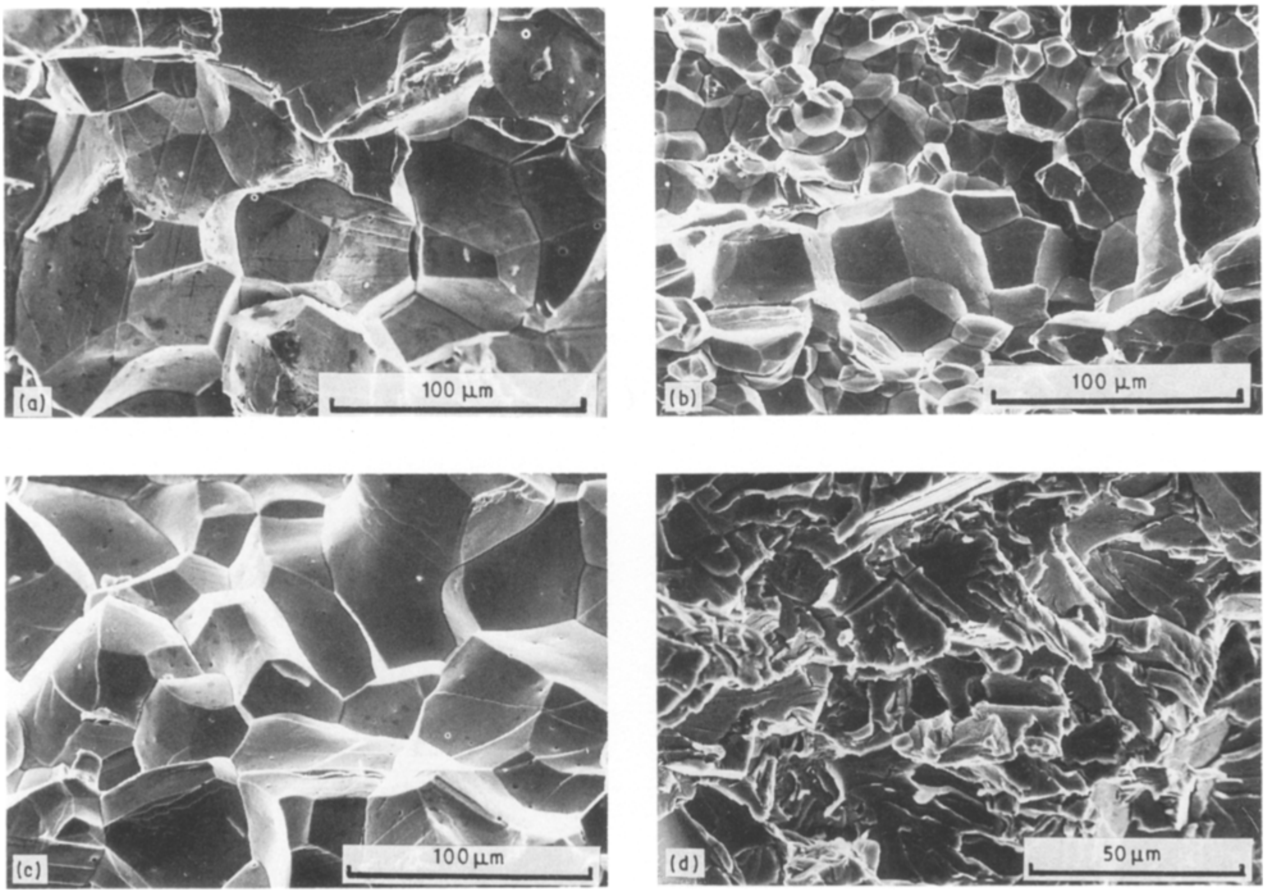

Figure 8 Scanning electron micrographs on fracture surfaces of irradiated specimens tested at $-196\,^{\circ}C$: (a) Cu-doped alloy (Heat I); (b) P-doped alloy (Heat II); (c) P–Cu-doped alloy (Heat III); (d) P–C-doped alloy (Heat IV).

decrease in the ductility of the Cu-doped alloy (I). The alloys without and with C exhibited intergranular and transgranular fracture, respectively.

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References
1. L. E. STEELE, L. M. DAVIES, T. INGHAM and M. BRUMOVSKY, ASTM STP 870 (American Society for Testing and Materials, Philadelphia, PA, 1985) p. 863.
2. J. R. HAWTHORNE, ASTM STP 782 (American Society for Testing and Materials, Philadelphia, PA, 1982) p. 375.
3. S. WOOD, R. G. LOTT, J. A. SPITZNAGEL, S. S. BRENNER and M. K. MILLER, *Trans. Amer. Nucl. Soc.* **44** (1983) 228.
4. L. E. REHN and R. P. OKAMOTO, in "Phase Transformations During Irradiation", edited by F. V. Nolfi Jr (Elsevier, New York, 1983) p. 247.
5. C. L. BRIANT and S. K. BANERJI, *Int. Met. Rev.* **4** (1978) 163.
6. J. M. BAIK, J. KAMEDA and O. BUCK, ASTM STP 888 (1986) p. 92.
7. J. KAMEDA and A. J. BEVOLO, *Acta Metall.*, submitted.
8. X. MAO, T. SHOJI and H. TAKAHASHI, *J. Test. Eval.* **15** (1987) 30.
9. X. MAO and H. TAKAHASHI, *J. Nucl. Mater.* **150** (1987) 42.
10. H. KIMURA, H. MATSUI, A. KIMURA, T. KIMURA and K. OGURI, in "Hydrogen Effects in Metals", edited by I. M. Bernstein and A. W. Thompson (Metallurgical Society of AIME, Warrendale, PA, 1981) p. 191.
11. H. WAGENBLAST and A. C. DAMASK, *J. Phys. Chem. Solids* **23** (1962) 211.
12. K. ABIKO, S. SUZUKI and H. KIMURA, *Trans. Jpn Inst. Metals* **23** (1982) 43.

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