9th INTERNAT. CONGRESS ON ELECTRON MICROSCOPY, TORONTO, AUG 1-9, 1978

ABSTRACT FORM CONF-780818-6

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DAMAGE STRUCTURE IN ZIRCONIUM ALLOYS IRRADIATED AT 573 TO 923°K. R.W. Gilbert[1] & K. Farrell[2]

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The microstructures of annealed zirconium, Zircaloy-2 and Zr-2.5 wt% Nb alloy and of Zr-2.5 Nb containing α' were studied after neutron irradiation to fluences ≈1 x 10^{25} $n{\cdot}m^{-2}$ in the temperature range 573 to 923 K. The principal form of damage was dislocation loops which increased in size and decreased in density with increasing temperature and which did not exist above 773 K. The Burgers vector of the loops was consistent with a/3 $\langle 11\bar{2}0\rangle$. Half or more of the loops were of vacancy type. No dislocation networks or voids were seen. It is argued that the bias of loops for self-interstitial atoms in α-zirconium is very weak, permitting competitive vacancy and interstitial loops, preventing growth of loops into gross dislocation structure, and depressing the vacancy super-saturation so that voids cannot arise.

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Received 21 Feb '78 - KF.

D AGE STRUCTURE IN ZIRCONIUM ALLOYS NEUTRON IRRADIATED AT 573 TO 923 K

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INTRODUCTION. Zirconium alloys are resistant to void formation[1] and associated swelling, but are subject to radiation-enhanced creep[2], radiation growth[3] and radiation hardening[4], all of which are dependent on the damage microstructures. Information on the microstructures will aid in understanding these phenomena. We have examined specimens of zirconium, Zircaloy-2, and Zr-2.5 wt% Nb irradiated at each of 573,673,773, and 923 K to fluences of 1.3, 1.2, 1.0, and 0.7 x 10^{25} n.m^{-2}, respectively, in the Oak Ridge Reactor. Foils were electropolished using a 4:1 ethyl alcohol:perchloric acid solution at 240 K and examined at 100 kV in a Phillips EM300G electron microscope using a double axis tilt stage.

OBSERVATIONS. Annealed Crystal Bar Zirconium. Dislocation loops were present after irradiation at 573 and 673 K (Fig.1,2). The median diameter increased with irradiation temperature from 18.6 to 50 nm and corresponding loop concentrations decreased from 5 x 10^{21} to 5 x 10^{20} m^{-3} respectively. Both interstitial and vacancy loops were observed, and in specimens irradiated at 673 K vacancy loops predominated. At 773 and 923 K no loops were evident. A few, $\approx 10^{16}$ m^{-3}, cavities were found in some specimens, often on grain boundaries or at inclusions.

Annealed Zircaloy-2. Specimens irradiated at 573 and 673 K contained dislocation loops with median diameters and densities of 6.5 nm, 1.9 x 10^{22} m^{-3} and 60 nm, 2.8 x 10^{20} m^{-3}, respectively,(Fig.3,4). At 673 K, vacancy loops predominated over interstitial loops. A few cavities were found.

Zr-2.5 wt% Nb, Slow Cooled from 1 h at 1073 K. This structure consisted of large grains of α-Zr containing islands of β-Zr. After irradiation at 573 and 673 K, the α-grains were filled with dislocation loops,(Fig.5,6), median sizes 7 nm and 37 nm respectively. A fine precipitate of β-Nb was also present. At 773 K dislocation loops were absent and the β-Nb precipitates were in the form of large needles. At 923 K the structure consisted of large equiaxed α-grains containing islands of β-Zr and no irradiation damage contrast was evident. A few cavities were observed.

Zr-2.5 wt% Nb, Water Quenched from 1273 K and Aged 24 h at 773 K. This structure was entirely α' martensite. Aging for 24 h at 773 K had produced a high concentration of β-Nb precipitates. "Black spot" damage was seen in specimens irradiated at 573 and 673 K, the loops being difficult to distinguish from the precipitate. No radiation-induced loops were seen in specimens irradiated at 773 or 923 K.

Zr-2.5 wt% Nb, Water Quenched from 1123 K and Aged 24 h at 773 K. This structure was composed of a matrix of α' martensite containing islands of α-Zr. Irradiation at 573 and 673 K produced dislocation loops in both phases. The loops were clearly revealed in the α-phase, while in the α' martensite they were difficult to resolve due to their small size and the presence of β-Nb precipitate. The loop size increased with increased irradiation temperature. Loops were not found in specimens irradiated at 773 and 923 K.

DISCUSSION. Neutron irradiation at 573 and 673 K produced both vacancy and interstitial loops that increased in size with increasing irradiation temperature. Few dislocation lines emerged and, at 773 K, there were sparse tangles, implying that development of gross dislocation structures from growth and climb of loops is stunted. The presence of fewer larger loops at 673 K shows a decline in the number of point defects that survive recombination or absorption. Thus, the number of point defects involved decreases with temperature toward zero at 773 K, about 0.42 of the extrapolated melting temperature of α-Zr. This is significantly lower than the 0.5 T_m upper limit observed for survival of damage in fcc metals. The presence of a mixture of vacancy and interstitial loops which progressively increase in size and decrease in density with increasing irradiation temperature shows that there is a weak bias for interstitials and thus no excess of vacancies to fuel void formation. The few cavities are characteristic of gas bubbles, probably helium generated from (n,α) reactions. We conclude that the damage structure in neutron irradiated zirconium alloys is determined and controlled by a very weak bias of loops for interstitials.

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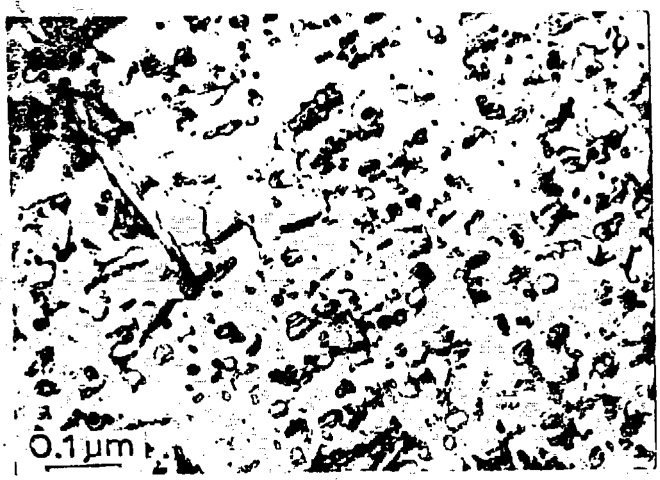

FIGURE 1 573 K

FIGURE 2 673 K

Annealed Crystal Bar Zirconium

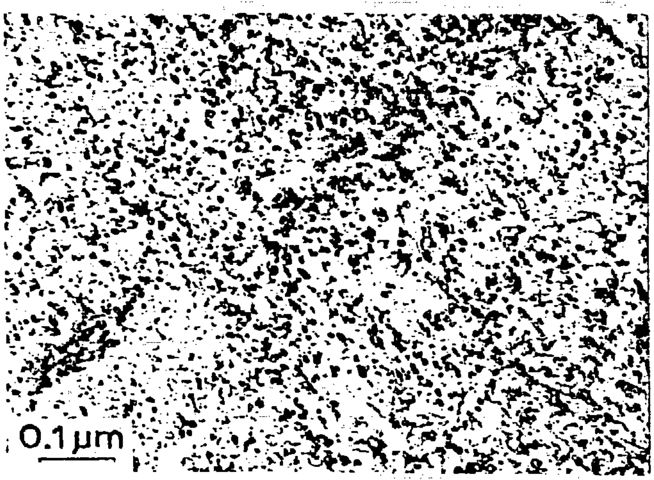

FIGURE 3 573 K

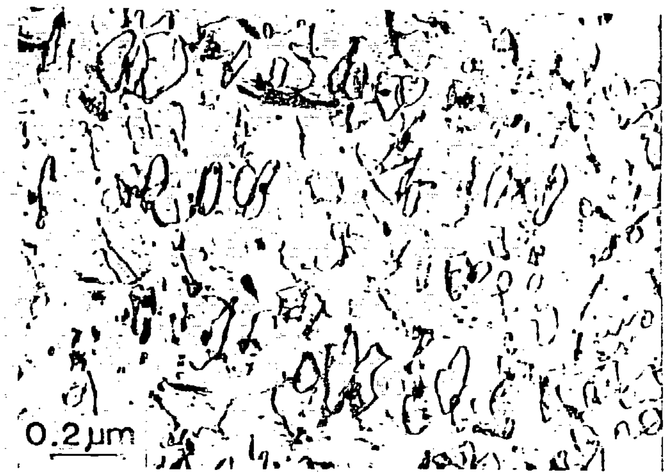

FIGURE 4 673 K

Annealed Zircaloy-2

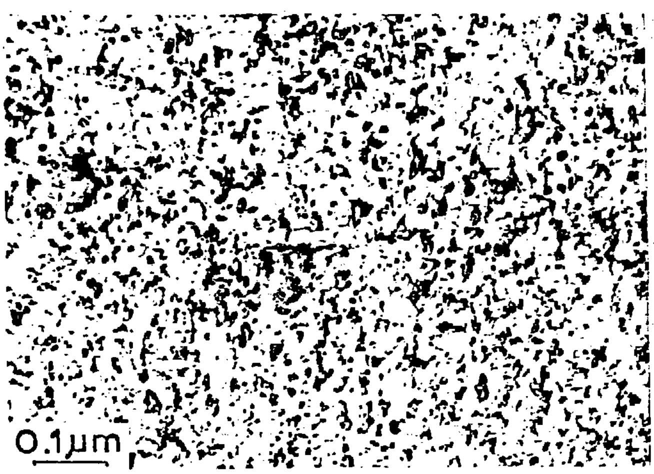

FIGURE 5 573 K

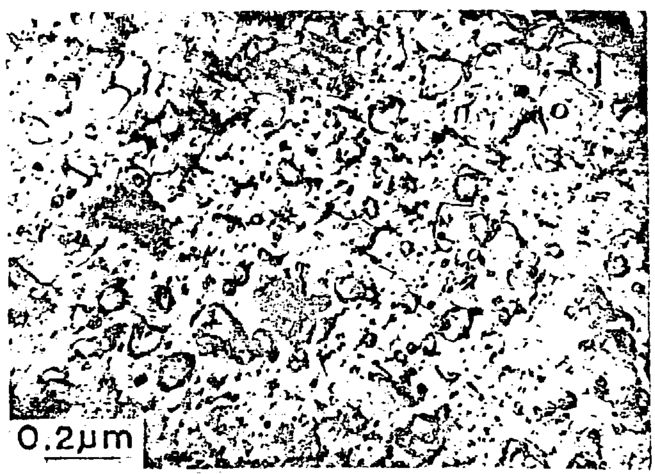

FIGURE 6 673 K

Zr-2.5 wt% Nb, α-Phase

FIGURES 1 TO 6: Dislocation Loop Structures in Zirconium Alloys Irradiated in the Oak Ridge Reactor at 573 K and 673 K to Fluences of 1.3 and 1.2 x 10^{25} $n \cdot m^{-2}$ respectively.