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MICROSTRUCTURE AND TENSILE PROPERTIES OF HEAVILY IRRADIATED 1100-0 ALUMINUM

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SUMMARY

Microstructural changes and the resulting tensile properties of commercial purity (1100 grade) aluminum were measured after neutron irradiations up to 1.4×10^{27} n/m^2 (E > 0.1 MeV) and 2.3×10^{27} n/m^2 (E < 0.025 eV) at ~328 K (0.35 T_m) corresponding to displacement levels of 200 dpa and the generation of 5.5 wt % Si. The material displayed up to about 8% swelling. Microstructural features included dislocation tangles, a precipitate of elemental silicon, and heterogeneously distributed voids coated with silicon. Alongside grain boundaries were sheets of large voids. These microstructural features are shown to be responsible for increasing the 0.2% flow stress and the UTS at 323 K by factors of 5 and 3 to values of 220 and 250 MPa, respectively, and by factors of 6.5 and 3.5 to about 190 MPa at 423 K (0.45 T_m). Elongation was reduced at 323 K from 32% to a plateau at 12% in the fluence range 4×10^{25} n/m^2 to 7×10^{26} n/m^2 (> 0.1 MeV), followed by a sharp fall to 4% at the highest fluences, the latter concomitant with a change to intergranular-type fracture. In the 423 K tests the elongation declined from 56% to 4% at the highest fluences where intergranular-type fracture prevailed. Most of this loss in ductility is accountable in terms of dispersion hardening through changes in strain hardening parameters. It is suggested that the apparent intergranular fractures are caused by tearing of sheets of voids adjacent to grain boundaries.

KEY WORDS: Commercial aluminum, neutron irradiation, high fluence, microstructure, voids, silicon precipitate, tensile tests, strengthening, ductility loss, fracture.

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MICROSTRUCTURE AND TENSILE PROPERTIES OF HEAVILY IRRADIATED 1100-0 ALUMINUM*

K. Farrell and A. E. Richt†

INTRODUCTION

The 1100-0 aluminum alloy is not really an alloy in the strict sense; it is simply commercial purity aluminum consisting of 99+% Al, the balance being tramp elements, mostly silicon and iron. The "O" temper designates a fully annealed condition. Since 1100 Al has good corrosion resistance in flowing water, has good weldability, good ductility and low residual radioactivity, it finds applications in some water-cooled reactors where strength is not a requirement.

Radiation-induced microstructural changes in 1100 Al have been shown to involve the formation of dislocation loops[1] at fluences below about 10^{24} n/m^2, and the development of dislocation tangles, voids, and a fine precipitate of transmutation-produced silicon[2] at higher fluences.[1–6] This silicon is created by the reaction, $^{27}Al(n,\gamma) \rightarrow {}^{28}Al$ with thermal neutrons, followed by β decay to ^{28}Si. The cross section for the reaction is 2.3×10^{-29}m^2 for $E < 0.025$ eV. Thus, a thermal fluence of only 1×10^{25} n/m^2 produces 2.3×10^{-4} atomic fraction silicon. Since the solid solubility[7] of silicon in aluminum at a typical irradiation temperature of 328 K is less than 10^{-4}, and since point defects are mobile at this temperature (0.35 T_m) under displacive irradiation, the silicon

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forms precipitate particles. These particles, together with the voids and the dislocation structure developed by displacement damage from fast neutrons, have been shown to be responsible for radiation-induced increases in hardness,[6] tensile strength,[8] and creep resistance[9] in 1100-0 aluminum. However, the data on tensile properties of neutron-irradiated 1100-0 alloy are rather meager and further information was considered to be desirable, especially since such a simple system could provide a reference data base against which the radiation response of other aluminum alloys could be compared. Accordingly, specimens of 1100-0 alloy were included in the Oak Ridge National Laboratory aluminum surveillance program. This paper describes the microstructural changes and tensile properties of these specimens.

EXPERIMENTAL CONDITIONS

The major impurities in the unirradiated 1100 aluminum were 0.21 wt % Cu, 0.44 Fe, and 0.10 Si, with traces of Cr, Mn, Ni, and Ti. Button-headed tensile specimens of gage length 28.6 mm and gage diameter 3.2 mm were machined from cold swaged 6.3-mm-dia rods. These specimens were annealed for 0.25 h at 616 K, followed by air cooling, resulting in a recrystallized grain size of 8 μm. The annealed specimens were irradiated in contact with flowing cooling water at 318 to 328 K in the peripheral target area of the High Flux Isotope Reactor (HFIR) for times up to 2.8 y and maximum fluences of $1.4 \times 10^{27}/m^2$ of fast neutrons with $E > 0.1$ MeV, and $2.3 \times 10^{27}/m^2$ of thermal neutrons with $E < 0.025$ eV. The corresponding displacements per atom, calculated using the appropriate cross section for the HFIR spectrum and an effective

threshold displacement energy of 25 eV, is 200. The estimated maximum silicon concentration generated from (n,γ) reactions is 5.3 at. % (5.5 wt %). Other major transmutation products are estimated to be 3.8×10^{-4} at. fraction hydrogen from Al(n,p) reactions and 6.4×10^{-5} helium from Al(n,α) reactions. These irradiation levels are considerably higher than any previously reported for 1100 grade aluminum.

After irradiation, an oxide scale that had formed on the specimens after prolonged exposure in the cooling water was removed by etching in Alcoa bright-dip solution, and the densities of some of the specimens were measured by Archimedes' displacement. The specimens were then tensile tested to failure at two test temperatures, 323 and 423 K (0.35 and 0.45 T_m) and a strain rate of 7.4×10^{-5}/s. Following testing, thin foils for transmission electron microscopy examination were prepared from the gage regions and from the unstrained heads of a few of the specimens.

RESULTS

Dealing first with the irradiation-induced microstructural changes, it was found that the nature of the microstructural damage was essentially the same as that reported earlier[1–6] for 1100-0 aluminum, to wit dislocation tangles, heterogeneously distributed voids, and a precipitate of transmutation-produced silicon. But the voids were larger at these higher fluences and there was up to ten times as much transmutation-produced silicon as in the earlier studies.

Some typical microstructures are shown in Fig. 1. At all fluences the voids tended to occur in patches and to be concentrated around large, incoherent

inclusions and, most obviously, to congregate in layers parallel to and on each side of grain boundaries [Figs. 1(a) and (b)]. Those adjacent to the grain boundaries were also the larger voids, and a few of these were seen to be touching and coalescing with a neighbor. The concentration of voids was independent of neutron fluence over the range of fluences examined, but the average size of the voids increased with fluence. At the highest fluence the concentration of voids within the grains was about $1.4 \times 10^{20}/m^3$ with a mean diameter of 80 nm. Near the grain boundaries the concentration of voids was in the region of $2.8 \times 10^{20}/m^3$ with a mean diameter of 96 nm. Large void halos, or spherical shells of voids, about 4.1 μm dia, were also present, centered around some of the inclusion particles. These halos have been noted previously in aluminum[3] and the measured diameter is consistent with the expected range of recoiled α particles from burnup of ^{10}B. Aluminum normally contains traces of boron, typically $< 4 \times 10^{-6}$ at. fraction, but it is concentrated in a few inclusion particles. The helium resulting from burnup of the boron comes to rest at the sites of the void halos where its concentration greatly exceeds the average helium level of the matrix. Density measurements indicated that total swelling at the highest fluences was about 7.5 to 10%. Of this, the silicon precipitate with a density of 2.3293×10^3 kg/m^3 versus 2.7101×10^3 kg/m^3 for aluminum is estimated to contribute a little less than 1%. Density changes will be described in full and compared with other aluminum alloys in a later paper.

A very interesting feature of the voids is that they are rounded rather than facetted and they are coated with a layer of silicon, especially noticeable at the higher fluences where the coated voids can be extracted intact on carbon replicas. A more detailed description of these coated voids and analyses of the coatings has been published.[10] The balance of the transmutation-produced

silicon is manifest as small, rounded particles uniformly distributed through the aluminum grains, and as much larger, slab-like particles on grain boundaries. Those silicon particles on grain boundaries are not continuous and cover only a small fraction of the total grain boundary area; indeed, for the most part the grain boundaries and the adjacent regions about 100 nm wide out to the layers of voids are relatively free of damage structure. At the highest fluence, the concentration of silicon particles within the grains was $1.6 \times 10^{21}/m^3$, with an average diameter of 25 nm. The dislocation structure [Fig. 1(d)] was uniform and rather dense and, because of contrast interference from the silicon precipitate, was difficult to measure but was in the vicinity of 1.3×10^{14} m/m^3 at the highest fluence. No differences were seen between the dislocations in the unstrained heads of the tensile specimens and those in the strained gage length, neither in nature nor concentration.

Examples of changes in the shapes of tensile curves with fluence and with test temperature are given in Fig. 2, where it is clearly evident that irradiation causes marked increases in strength and decreases in elongation. The fluence dependence of these changes is plotted in Figs. 3 and 4 for the two test temperatures, respectively. The fast fluence is used to label the curves in Fig. 2 and as the abscissa in Figs. 3 and 4, but it must be recognized that the fast fluence alone does not determine the changes in mechanical properties; the thermal fluence makes a major contribution via the precipitate of transmutation-produced silicon. Thermal fluences are obtained by multiplying the fast fluence by about 1.7. The levels of transmutation-produced silicon are indicated by the additional scales on Figs. 3 and 4.

In the 323 K tests, irradiation raises the 0.2% flow stress and the UTS by factors of 5 and 3 to values of about 220 and 250 MPa, respectively, and

by factors of 6.5 and 3.5 to about 190 MPa in the 423 K tests, the latter factors being larger because of temperature-dependent softening of the unirradiated material. Ductility, as measured by elongation, was reduced in the 323 K tests from a total elongation of 32% in the unirradiated material to a plateau of about 12% for fast fluences in the range 4×10^{25} n/m^2 to about 7×10^{26} n/m^2, above which fluence the elongation fell sharply to about 4%. Corresponding to this drop from 12 to 4% the fracture mode changed from local, ductile-type cup and cone failure to a facetted, seemingly intergranular, separation. At fluences above about 3×10^{25} n/m^2 the uniform elongation was reduced from 17 to 4% where it remained despite further increases in fluence. In the 423 K tests, total elongation progressively declined from 56 to 4% for fluences up to 4×10^{26} n/m^2 then remained constant at 4% with higher fluence; uniform elongation fell rapidly from 14 to 2% at fluences below 3×10^{25} n/m^2 and remained at 2% for higher fluences. Specimens with these low ductilities demonstrated reduced necking and exhibited facetted fracture surfaces.

DISCUSSION AND CONCLUSIONS

The highly heterogeneous spatial distribution of the voids in this material deserves comment since they represent a gross example of non-homogeneous void nucleation, and it will be argued later that the heterogeneous voids impair the mechanical behavior. The void distribution can be explained readily in terms of the distribution of helium and the effects of major point defect sinks. Helium, or α particles, is known to be a strong void nucleant[11–13] whereas many alloying elements or substitutional-type impurities tend to suppress void formation, possibly by promoting point defect recombination.[14] The apparent segregation of voids into patches or bands within the grains is believed to be initiated by clusters

of α particles recoiled from ^{10}B particles. The capture cross section for the $^{10}B(n,\alpha)$ reaction with thermal neutrons is very large, 2.5×10^{-25} m^2, and much of this heterogeneously-generated helium would be created early in the irradiation history and in localized regions would swamp the effects of the homogeneously-generated helium from the fast $^{27}Al(n,\alpha)$ reactions, and hence would control and encourage void nucleation in those regions. On the other hand, the higher concentrations and larger sizes of voids near grain boundaries and incoherent inclusions are thought to be related more to the special nature of these sites than to the helium distribution, although it was frequently noted that when a grain boundary intercepted an obvious halo of voids the void concentration near the grain boundary was higher than average for the near grain boundary regions. The regions adjacent to incoherent interfaces such as grain boundaries,[3,6,8,13] twins,[15] and inclusions[3] are known to be favored sites for void growth but there is no widely accepted reason for the phenomenon. Tentative explanations involve, on the one hand, the efficiencies of the interfaces as point defect sinks,[3] and on the other their effects as dislocation barriers which enhance radiation-induced dislocation levels in their vicinity.[15] Impurities are probably not involved to any large extent because enhanced void growth is observed in these regions in pure metals.[3,16]

The heterogeneity of the void distribution is further emphasized when swelling from the voids is considered. In one of the highest fluence specimens that exhibited a 7.7% decrease in density, it was estimated from electron microscopy observations that the local swelling in a 0.5-μm-wide band embracing the grain boundary regions was 15% whereas that within the grains was no more than 3.7%. The average swelling from voids alone was 5.1%, of which the near

grain boundary voids contributed approximately one-third. The silicon precipitate donated about 1% swelling for a total of 6.1%. While not in precise agreement with the density change, the microscopy measurements are believed to reflect accurately the extent and nature of the non-uniform swelling.

The transmutation-produced silicon, although created homogeneously, is not distributed uniformly in the microstructure, and the bulk of it is not in the small silicon precipitates. In fact, most of it is in the grain boundaries, and a large fraction is associated with the voids. The silicon coatings on the voids were usually about 4 nm thick but in some cases were about 11 nm. Taking a typical coating thickness of 5 nm and adding this to the measured void sizes, we estimate that 0.023 vol fraction silicon of a total vol fraction of 0.064 was on the voids. The precipitate of small silicon particles consumed only 0.013 vol fraction. The balance of 0.028 vol fraction has presumably been used to form the large grain boundary precipitates. Hence, 20% of the silicon is in small precipitates within the grains, 36% is on voids, and 44% is in grain boundary precipitates.

As with the previously mentioned hardness[6] and tensile data[8] on 1100-0 Al it can be demonstrated that the measured radiation-induced strengthening can be accounted for in terms of the silicon precipitate and the dislocation structure. In essence, the irradiation converts the originally annealed, soft single phase alloy into a cold-worked, dispersion-hardened Al-Si alloy of increasing silicon level. At the highest fluence the yield stress, or 0.2% flow stress, at 323 K is raised from 45 to 223 MPa, an increase of 177 MPa. In terms of strengthening theories this increase, $\Delta\sigma_y$, can be derived from $\Delta\sigma_y = \Delta\sigma_{Si} + \Delta\sigma_{dis} + \Delta\sigma_{cv}$ or, in some cases, from $\Delta\sigma_y = [(\Delta\sigma_{Si})^2 + (\Delta\sigma_{dis})^2 + (\Delta\sigma_{cv})^2]^{\frac{1}{2}}$

where $\Delta\sigma_{Si}$ is the strengthening contribution from the silicon dispersion, given[17] by $\Delta\sigma_{Si} = \frac{1.04}{\pi} Gb(Cd)^{\frac{1}{2}} \ln\left(\frac{d}{4b}\right)$, similarly for coated voids, $\Delta\sigma_{cv}$. The increase in strength from the dislocation structure, $\Delta\sigma_{dis}$, is given[18] by $\Delta\sigma_{dis} = Gb\lambda^{\frac{1}{2}}$. Here G is the shear modulus for aluminum (2.648×10^4 MPa), b is the Burgers vector (0.286 nm), C is the concentration of particles of diameter d, and λ is the dislocation density. Substituting the appropriate measured values and neglecting the near-grain boundary voids as being unamenable to treatment (the $\Delta\sigma$ equations are based on uniform distributions of particles), we find that $\Delta\sigma_{Si}$ = 49 MPa, $\Delta\sigma_{dis}$ = 86 MPa, and $\Delta\sigma_{cv}$ = 39 MPa. A simple summation gives $\Delta\sigma_y$ = 174 MPa, an agreement with the measured value that is probably more fortuitous than real. The second summation, which is really an indirect summation of the defect concentrations rather than their stress increments, and which should be the most appropriate one, gives 106 MPa. A realistic estimate of $\Delta\sigma_y$ probably lies between these two summations. Considering the poor spatial distribution of the microstructural defects, and the consequent probable errors in C and λ the correspondence between measured and calculated $\Delta\sigma_y$ is reasonably good.

A consequence of second-phase hardening (and of strain hardening) is that the capacity for plastic deformation — the ductility — is reduced. The work hardening rate, $\frac{\delta\sigma}{\delta\varepsilon}$, is increased and a given stress is reached at a lower level of strain. Concurrently the material becomes plastically unstable earlier. If the tensile curve can be described by a power law of the form $\sigma = \kappa\varepsilon^n$, as with many fcc metals including aluminum, the work-hardening exponent, n, is equal to the strain at which plastic instability, or necking, occurs. A decrease in n indicates a loss in uniform elongation, or vice versa. It is clear from Figs. 3 and 4 that there is a significant loss in uniform elongation, and the corresponding uniform strains are found to be equal to rough measurements of n

made from the tensile curves, with n falling from 0.19 to about 0.05 at 323 K and to about 0.015 at 423 K. The greatest loss in uniform elongation occurs fairly early in the irradiation history, presumably at the point where the final defect concentrations are established, and then saturates at a low level implying that growth of the defects thereafter has relatively little effect on the uniform elongation even though they continue to raise the strength.

The loss in uniform elongation does not just pull down the total elongation by an equal amount because, of course, the effects of the second phases on ductility continue to be felt even after the onset of necking, and the necking strain is reduced also. If the deformation mode and the fracture mode remain unchanged, the loss in total elongation should be proportional to the loss in uniform elongation. This seems to be so at 323 K except at the highest fluences where an apparent change in fracture mode to intergranular failure was associated with a greater loss in total ductility. At 423 K the total elongation at the lower fluences was greater than expected under this line of reasoning, possibly because some accelerated recovery of strain hardening may have occurred in the neck in the dispersion-hardened material at this higher temperature. This benefit, however, was cancelled at the higher fluences where a change in fracture mode to one involving flat facets considerably reduced the total elongation; this occurred at a lower fluence than for the 323 K tests.

We were not able to make a detailed fractographic study of the high fluence specimens with facetted fracture surfaces because a thick, dark crust formed on them before we could remove them from the hot cells. When freshly broken, the surfaces had a clean, apparently intergranular appearance as observed under a low-power remote telescope. But by the time they were cut from the specimens

and packaged for out-of-cell examination they had presumably reacted with the humid atmosphere in the hot cell and developed the thick, impervious film. Cup and cone fractures did likewise. Saw cuts and the fractures of unirradiated specimens retained their metallic luster. We assume that the reaction product involved silicon, and the severity of the coverage was caused by a high incidence of silicon particles on the fracture surfaces. This peculiar behavior might be compatible with exposure of, and reaction with, the large silicon precipitates on grain boundaries. However, similar precipitates in heavily irradiated 6061 aluminum alloy[19] do not induce intergranular fracture. Therefore, we suspect, but cannot prove, that the facetted fractures were not true grain boundary failures but resulted from joining up of the near-grain boundary voids, the three-dimensional equivalent of "tearing along the dotted lines." The layers of voids near the grain boundaries are prime candidates for such a failure mechanism. At the very least, their presence effectively reduces the cross-sectional area of the tensile specimens. For the void concentrations and sizes that we quoted earlier, the area fraction occupied by a sheet of near-grain boundary voids is 0.15. And the material between these voids is the very soft, albeit ductile, defect-free "pure" aluminum. It seems most unlikely that such a weak and perforated material could continue to tolerate the progressively higher stresses that can be sustained by the dispersion-hardened matrix.

If this mechanism of near-grain boundary separation really does prevail, we can expect it to limit both the ductility and the loads that can be sustained

for fluences beyond those explored in this work. Interestingly, we noticed that when electron microscopy specimens containing the near-grain boundary voids were handled several times in and out of specimen holders they developed long cracks joining the large cavities. Although this is certainly not evidence that the same phenomenon occurred in the bulk specimens during testing, it does confirm that the sheets of near-grain boundary voids are a weak link in the system.

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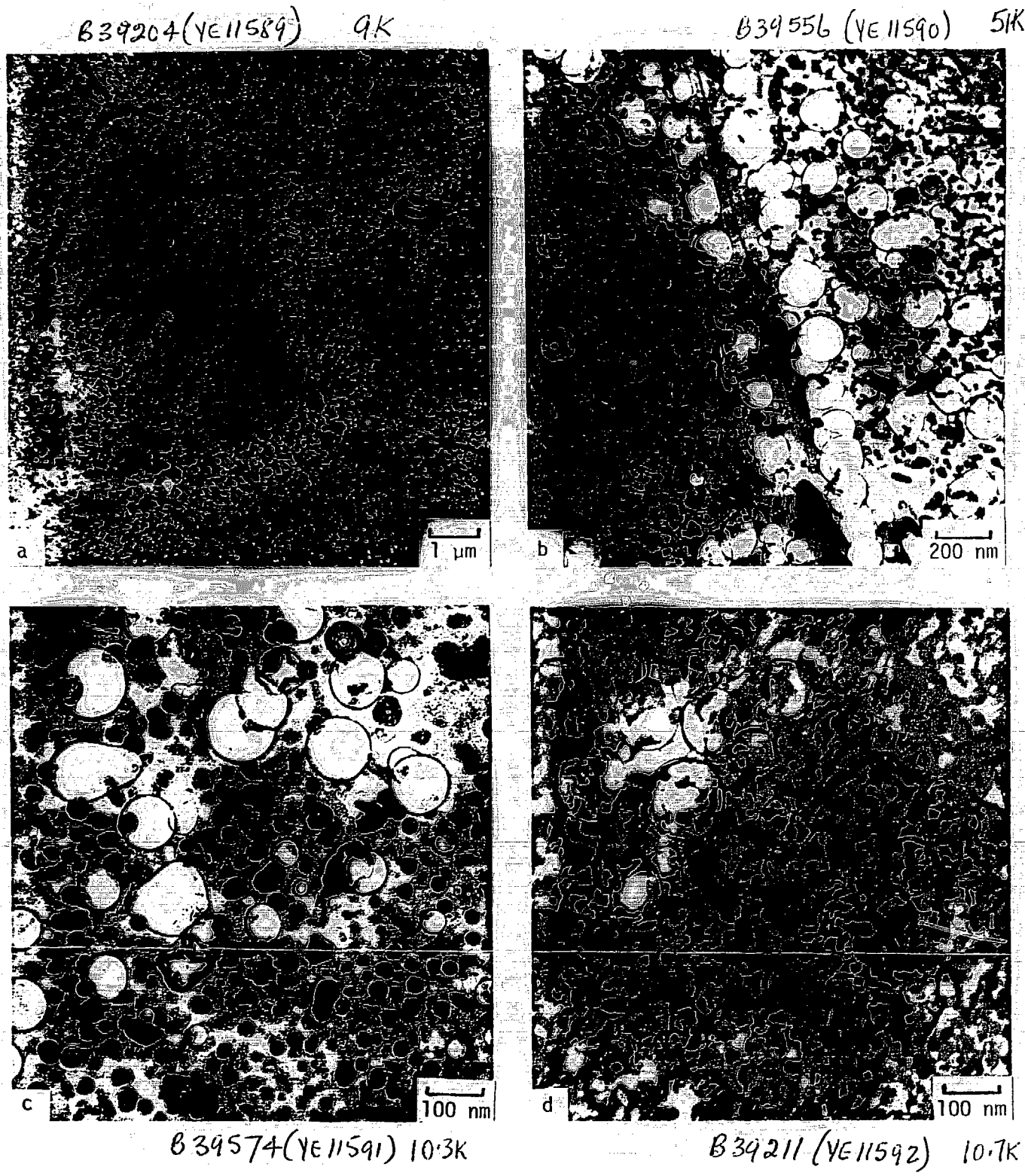

Fig. 1. As-Irradiated Microstructural Features. (a) and (b) Large voids near grain boundaries, (c) voids and small silicon precipitates within grains, and (d) dislocation structure.

Fig. 2. Changes in Shapes of Tensile Curves with Increasing Fluence at the Two Test Temperatures. Fast fluences are shown. The ratio of thermal-to-fast fluence is about 1.7.

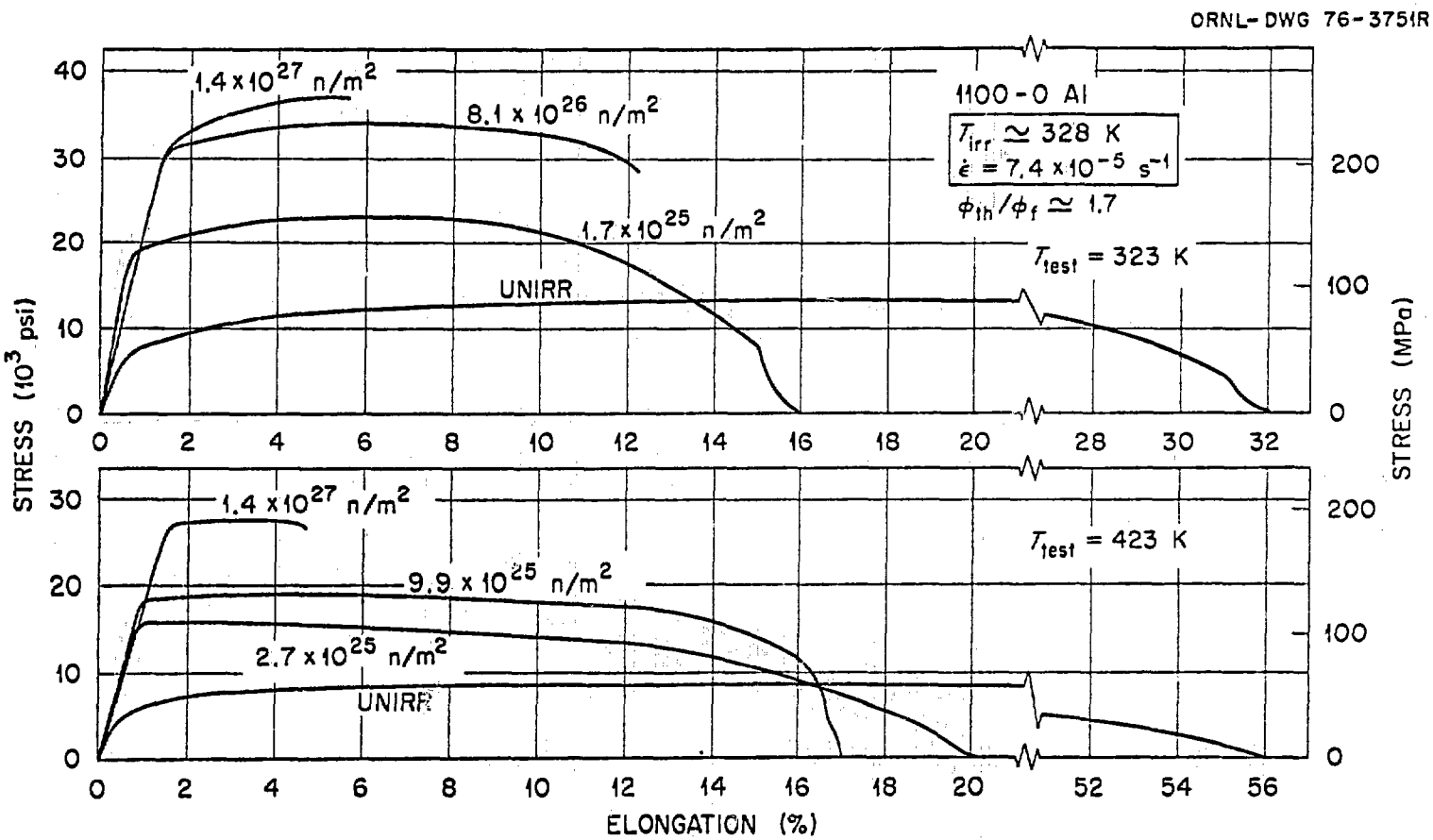

Fig. 3. Fluence Dependence of Tensile Properties at 323 K.

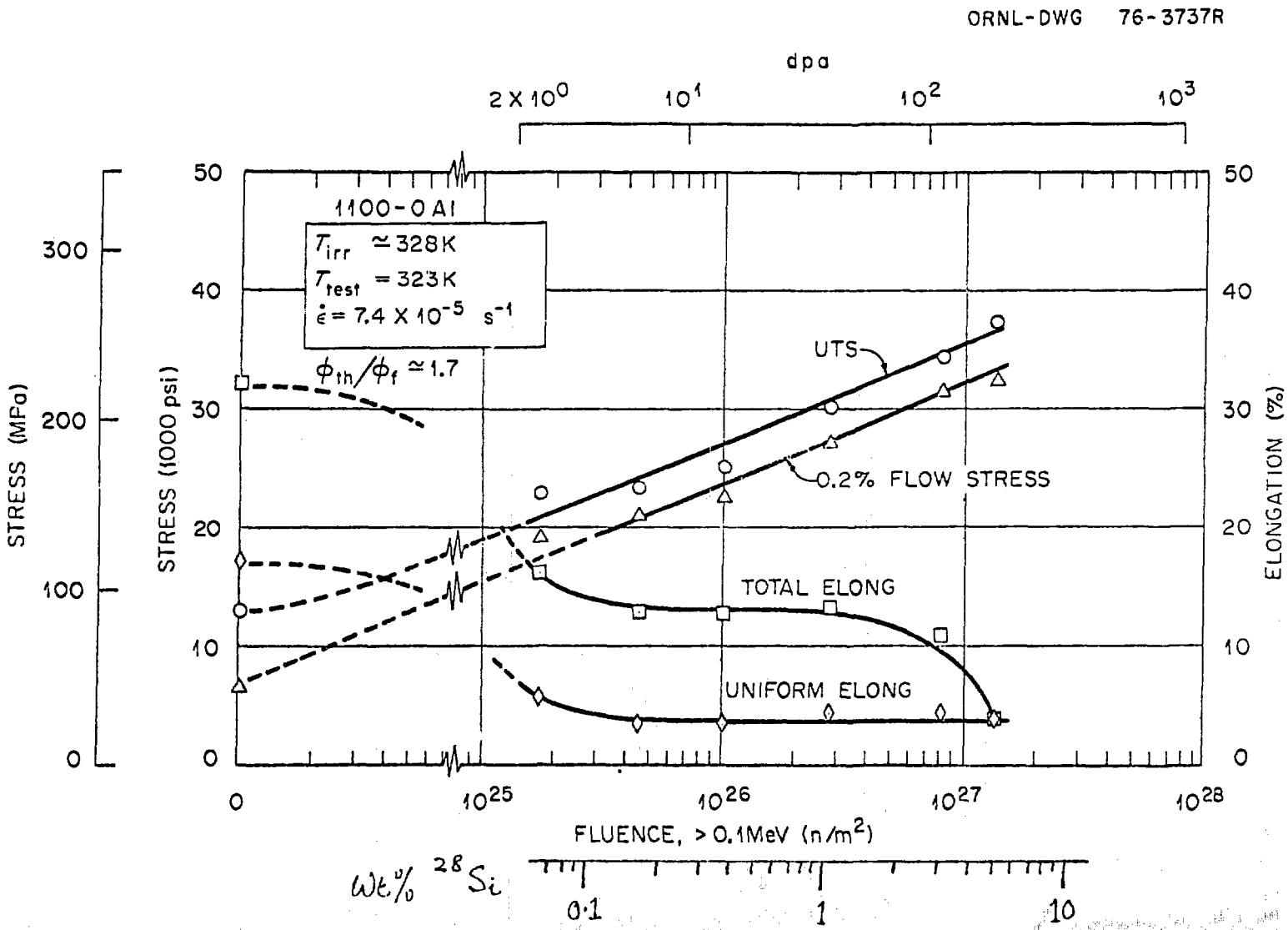
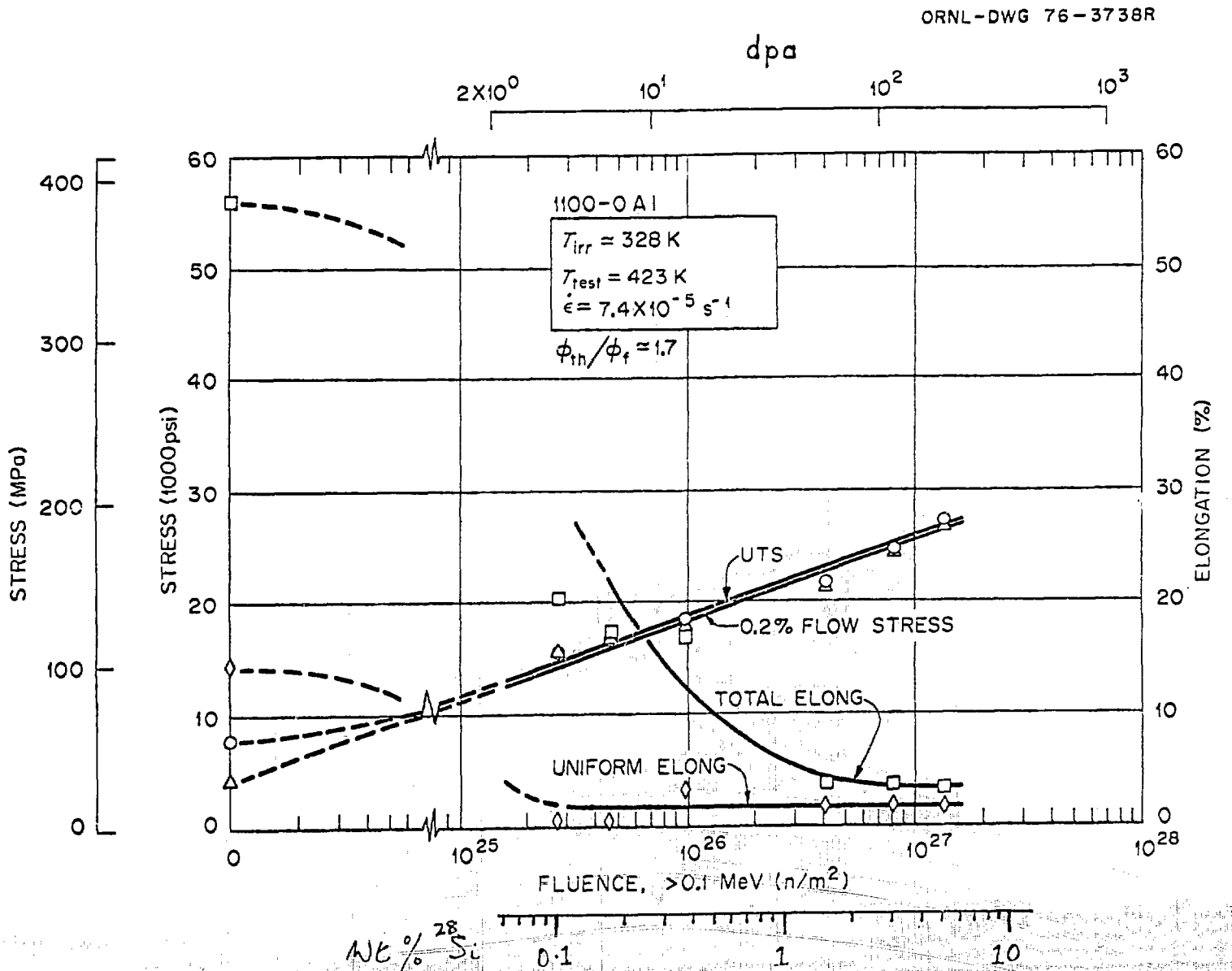
Fig. 4. Fluence Dependence of Tensile Properties at 423 K.