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MICROSTRUCTURE AND TENSILE PROPERTIES OF HEAVILY IRRADIATED 5052-0 ALUMINUM ALLOY*

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ABSTRACT

During neutron irradiation of an aluminum 2.2% magnesium solid solution alloy in the High Flux Isotope Reactor to fast and thermal fluences > 10^{27} neutrons (n)/m^2 at 328K (0.35 T_m) about seven percent insoluble, transmutant silicon was produced. Some of this silicon reacted with the dissolved magnesium to form a fine precipitate of Mg_2Si. A tight dislocation structure was also created. The alloy showed good resistance to cavity formation. Tension tests at 323, 373, and 423 K (0.35, 0.40, and 0.45 T_m) showed pronounced irradiation-induced strengthening and an associated marked loss in ductility. These changes were greater than in magnesium-free aluminum and in alloys containing preexisting, thermally-aged Mg_2Si precipitate. Increasing the thermal-to-fast flux ratio from 1.7 to 2.1 caused further strengthening beyond that expected from a simple increase in silicon level.

Key Words: Aluminum-magnesium alloy, neutron irradiation, microstructure, cavities, precipitates, tension tests, strengthening, ductility loss.

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INTRODUCTION

Many aluminum alloys depend upon thermally-induced precipitates to provide satisfactory mechanical strength for service applications. The 5000-Series alloys are exceptions in that they are solid solution strengthened by magnesium, one of the few elements with significant solid solubility in aluminum. The 5052 alloy consists nominally of 2.5 weight percent magnesium and 0.25 chromium. It is normally used in "O"-fully annealed, or "H"-partially strained conditions. Such alloys have good formability and resistance to corrosion in flowing water. They have found applications in some water-cooled reactors for situations where the strength requirements lie between those of the softer 1000 Series alloys and the stronger precipitation-hardened 6000 Series alloys.

PREVIOUS IRRADIATION STUDIES

Tensile tests[1] on 5052 and 5154 (3.5 Mg) alloys in O and H tempers irradiated to low fluences (1×10^{24} fast n/m^2, 1.26×10^{25} total n/m^2) at $\sim$ 340K ($\sim 0.36T_m$) showed an increase in strength and a small loss in ductility for the O temper; the H temper material showed a smaller increase in strength and an increase in ductility, suggesting some recovery of cold work during irradiation. Irradiations of the 5052-O alloy to much higher fluences [up to 3.6×10^{26} n/m^2 (fast) and 5.8×10^{26} n/m^2 (thermal)] at $\sim$ 330K caused substantial increases in creep-rupture strength at 323K and considerable loss in ductility with a propensity for intergranular failure[2]. Examination of the microstructures[2,3] revealed the presence of a fine precipitate of Mg_2Si

which was held largely responsible for the irradiation-induced increases in strength. This Mg_2Si precipitate is produced by the interaction of transmutation-produced silicon with the dissolved magnesium. The silicon is created primarily by thermal neutrons through the sequential reactions $^{27}Al(n,\gamma) \rightarrow {}^{28}Al$; $28Al \rightarrow {}^{28}Si + \beta$, for which the cross-section is high, 2.3×10^{-29} m^2 for 2200 m/s neutrons (i.e., $E \lesssim 0.025$ eV). Sufficiently high temperatures and/or the presence of atomic displacements from fast neutrons allow transport of the silicon and development of Mg_2Si precipitates. In essence, irradiation of originally-solid solution 5000 Series aluminum alloys in a reactor of mixed neutron spectrum gradually converts them to precipitation-hardened 6000-type alloys.

The present work takes these observations to higher neutron fluences and explores the effects of test temperature on the tensile properties of irradiated 5052-0 alloy.

EXPERIMENTAL CONDITIONS

The chemical composition of the commercially-produced 5052 alloy was 2.2 weight percent Mg - 0.2 Cr - < 0.3 Ni - 0.18 Fe - < 0.1 Si. Button-headed tension specimens of gauge length 28.6 mm and guage diameter 3.2 mm were machined from cold-swaged rod and were annealed for 0.5 h at 344°C (0.66 T_m) in a salt bath to give a fully-recrystallized "O" temper.

The specimens were irradiated in the peripheral target positions in the Oak Ridge High Flux Isotope Reactor in contact with the cooling water at 328K (0.35 T_m) for periods up to three and one-half years, during which they accumulated fast (E > 0.1 MeV) neutron fluences up to 1.8×10^{27} n/m^2 corresponding to 260 displacements per atom (dpa). Thermal fluences (E < 0.025 eV) were, for the most part about 1.7 times greater than the fast fluences and caused the creation of up to 6.9 atomic percent (7.15 weight percent) silicon. Neutron-generated gases were calculated to range up to 8.5×10^{-5} atomic fraction helium and 5×10^{-4} atomic fraction hydrogen.

After irradiation, the specimens were tested in tension to failure. Pieces cut from the heads and gauge sections of the broken specimens were examined by transmission electron microscopy (TEM).

MICROSTRUCTURAL CHANGES

The microstructure of the alloy before irradiation consisted of a solid-solution matrix containing a few inclusion particles. At the lowest fast fluences of 1 – 1.5×10^{25} n/m^2 (~ 2 dpa), radiation damage structure was present in the form of small, unfaulted loops very close to grain boundaries and around residual inclusions (Fig. 1). At some of the inclusions, the loops had grown into dislocation tangles. The transmutation-produced silicon level was about 5.8×10^{-2} atomic percent at this fluence, and no Mg_2Si precipitates were discernible. With increasing fluence, a generally-dispersed, closely-knit, tangled dislocation structure was developed and a finely-divided precipitate of Mg_2Si appeared (Fig. 2a). The precipitate was continuous to the grain boundaries, i.e., no obvious near-grain boundary denuded regions. The identity of the precipitate is evident from the (001) diffraction pattern showing streaking in <100> and <010> passing through the 100 and 110 reciprocal lattice planes. Such features are characteristic of thermally-produced Mg_2Si phase in aluminum alloys[3,4]. The rings of spots in the pattern are not characteristic of thermally-produced Mg_2Si and further comment on them is withheld for the discussion.

A few cavities were present after irradiation to fast fluences above about 5×10^{26} n/m^2, corresponding to about 70 dpa. These cavities were most often located in loose clusters within the grains and attached to residual inclusions; some were also noted on grain boundaries. The cavity concentration was low, about $10^{19}/m^3$, and their mean size was 25 nm for fast fluences in the vicinity of 5×10^{26} n/m^2. At the maximum fast fluence of 1.8×10^{27} n/m^2 (260 dpa) there was no significant change in the concentration of cavities but their size had increased to about 50 nm. Several cavities are shown in Fig. 2b; note that the size of the precipitate has increased, too. Reflections from Mg_2Si precipitates persisted in the diffraction patterns at these high fluences. Also, in the grain boundaries there was pronounced accumulation of precipitate in large, spongy-like discontinuous particles (Fig. 2c).

The microstructures in the strained gauge sections of the more heavily irradiated specimens were indistinguishable from those in the unstrained heads of the specimens. This is not surprising since it would be very difficult, if not impossible, to recognize slip dislocations in a structure containing preexisting, dense, radiation-induced dislocation tangles. Only if there was an unusual plastic deformation mechanism, such as dislocation channeling, would the slip dislocations be discernible. The conclusion is that the plastic deformation mechanism was normal for a precipitation-hardened material.

The thermal stability of the radiation damage structure was tested by cutting disks from specimens irradiated to fluences of about 6×10^{26} n/m^2 (fast) and 1×10^{27} n/m^2 (thermal) and annealing them for 0.5 h at temperatures of 423, 473, 523, 573, and 623 K, followed by air cooling. TEM examination revealed no change in the precipitate structure for temperatures up to 523 K, but cavities disappeared at 523 K. At 573 K there was obvious coarsening of the precipitate particles and a loss of dislocations. At 623 K there was further precipitate coarsening in the matrix and the formation of large particles on grain boundaries.

TENSILE PROPERTIES

Tension tests were conducted at a strain rate of 7.4×10^{-5}/s in air at temperatures of 323, 373, and 423 K (0.35, 0.40, and 0.45 T_m), where the radiation-produced microstructures were stable. In tests at 323 K, the unirradiated alloy displayed a well-defined yield point and continuous serrated, or jerky, flow throughout the test, as seen in the upper half of Fig. 3. The frequency of serrations was particularly high after the maximum load was exceeded and necking had commenced. Progressive irradiation eliminated both the yield point and the jerky flow, and caused pronounced strengthening and loss in ductility.

Strengthening occurred primarily through increased yield or initial flow stress. Because the flow stress increased more rapidly than the UTS, the work-hardening rate tended to decrease at the higher fluences. The fluence dependence of the tensile properties at 323 K is shown in Fig. 4. Although the fast fluence is shown on the abscissa, recognition of the contribution of thermal fluence to mechanical property changes is indicated by the addition of an appropriate scale for the generation of transmutant silicon at $\phi th/\phi_f = 1.7$. With increasing fluence the 0.2 percent flow stress was increased by a factor of 6.5 and the UTS by a factor of 2.7. Correspondingly, the ductility (expressed here as plastic elongation, the elastic strain being ignored) fell from 26 percent elongation for the unirradiated material to about 8 percent at a fast fluence of about 2×10^{26} n/m^2, above which the ductility remained seemingly independent of fluence. The loss in ductility was caused entirely by loss of uniform strain. The fracture path was transgranular by locally ductile tearing and was invariant with fluence.

At the two higher test temperatures (Figs. 5 and 6), similar patterns of fluence dependency of tensile properties prevailed, but the strengths and ductilities were less than those at 323 K. At 423 K, the 0.2 percent flow stress and UTS values were equal, and at the higher fluences necking instability occurred immediately after yield (lower part of Fig.3). Correspondingly, the total elongation fell to about 3 percent with no measurable uniform elongation. Under these conditions, fracture continued to follow a locally-ductile transgranular path. At the highest fluence a facetted surface was evident. It was not clear, though, whether the facets were intergranular. A single test performed on one of the highest fluence specimens at 423 K and a strain rate 40 times higher than those in Fig. 6 showed small increases in strength and increases in total and uniform elongations to 6.6 and 1.9 percent, respectively, with non-facetted fracture.

At all test temperatures it was found that a few specimens, which had been irradiated in sites where the thermal-to-fast flux ratio was ~ 2.1, appeared to consistently display higher strengths than the more common specimens with a thermal-to-fast flux ratio of 1.7. The data for these aberrant specimens are indicated by the dotted lines in Figs. 4–6 They are particularly apparent at 373 K (Fig. 5), where enough specimens were tested to demonstrate a pronounced upward shift in the flow stress and UTS curves.

DISCUSSION

This work confirms and extends the earlier reports[1–3] of radiation-induced strengthening and microstructural changes in 5052-0 aluminum alloy, and reveals some new features. It also provides the most comprehensive description of the effects of neutron fluence on the tensile properties of the originally 5052 alloy.

Before proceeding to a discussion of the more obvious findings of this work, attention is drawn to two microstructural aspects that might otherwise pass unnoticed, but which are considered to be valuable observations. The first is that the 5052-0 alloy has very good resistance to cavity formation and swelling. This becomes more readily apparent when the void parameters are compared with those of high purity aluminum.[7], commercial-purity 1100-grade aluminum[6,8,9] and the Mg_2Si precipitation-hardened 6061 alloy[10,11], irradiated under similar conditions. In the high-purity aluminum, cavities are created at a fast fluence as low as 1.5×10^{23} n/m^2, and at 10^{26} n/m^2 there is > 5 percent swelling. To achieve the same level of swelling in 1100 Al, a fast fluence of about 10^{27} n/m^2 is required. At this high fluence there is less than 1 percent swelling in 6061 alloy and less than 0.1 percent in the present 5052-0 alloy. This reduced swelling in the 5052 alloy is achieved through significant decreases in both cavity concentrations and cavity growth. In particular, the incubation dose for cavity formation is rather high, about 5×10^{26}n/m^2, which is more than three thousand-fold that for pure aluminum[7]. Such strong resistance to cavitation must be imparted, initially at least, by the magnesium and other minor impurities. While these remain in solution, they are assumed to act as trapping and recombination sites for vacancies and interstitial atoms, thus depressing the vacancy supersaturation, as discussed in an earlier paper.[12] As the magnesium is drawn from solution to form the Mg_2Si precipitates, trapping and recombination is presumably shifted to the precipitates whose high spatial density might provide overlapping point-defect capture zones.

Gases are known to enhance cavity formation during irradiation;[13] yet in this 5052 alloy cavity nucleation is restrained despite high levels of transmuted gas. This could, of course, be due entirely to a very low supersaturation of vacancies. Another factor could be trapping of gases by the high concentrations of dislocations and precipitates, making such gases unavailable for cavity nucleation. Nevertheless, the fact that many of the cavities which do form after prolonged irradiation, are associated with grain boundaries and stable particle-matrix interfaces, sites which are unsuitable for nucleation of pure vacancy clusters but are favored for formation of gas bubbles, suggests that the cavities are developed on gas bubbles. Their disappearance during heating at 523 K, which agrees with the temperatures required to sinter voids in purer aluminum,[8,14] indicates that they are not helium filled. They could, possibly, be filled with the more mobile hydrogen, since hydrogen forms bubbles readily in aluminum[15] and they become unstable above 473 K[15,16] as the hydrogen solubility in aluminum increases.

The second unobtrusive aspect of the microstructure is that the cavity distribution in the 5052 alloy is markedly different in one major respect from those in the other aluminum materials. In pure aluminum, 1100 Al and 6061 Al, the first cavities are developed in sheets lying close to and on each side of grain boundaries. These grow to be the largest cavities and provide planes of mechanical weakness and potential fracture sites. No such cavities were found in the 5052 alloy, even though the initial damage structure - dislocation loops - was developed in the vicinity of grain boundaries. It was noted, too, that there was no denuding of the Mg_2Si precipitate in the vicinity of grain boundaries.

The formation of Mg_2Si precipitate[2,3] is confirmed. Moreover, it is found that this precipitate persists to the highest fluences examined despite the presence of considerable excess silicon and the possible precipitate restructuring effects of the very high atomic displacement levels. About 1.2 atomic percent silicon is required to fix the total magnesium content as stochiometric Mg_2Si precipitate. This quantity is reached at a fluence of about 3×10^{26} fast n/m^2 ($\sim 5 \times 10^{26}$ thermal n/m^2). Since silicon is essentially insoluble in aluminum, any excess beyond this 1.2 percent must be accommodated as precipitate. About 7 percent is generated at the highest fluences. In magnesium-free aluminum(5,6) the silicon is manifest as elemental silicon. However, in the present irradiated alloys, the electron diffraction patterns are too complicated to permit easy recognition of spots from elemental silicon. Furthermore, in the presence of a co-generated precipitate of Mg_2Si, there is no guarantee that the excess silicon will assume elemental form. It may be taken up by the Mg_2Si or perhaps form a new precipitate. These considerations are currently under study to account for the many extra precipitate spots that give rise to the discontinuous rings in the diffraction patterns. Some of the complications arise from superimposed spots from particles freed from the matrix during electropolishing then trapped in random orientations on the foil surfaces. At present, no firm conclusions regarding the extra spots are available. When such conclusions are reached they will be published.

Whatever the final outcome of the search for the source of the extra diffraction spots, there is no doubt that the dominant radiation-induced microstructural defects in this irradiated 5052-0 alloy are the dislocations and the finely-dispersed precipitates. These defects are assumed to be responsible for the considerable increases in tensile strength and losses in ductility with increasing irradiation. A quantitative accounting of the tensile strengths in terms of hardening by the microstructural defects is not attempted here because previous work on 1100 Al (6,8,17), 6061 Al (18) and 5052 Al (2) has shown satisfactory correlations, and there is little to be gained in repeating the exercise. The decreases in strength with increasing test temperature are not caused by recovery or coarsening of the microstructure, and they are larger than the 5% or so decrease expected for the change in shear modulus.[19] Thermally-assisted surmounting of obstacles by dislocations is a likely cause.

It is worth noting that the strength levels attained in the 5052 alloy are much higher than those reached in 1100-0 (6), 6061-0 (11) and 6061-T6 (11) aluminum alloys irradiated and tested under the same conditions. The precipitate was also more finely distributed in the 5052 alloy which would favor greater strength. This is consistent with the conclusions of King and Jostsons[2] who pointed out that the radiation-induced Mg_2Si precipitate in 5052 alloy is finer than that produced in 6061 alloy by thermal aging.

The fineness of the precipitate may also be at the root of the increase in strength with increases in thermal-to-fast flux ratio. These strength increases are greater than would be expected from a simple increase in the silicon level. For example, in Fig. 5 the 0.2 percent flow stress at 1×10^{26} n/m^2 on the unbroken curve which represents $\phi th/\phi_f$ = 1.7 is 310 Mpa and the estimated silicon concentration is 0.4 weight percent; at $\phi th/\phi_f$ = 2.1 (the dotted curve) the flow stress is 375 Mpa and the silicon level is ~ 0.5 percent. Yet the flow stress corresponding to ~ 0.5 percent silicon on the $\phi th/\phi_f$ = 1.7 curve is only 325 Mpa. The difference may be due to the higher silicon production rate at $\phi th/\phi_f$ = 2.1 causing faster, and hence finer, formation of Mg_2Si particles.

The loss in ductility from irradiation appears to be a natural consequence of the radiation hardening. The bulk of the loss occurred through reduction in uniform strain, a reflection of the increased resistance to movement of slip dislocations provided by the defect structure. Microstructural examination revealed nothing untoward about the deformation mechanism, and the fractures were apparently transgranular and locally ductile, even those occurring at the lowest bulk strains. In only one case, a high fluence specimen tested at 423K, did the fracture path change, and this displayed a facetted surface similar to the intergranular failures observed by King and Jostsons(2) in their creep specimens. The implication is that slow strain rates and/or high test temperatures may encourage intergranular separation. At normal tensile test rates, the alloy demonstrated good resistance to grain boundary failure, more so than 1100 and 6061 alloys (6, 11) under similar conditions. The absence of grain boundary denuded zones and near-grain boundary cavities may be contributing factors. Nevertheless, the overall ductility of 5052 alloy was lower than that of 1100 and 6061 alloys at fast fluences greater than 2×10^{26} n/m^2, and was very low at 423K. Modest necking strains persisted at all test temperatures.

One final comment concerns the suppression of serrated yielding by irradiation. Serrated yielding is often attributed to the sudden release of dislocations from an impeding atmosphere of solute atoms, followed by re-segregation of the atmosphere and a repeat process. Presumably in the unirradiated 5052 alloy, the dissolved magnesium provides the Suzuki atmosphere. Interestingly, irradiation eliminates serrated flow long before all of the magnesium is estimated to be removed from solution. Serrated flow disappears after irradiations in the mid $10^{25} n/m^2$, at which point there is only about one-sixth of the transmutation-produced silicon required to draw all the magnesium from solution as soluble Mg_2Si. It is quite possible, though, that nucleation of Mg_2Si embryo is complete at such fluences, and is on a fine enough scale to inhibit segration of magnesium to dislocations.

CONCLUSIONS

1) Irradiation of the 5052 aluminum-2.2% magnesium, solid solution alloy to high fluences in a mixed neutron spectrum at 328 K converts the alloy to a precipitation-hardened 6000 type alloy.

2) The precipitates are derived from insoluble transmutation-produced silicon, and include Mg_2Si.

3) The alloy displays a high resistance to cavity formation.

4) The damage microstructure is stable up to 523 K in short-term anneals.

5) The flow stress and tensile strength are increased by factors up to 6.5 and 2.7, respectively, with accompanying loss of ductility associated with marked reduction in uniform strain.

6) The changes in tensile properties are consistent with hindrance of slip dislocations by dispersed phases.

7) Increasing the thermal/fast neutron flux ratio from 1.7 to 2.1 gives further strengthening beyond that expected from a simple increase in silicon level.

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FIGURE CAPTIONS

Fig. 1. Dislocation loops developing near grain boundaries and around inclusions at $1.5 \times 10^{25} n/m^2$, >0.1 MeV.

Fig. 2. (a) Precipitates and dislocations at $5.7 \times 10^{26} n/m^2$, with (001) diffraction pattern. (b) Cavities and larger matrix precipitates at $1.8 \times 10^{26} n/m^2$. (c) Heavy discontinuous precipitation at grain boundaries. Thermal fluences are $\sim$1.7 times the quoted fast fluences.

Fig. 3. Effects of radiation on tensile curves at 323 and 423 K.

Fig. 4. Fluence dependence of tensile properties at 323 K ($0.35T_m$).

Fig. 5. Fluence dependence of tensile properties at 373 K ($0.4T_m$).

Fig. 6. Fluence dependence of tensile properties at 423 K ($0.45T_m$).

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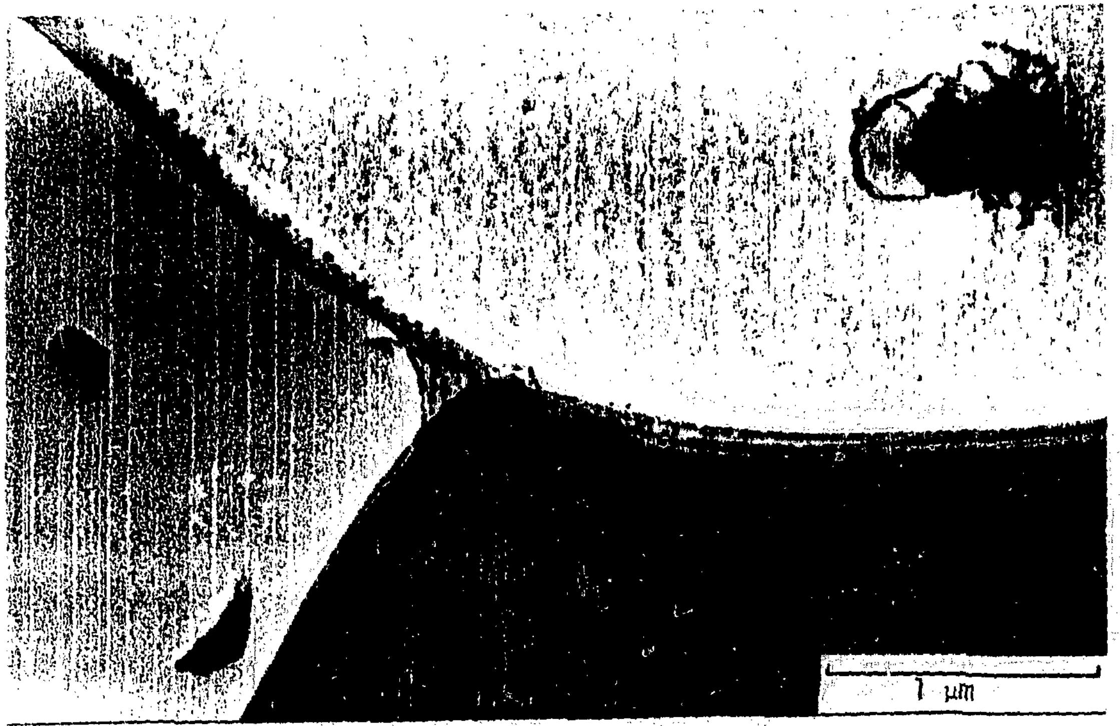

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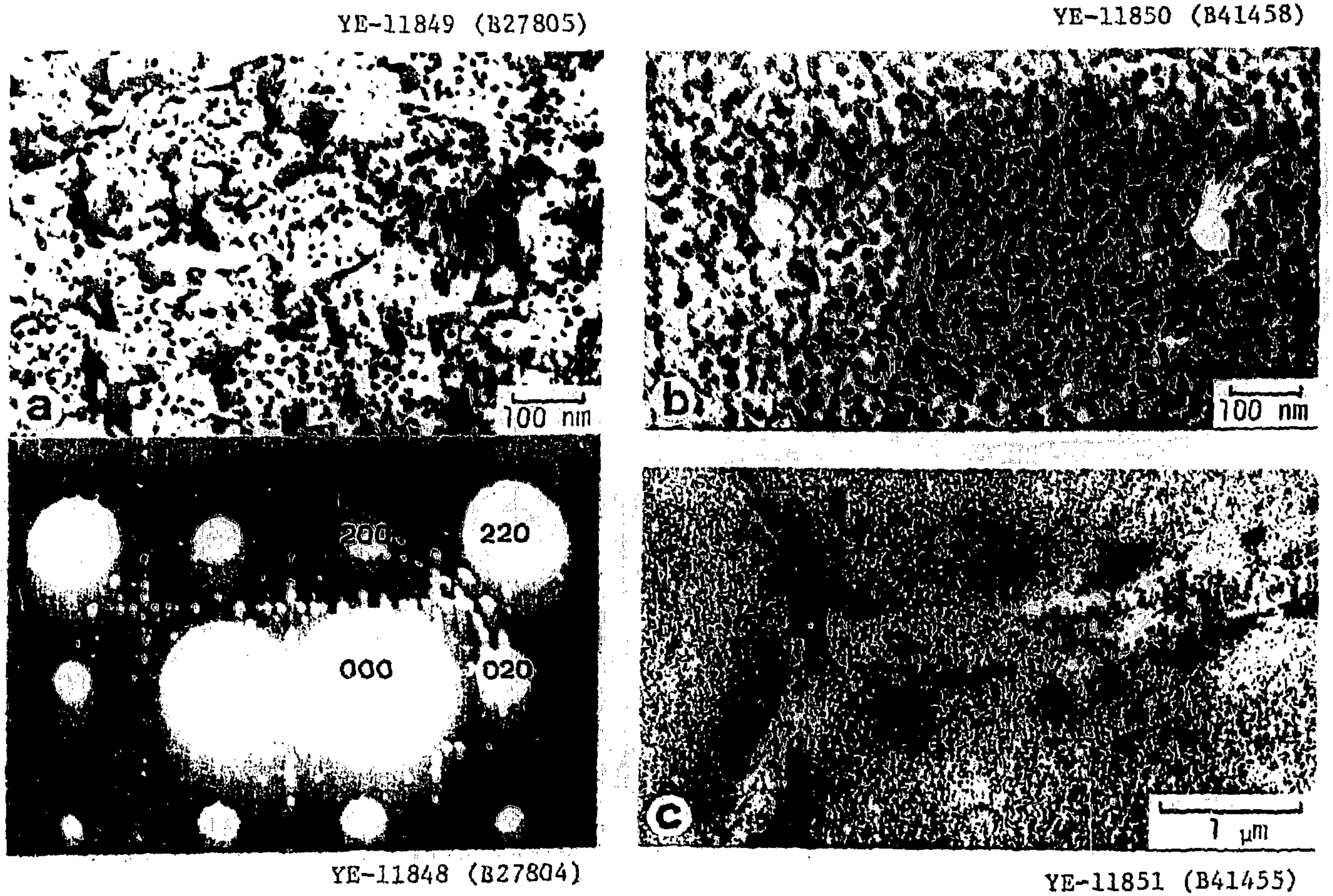

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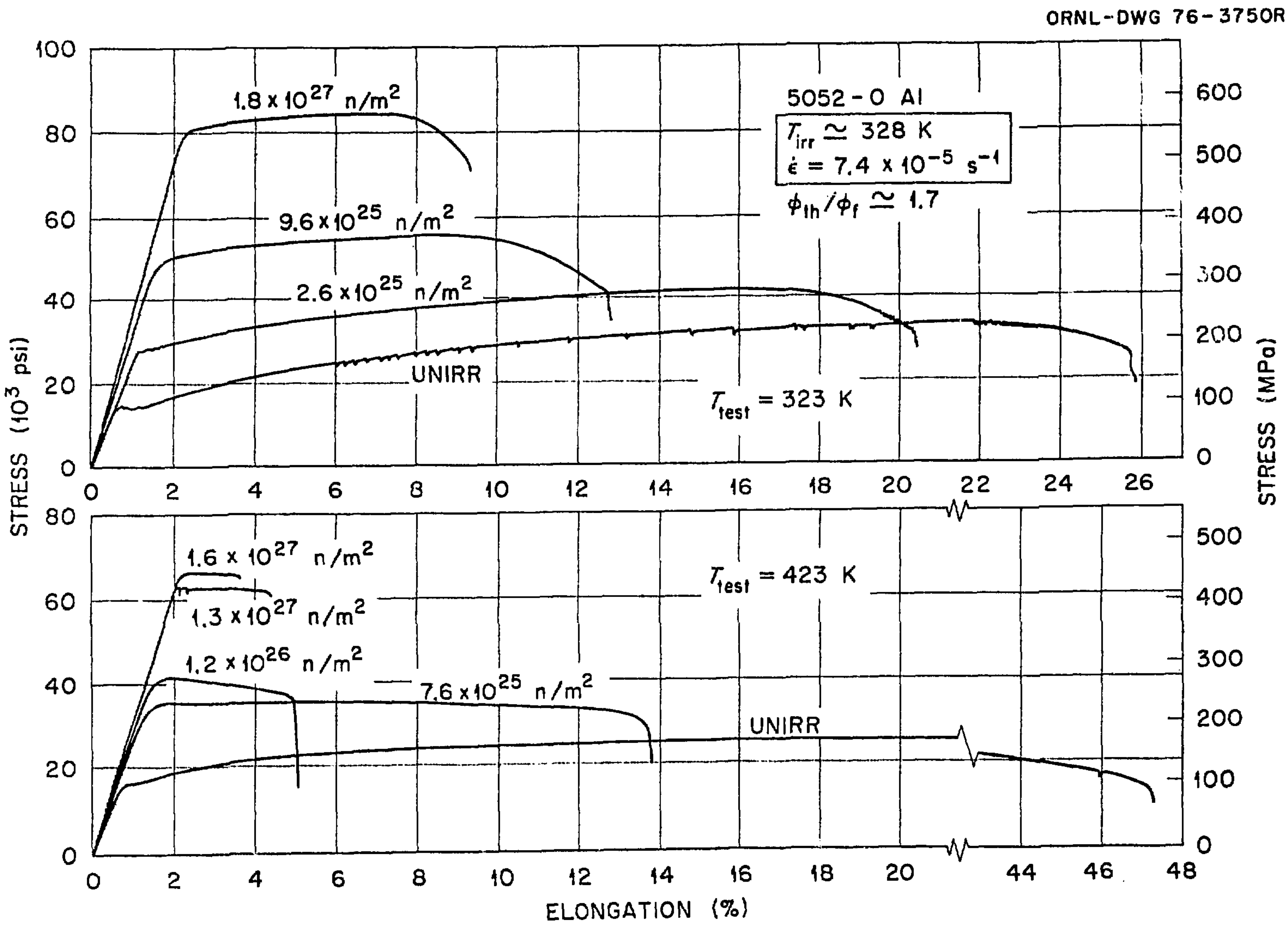

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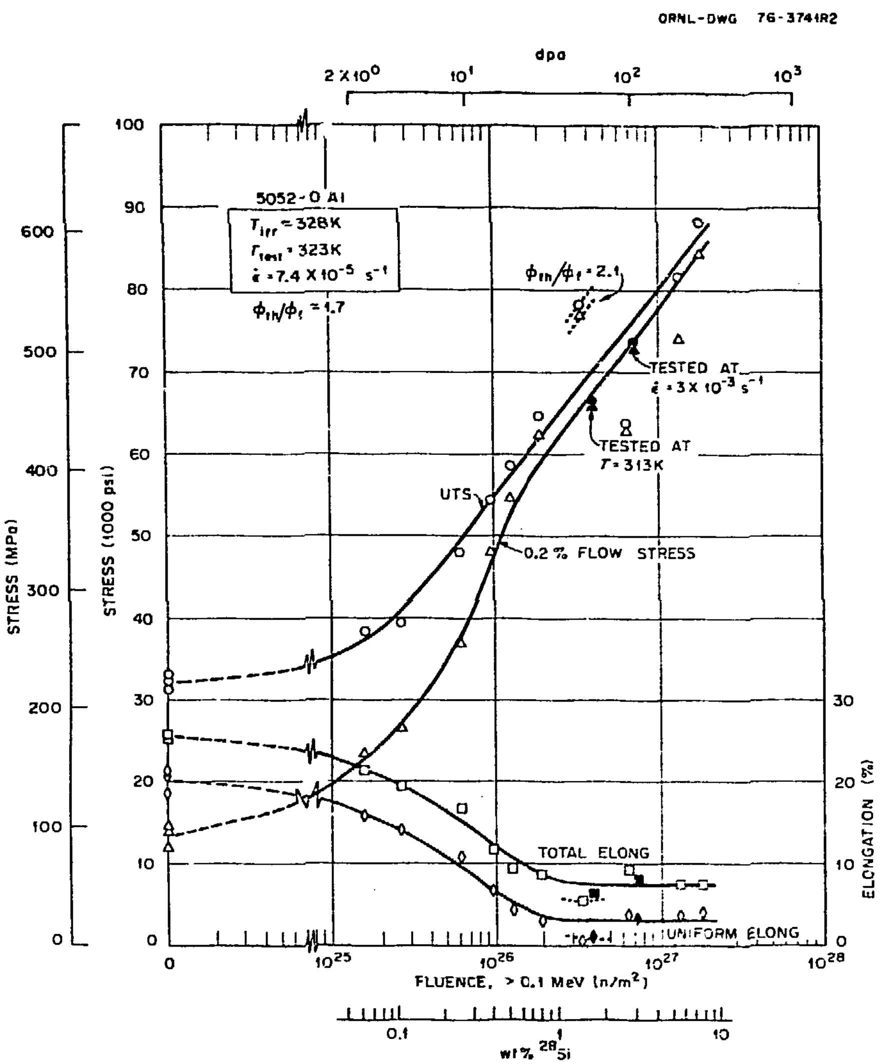

Fig. 4. Fluence dependence of tensile properties at 323 K (0.35 T_m).

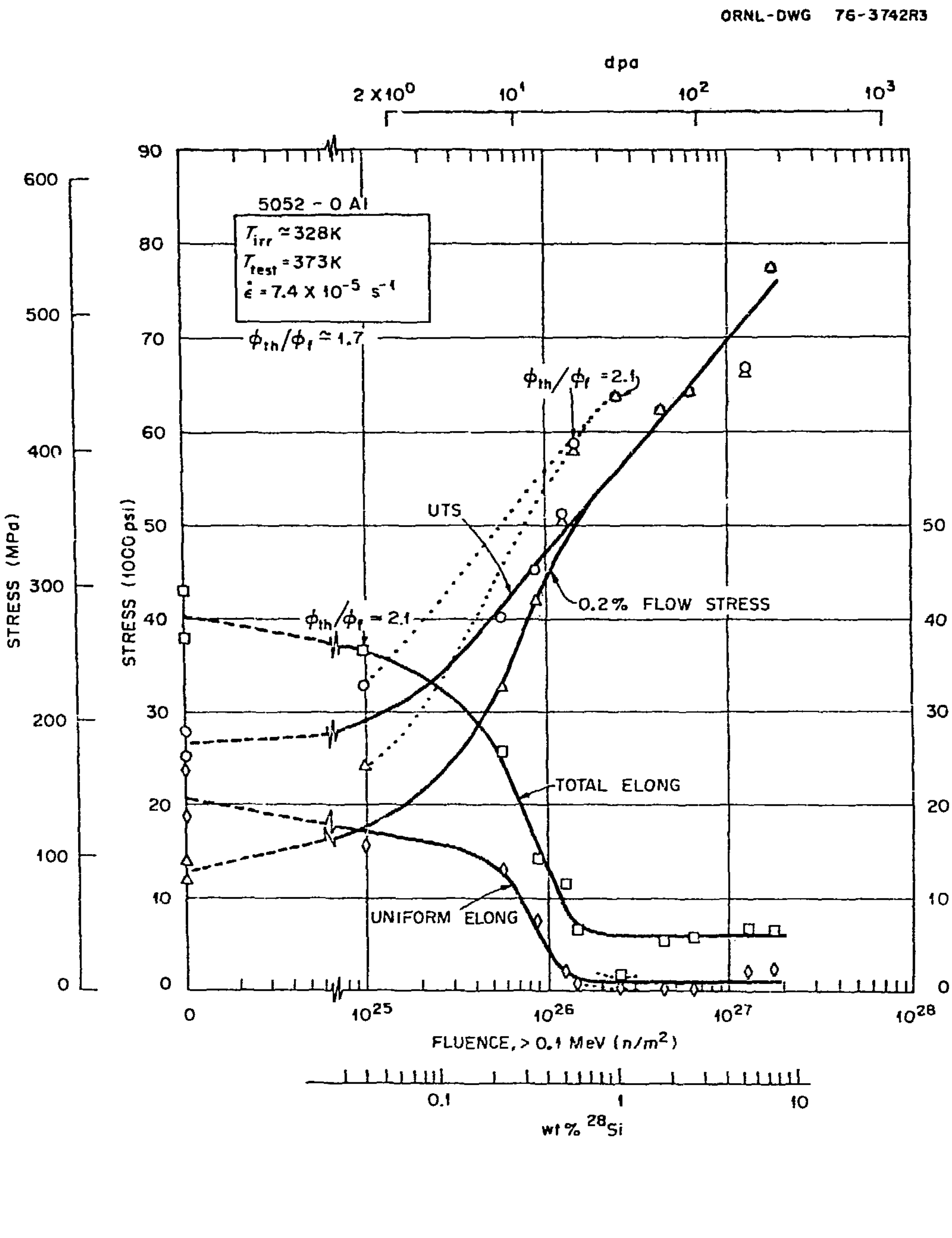

Fig. 5. Fluence dependence of tensile properties at 373 K (0.4 T_m).

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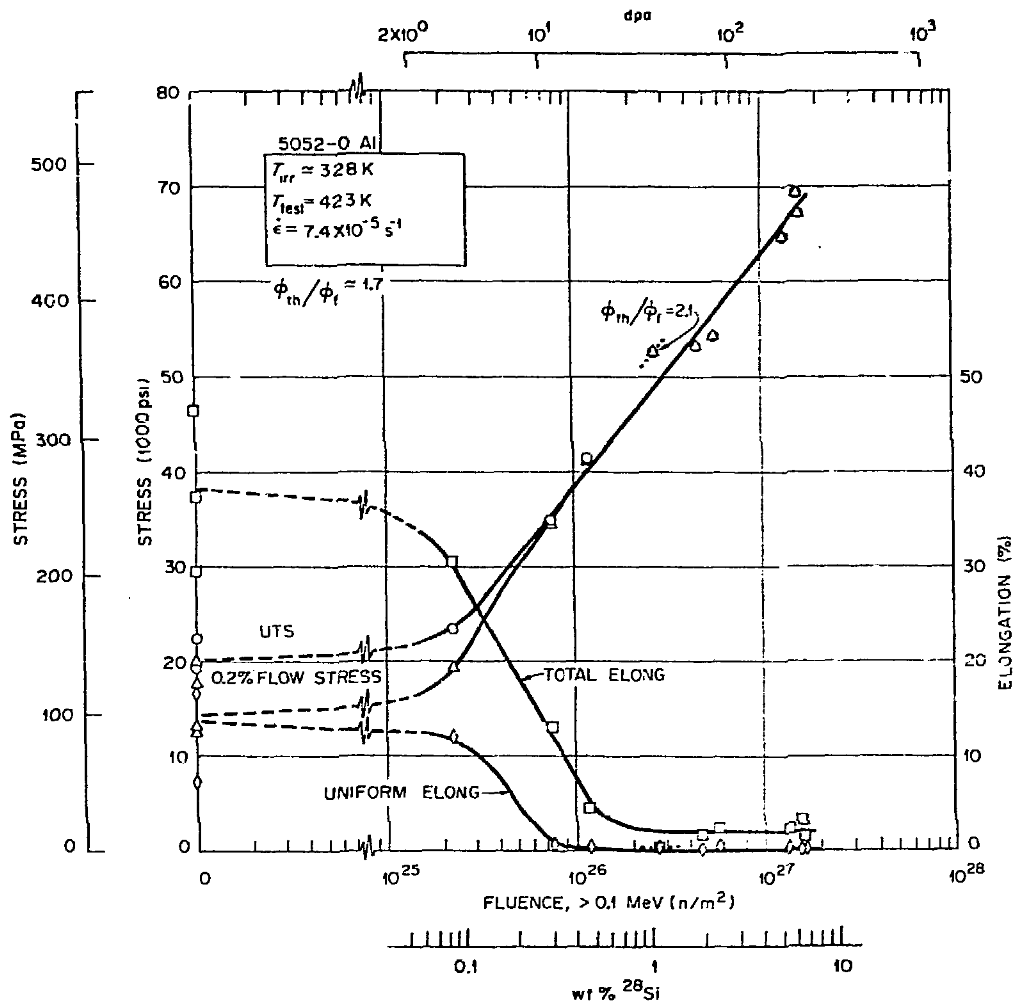

Fig. 6. Fluence dependence of tensile properties at 423 K (0.45 T_m).