

2. 1 .

Conf-91111--24

PNL-SA--19472

DE92 006422

TENSILE PROPERTY CHANGES OF METALS
IRRADIATED TO LOW DOSES WITH FISSION,
FUSION AND SPALLATION NEUTRONS

RECEIVED BY OSTI
JAN 21 1992

H. L. Heinisch
W. F. Sommer

M. L. Hamilton
P. D. Ferguson

November 1991

Presented at the
5th International Conference on
Fusion Reactor Materials
November 17-22, 1991
Clearwater, Florida

Work supported by
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

TENSILE PROPERTY CHANGES OF METALS IRRADIATED TO LOW DOSES
WITH FISSION, FUSION AND SPALLATION NEUTRONS

H. L. Heinisch, M. L. Hamilton,
W. F. Sommer* and P. D. Ferguson**

Pacific Northwest Laboratory, Richland, WA 99352

*Los Alamos National Laboratory, Los Alamos, NM 87545

**University of Missouri-Rolla, Rolla, MO 65401

ABSTRACT

Radiation effects due to low doses of spallation neutrons are compared directly to those produced by fission and fusion neutrons. Yield stress changes of pure Cu, alumina-dispersion-strengthened Cu and AISI 316 stainless steel irradiated at 36-55°C in the Los Alamos Spallation Radiation Effects Facility (LASREF) are compared with earlier results of irradiations at 90°C using 14 MeV D-T fusion neutrons at the Rotating Target Neutron Source and fission reactor neutrons in the Omega West Reactor. At doses up to 0.04 displacements per atom (dpa), the yield stress changes due to the three quite different neutron spectra correlate well on the basis of dpa in the stainless steel and the Cu alloy. However, in pure Cu, the measured yield stress changes due to spallation neutrons were anomalously small and should be verified by additional irradiations. With the exception of pure Cu, the low dose, low temperature experiments reveal no fundamental differences in radiation hardening by fission, fusion or spallation neutrons when compared on the basis of dpa.

Introduction

The Los Alamos Spallation Radiation Effects Facility (LASREF) is a potential irradiation facility for fusion reactor materials testing, including ITER materials and components. Spallation neutron sources have not been utilized extensively in fusion materials research, primarily because there are no high flux sources, but also because of concerns about their suitability relative to fusion reactor neutron environments.

The primary question concerning the use of spallation neutrons for fusion materials testing is whether there are undesirable effects of the "high energy tail," i.e., the neutrons in the energy range from about 20 MeV to hundreds of MeV that occur in diminishing numbers with increasing energy. The effects of transmutations and displacement damage produced by these neutrons are largely unknown. The present experiment is the first step of a potentially broader study designed to investigate whether there are fundamental differences in displacement damage produced by spallation neutrons relative to damage produced in fission and fusion reactor neutron spectra.

Yield stress changes of metals and alloys due to low dose irradiations with spallation neutrons in LASREF are compared to results obtained on the same materials irradiated to similar doses at 90° C with fission reactor neutrons in the Omega West Reactor (OWR) and with 14 MeV D-T fusion neutrons in the Rotating Target Neutron Source (RTNS-II)[1,2,3]. In the earlier comparisons of OWR and RTNS-II it was found that, for all the alloys tested, the fission and fusion irradiation effects correlate well when compared on the basis of displacements per atom (dpa). However, in the case of pure Cu [1] and pure Nb [4] some differences between fission and fusion neutron irradiation effects were noted.

A limited examination of microstructures has been done by transmission electron microscopy (TEM) on the same Cu [5,6] and 316 SS [7] irradiated at 90°C in RTNS-II and OWR for the tensile tests. In general, the radiation-induced hardening scales with the visible microstructure. Comparative examinations of the microstructure of pure Cu specimens have recently been

performed on specimens irradiated in LASREF, RTNS-II and OWR, and the results are reported in another paper in these proceedings [8].

Experimental Details

The LASREF irradiation contained miniature tensile specimens and TEM disks of 99.999% pure Cu (annealed at 450°C for 15 m), an alumina-dispersion-strengthened Cu alloy (CuAl25) with 0.25% Al in the form of Al₂O₃ (annealed at 982°C for 60 m) and AISI 316 stainless steel (annealed at 1000°C for 10 m). These three materials are a subset of the materials irradiated in the earlier OWR/RTNS-II comparisons [3] that includes the general range of behaviors observed in those studies.

The specimens were placed into four helium-filled capsules, which were placed into the LASREF facility through tubes also used for "fast" rabbit activation foil exposures [9,10]. The capsules were positioned at different distances from the center of the neutron-producing targets, exposing each capsule to a different flux level. The capsule temperatures were monitored with chromel-alumel thermocouples attached to each capsule. Details of the LASREF irradiations are given in Table 1.

Each capsule contained activation foils of Cu, Ni, Co and Fe for neutron dosimetry. The gamma ray intensities of the radiation-induced radioisotopes were measured at Los Alamos National Laboratory, and the results were analyzed with the STAY'SL [11] spectral-fitting code. Both the neutron spectra and He/dpa ratios are documented in reference 12. The average fluence experienced by each capsule during the irradiation of 3.8 x 10⁶ s duration is listed in Table 1 along with coarse spectral information. The fluences of two categories of high energy neutrons are listed: the fluence of neutrons with energies greater than 0.1 MeV, which produce most of the displacement damage, and the fluence of neutrons with energies greater than 20 MeV, the so-called "high energy tail."

The dosimetry foils in capsule 1 were badly corroded, and they were not useful for analysis. The total flux value for capsule 1 given in Table 1 was

estimated from the relative fluxes at the four positions determined in previous work [9]. Neutron energy-dependent displacement cross sections [13] for Cu were used to calculate the dpa in Cu for each capsule. The dpa value for capsule 1 was estimated assuming the same neutron spectrum as capsule 3. Dpa values for 316 SS were estimated by multiplying the Cu dpa values by 0.75, the ratio of the displacement energies in Cu and Fe.

The test specimens in capsule 1 were also corroded, but not so badly that tensile tests and TEM could not be performed on them. The cause of the corrosion still eludes the experimenters. The tensile tests on specimens from capsule 1 appear to be unaffected by the corrosion, and the results are included in this paper. However, since the neutron dose for capsule 1 is also uncertain, the capsule 1 data should be viewed with caution.

Tensile tests were performed at room temperature at Pacific Northwest Laboratory on an apparatus designed specifically for testing miniature specimens [14].

Results

The results of the tensile tests are reported in Table 2. Figure 1 shows the changes in 0.2% offset yield stress as a function of displacement damage for 316 SS irradiated in LASREF. Each point is the result of a single tensile test. The LASREF data are compared with earlier results from RTNS-II and OWR irradiations of the same material at 90°C [3]. The data are plotted against $\text{dpa}^{1/4}$ because the RTNS-II and OWR data are linear functions of that quantity. Dependence on the fourth root of dpa is typical for this dose range and temperature. It is consistent with the dispersed barrier model of irradiation hardening at doses where cascades interact but defect densities are not saturated. The error bar on the single point in the center of the graph indicates the minimum $\pm 5\%$ error in the value of the absolute yield stress, based on the estimated precision of the testing technique. The straight line through the data points was fitted to the RTNS-II and OWR data only. Overall, the agreement of data from all three neutron spectra is quite good. There are some minor systematic differences: the LASREF data exhibit

somewhat greater scatter than the RTNS-II and OWR data, and they appear to lie along a line of slightly greater slope (the results from capsule 1 at the highest dose are suspect).

Figure 2 compares LASREF data with earlier RTNS-II and OWR data for CuAl25 [2]. As in 316 SS, the yield stress changes are proportional to the fourth root of dpa. The line in the figure was fitted to only the RTNS-II and OWR data, and the error bar represents the 5% precision of the measurement of the absolute yield stress. There is good agreement among the results for LASREF, RTNS-II and OWR.

Figure 3 compares LASREF data for pure Cu with data from the earlier RTNS-II and OWR irradiations [3]. Both RTNS-II data and OWR data vary linearly with the fourth root of dpa, but the slope of the RTNS-II data is 1.4 times that of the OWR data. More than twice as many dpa are needed in OWR as in RTNS-II to produce a yield stress change of 200 Mpa in pure Cu. The LASREF data have considerably larger scatter than those of RTNS-II and OWR (although, if the two highest measured values of yield stress change are ignored, the scatter at each dpa value in the LASREF data is on the order of the $\pm 5\%$ precision of the testing). The remarkable characteristic of the LASREF data is that the yield stress changes are considerably less than in RTNS-II and OWR.

Discussion

The yield stress changes of 316 SS and CuAl25 irradiated with LASREF spallation neutrons correlate well with those measured on the same materials irradiated in RTNS-II and OWR, when compared on the basis of dpa. In Cu the yield stress changes due to the spallation neutrons are markedly lower than those due to the fission or fusion neutrons. They are too large and are in the wrong direction to be due to differences in temperature or dose rate.

Earlier irradiations of pure Cu in RTNS-II [3] at temperatures ranging from room temperature to 290°C demonstrated that radiation hardening is

sensitive to the temperature, since defects in Cu have significant mobility in this temperature range. Increasing the temperature in Cu delays the onset of the transition fluence at which significant hardening begins to take place. If temperature were the only variable, then Cu irradiated at 36-55°C should have slightly higher yield stress values than when irradiated at 90°C. However, the yield stress changes due to irradiation in LASREF are about the same as those obtained in RTNS-II at 200°C. Thermocouples were attached to each LASREF capsule, and the specimens were tightly packed within each capsule. There is no indication that the specimens experienced temperatures greater than about ten degrees above the reported temperatures. If the LASREF capsules had been near 200°C, the yield stress changes in 316 SS should also have been smaller [3] than shown in Fig. 1.

Displacement rates in the LASREF irradiations varied with distance from the target over the range from 4.2×10^{-10} to 5.3×10^{-9} dpa/s, approximately three times the rates for comparable dpa values in RTNS-II. In OWR the displacement rate is much higher, 3.7×10^{-8} dpa/s. Earlier RTNS-II irradiations were also done in pure Cu at damage rates four times higher than those for the data plotted here. At the higher dose rates the yield stress changes were about 10% greater than at the lower dose rate. Thus, there is no evidence to support rate effects as the cause of the differences in yield stress changes of Cu irradiated in LASREF, RTNS-II and OWR.

In a companion paper in these proceedings [8] the microstructures of Cu specimens irradiated in LASREF are compared with those of Cu irradiated in RTNS-II and OWR. The number densities of TEM-visible defects for specimens irradiated in all three neutron spectra correlate well when compared on the basis of DPA, and the visible defect size distributions are about the same. It is difficult to postulate a situation in which the visible microstructures are the same but the yield stresses are different. Perhaps the only possibility is that invisible defects are produced in RTNS-II and OWR that contribute to the hardening, but no invisible defects are produced in LASREF (or they were eliminated by an undetected temperature excursion). The search for a systematic error in the tensile tests of Cu specimens has been negative.

As to whether there are spectral differences in irradiation effects in pure Cu, the evidence based on yield stress data is inconclusive.

Conclusions

In low-dose irradiations, the changes in tensile properties of 316 SS and CuAl25 produced by fission reactor neutrons, D-T fusion neutrons and LASREF spallation neutrons are the same when compared on the basis of dpa. The small yield stress changes measured for pure Cu irradiated in LASREF remain unexplained.

Excluding the Cu data, these low dose, low temperature experiments show that any fundamental differences in damage production at low doses by LASREF spallation neutrons and those of a fission or fusion reactor can be accounted for by comparing the test data on the basis of dpa.

Acknowledgements

This work was supported in part by the U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

References

1. H. L. Heinisch, S. D. Atkin and C. Martinez, J. Nucl. Mater. 141-143 (1986) 807.
2. H. L. Heinisch and C. Martinez, J. Nucl. Mater. 141-143 (1986) 883.
3. H. L. Heinisch, J. Nucl. Mater. 155-157 (1988) 121.
4. J. B. Mitchell, R. A. VanKonynenburg, C. J. Echer and D. M. Parkin, Proc. Int. Conf. on Radiation Effects and Tritium Technology for Fusion Reactors, Gatlinburg, TN, Oct. 1-3, 1975, Vol. II, 172.
5. S. Kojima, S. J. Zinkle and H. L. Heinisch, J. Nucl. Mater. 179-181 (1991) 982.
6. A. Horsewell, B. N. Singh, S. Proennecke, W. F. Sommer and H. L. Heinisch, J. Nucl. Mater. 179-181 (1991) 924.
7. N. Yoshida, H. L. Heinisch, T. Muroga, K. Araki and M. Kiritani, J. Nucl. Mater. 179-181 (1991) 1078.
8. T. Muroga, H. L. Heinisch, W. F. Sommer and P. D. Ferguson, J. Nucl. Mater. (this conference).
9. D. R. Davidson, R. C. Reedy, L. R. Greenwood, W. F. Sommer and M. S. Wechsler, ASTM STP 956 (1987) 730.
10. W. F. Sommer, W. Lohmann, K. Graf, I. K. Taylor and R. M. Chavez, ASTM STP 956 (1987) 718.
11. F. G. Perry, Oak Ridge National Laboratory Report, ORNL-TM-6062 (1977).
12. W.F. Sommer, F.A. Garner and B.M. Oliver, Fusion Reactor Materials Semiannual Progress Report, DOE/ER-0313/11 (1991).

13. M. S. Wechsler, D. R. Davidson, I. R. Greenwood and W. F. Sommer, ASTM STP 870 (1985) 1189.
14. N.F. Panayotou, S.D. Atkin, R.J. Puigh and B.A. Chin, ASTM STP 888 (1986) 201.

Table 1. LASREF IRRADIATIONS

Capsule ID	Distance from Target, m	Temp °C	Total Fluence 10^{15} n/m ²	Fluence E>0.1 MeV 10^{15} n/m ²	Fluence E>20 MeV 10^{15} n/m ²	DPA in Cu**
7	0.38	36	8.9	2.7	0.47	0.0016
5	0.27	37	27	9.5	1.3	0.0037
3	0.18	44	40	21	2.9	0.011
1	0.12	55	73*	--	--	0.020*

* Estimate based on Ref. 9

** Multiply by 0.75 to get approximate dpa for 316 SS

Table 2. LASREF TENSILE TEST DATA

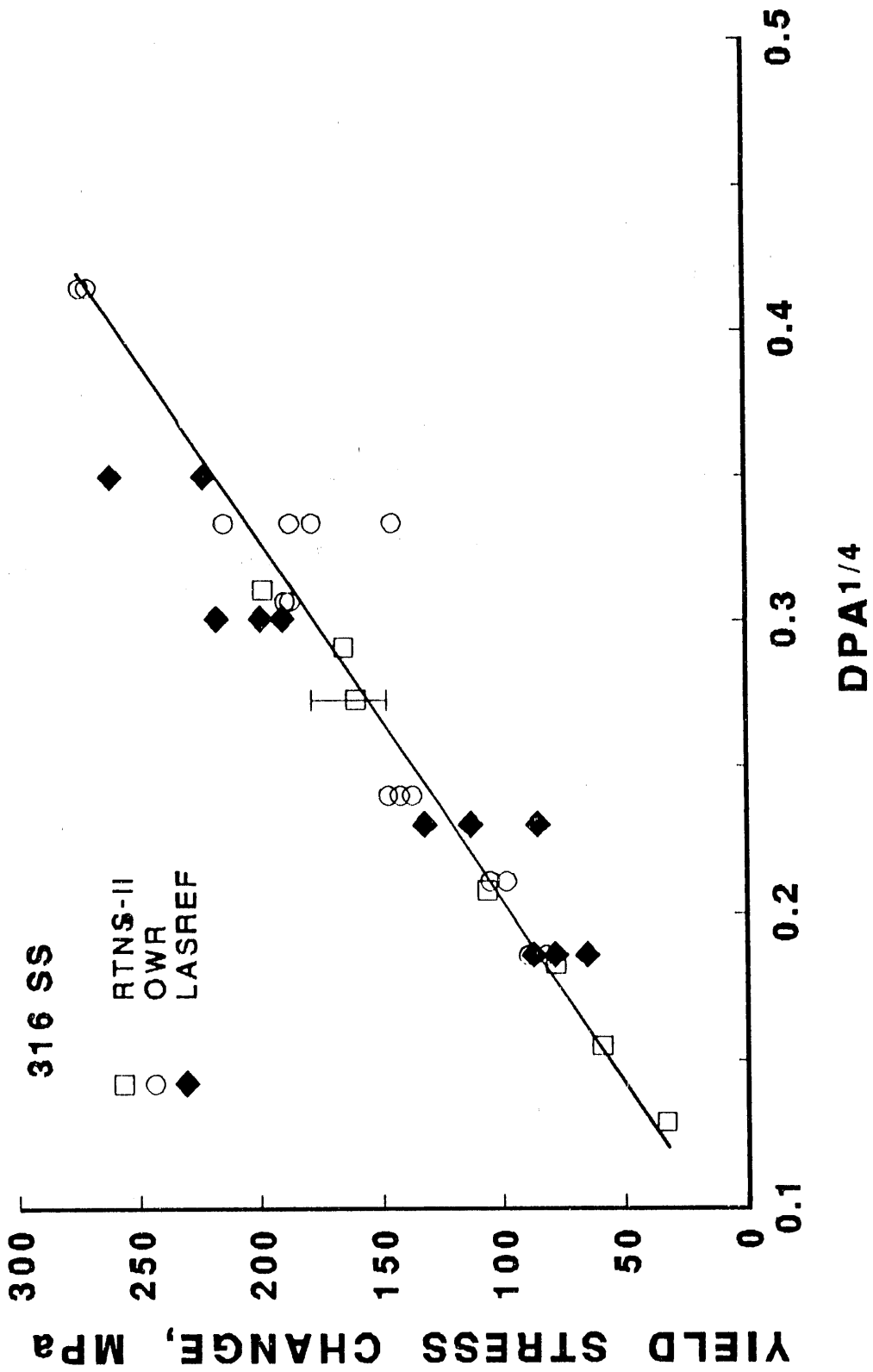
Capsule ID	Yield Stress MPa	Ultimate Strength, MPa	Uniform Elongation, %	Total Elongation, %
Cu				
unirrad	77	206	26.1	29.0
7	104	163	13.1	18.7
7	127	180	8.9	13.8
7	165	213	15.9	21.6
5	120	168	16.5	23.7
5	132	175	11.3	15.0
5	142	194	8.4	13.3
3	128	165	28.4	32.5
3	139	185	16.4	21.9
3	177	223	13.6	17.9
1	151	177	9.0	13.3
1	163	191	11.9	18.2
CuAl25				
unirrad	417	456	9.1	15.5
7	439	451	5.6	8.4
7	445	459	6.6	12.1
5	475	483	0.5	7.3
5	479	481	0.5	3.9
3	503	523	0.5	2.9
3	505	518	0.3	3.2
1	471	507	0.7	1.5
1	536	544	0.4	1.2

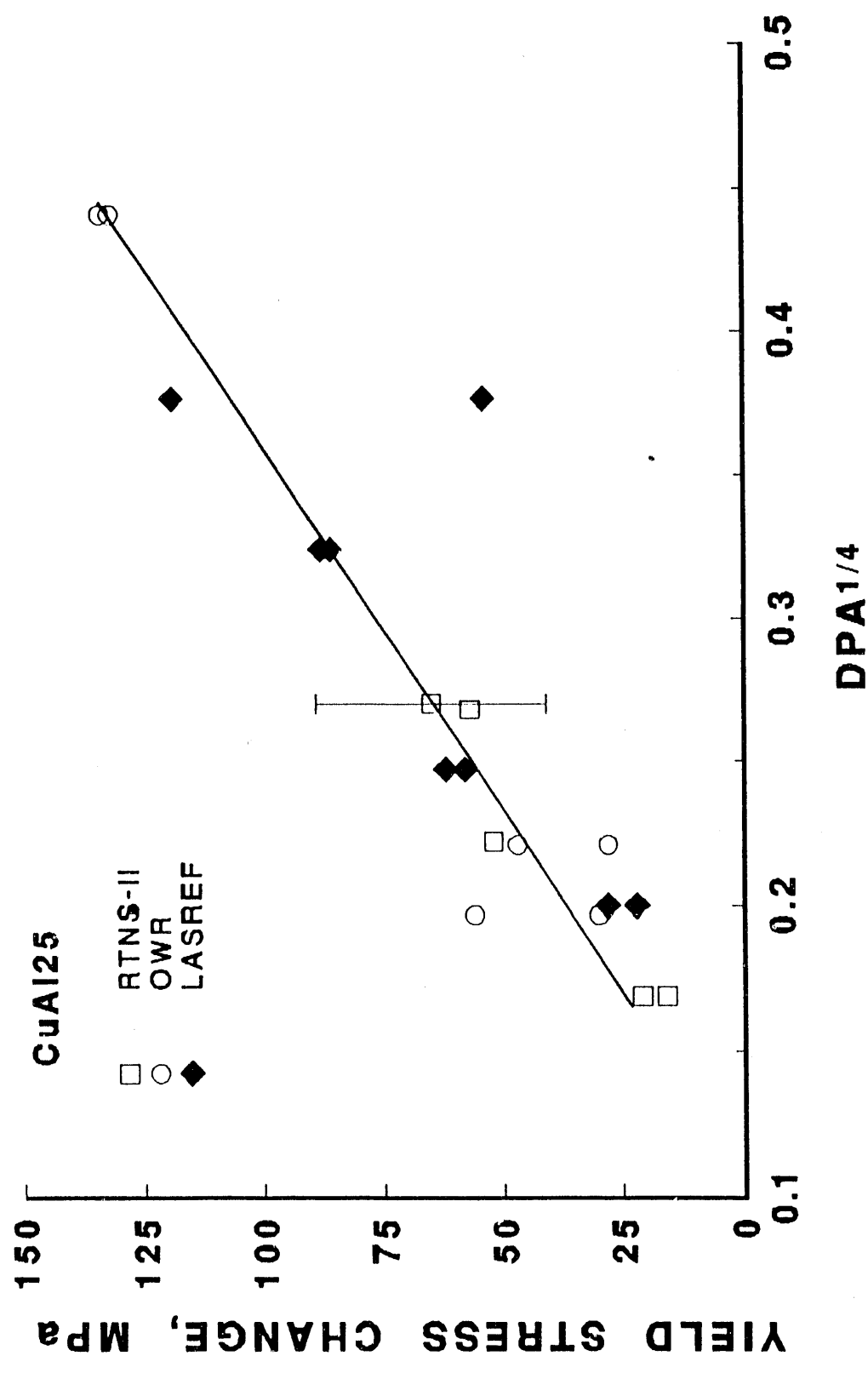
Table 2 continued

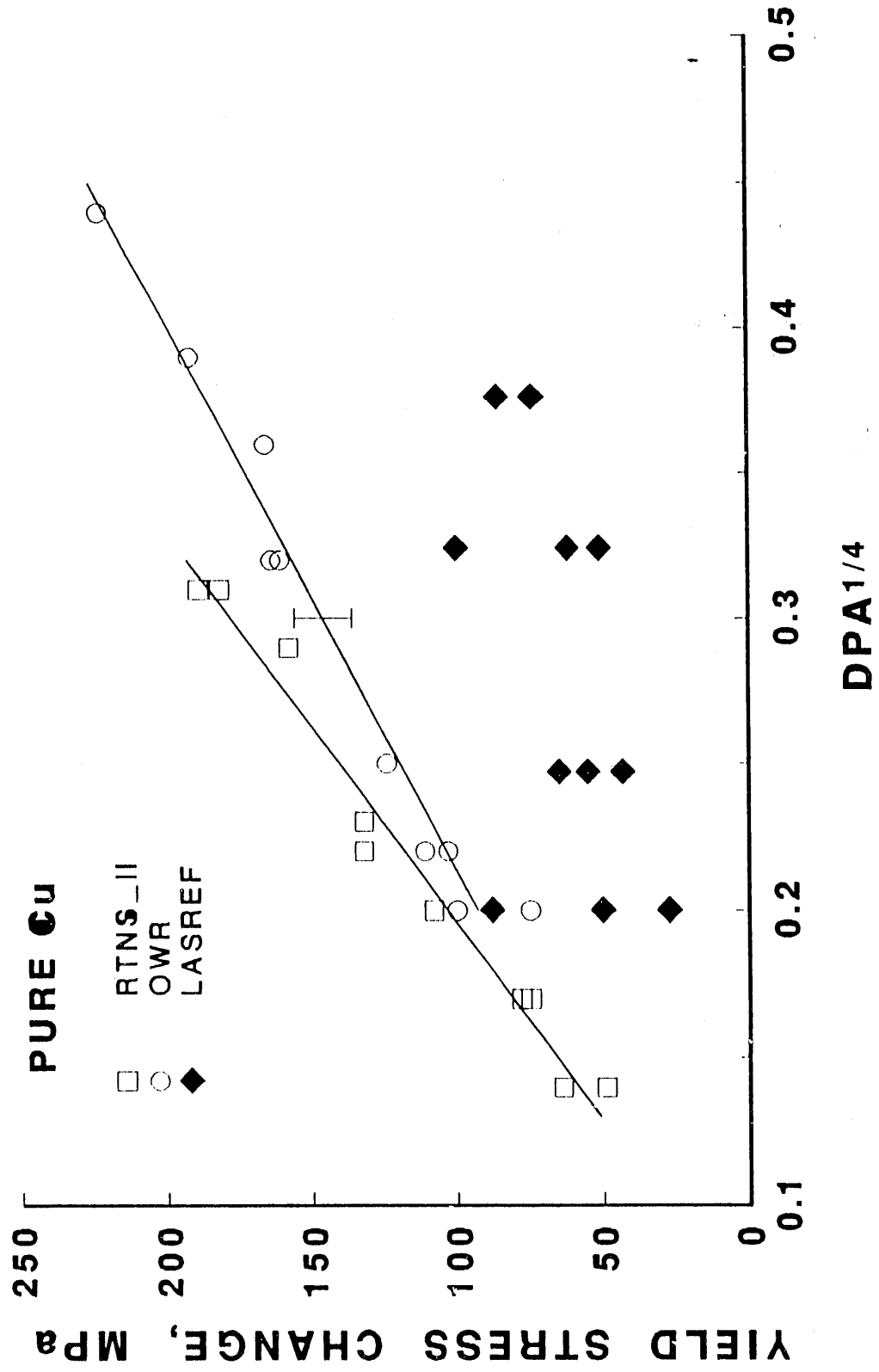
Capsule ID	Yield Stress MPa	Ultimate Strength, MPa	Uniform Elongation, %	Total Elongation, %
316 SS				
unirrad	269	648	57.2	61.7
7	334	608	50.1	55.6
7	347	633	50.0	55.3
7	356	655	50.9	55.8
5	354	564	34.0	37.7
5	382	619	47.1	51.9
5	401	651	47.1	52.9
3	459	673	36.7	43.3
3	468	687	39.7	43.9
3	486	715	32.3	37.7
1	491	675	37.2	42.9
1	491	679	32.3	40.7
1	530	703	21.7	25.9

FIGURE CAPTIONS

1. The radiation-induced change in yield stress plotted against the fourth root of dpa for AISI 316 stainless steel irradiated in LASREF, RTNS-II and OWR. The straight line is fitted to the RTNS-II and OWR data only. The error bar near the center of the graph represents the estimated $\pm 5\%$ uncertainty in the absolute yield stress due to the precision of the measurements.
2. The radiation-induced change in yield stress plotted against the fourth root of dpa for alumina-dispersion-strengthened Cu irradiated in LASREF, RTNS-II and OWR. The straight line is fitted to the RTNS-II and OWR data only. The error bar near the center of the graph represents the estimated $\pm 5\%$ uncertainty in the absolute yield stress due to the precision of the measurements.
3. The radiation-induced change in yield stress plotted against the fourth root of dpa for pure (99.999%) Cu irradiated in LASREF, RTNS-II and OWR. Straight lines are fitted to the RTNS-II and OWR data. The error bar near the center of the graph represents the estimated $\pm 5\%$ uncertainty in the absolute yield stress due to the precision of the measurements.







END

**DATE
FILMED**

2 / 24 / 92

