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THE EFFECT OF IRRADIATION ON THE BEND TRANSITION TEMPERATURES OF MOLYBDENUM- AND NIOBIUM-BASE ALLOYS

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H. E. McCoy and J. R. Weir

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ABSTRACT

Because of the potential use of refractory metals as structural materials in nuclear reactors, it is necessary that the influence of irradiation on the ductility of these materials be evaluated. The present study is concerned with the development of suitable test equipment for evaluating the ductile-to-brittle transition temperature of irradiated specimens and with the use of this equipment to evaluate the ductile-to-brittle temperature for several irradiated specimens. The equipment developed for this purpose fits in a standard tensile machine and is capable of remote operation over the temperature range of −73 to 261°C. The materials studied after irradiation to a neutron dosage of the order of 10^{20} nvt (> 1 Mev) include Mo–0.5% Ti, Nb–1% Zr, welded Nb–1% Zr, and D-43 (10% W–1% Zr–0.1% C). The variables studied were (1) the metallurgical state of the material prior to irradiation, (2) irradiation dose, and (3) postirradiation annealing. The transition temperature of the Mo–0.5% Ti was determined on the basis of the deflection of the material prior to fracture. The behavior of the Nb–1% Zr and D-43 alloys was analyzed on the basis of the yield strength.

INTRODUCTION

Refractory metals have been shown to have many properties that make them satisfactory for nuclear applications. However, not many of the refractory metals and their alloys have been studied in a nuclear environment to determine how their mechanical properties are altered. It is the purpose of the present investigation to help fill in this information through studies of the low-temperature ductility and strength of several molybdenum- and niobium-base alloys before and after neutron irradiation.

Many of the body-centered cubic metals have been shown to exhibit at some temperature a transition from ductile-to-brittle behavior.[1] At temperatures above the transition temperature, the material deforms plastically; at temperatures less than the transition temperature, the material fractures in a brittle manner without significant plastic deformation. This transition temperature has been shown to be lowered by (1) cold working, (2) decreasing grain size, (3) increasing purity, (4) decreasing strain rate and degree of triaxiality of imposed stress, and (5) environments that increase the surface energy of the metal. It has also been found that this decrease in fracture ductility with decreasing temperature is accompanied by a sharp increase in the yield strength of the metal.[1] Although much of the present knowledge of the ductile-brittle transition in body-centered cubic metals has come from studies of alpha iron, Owen and Hull[2] have shown that under suitable conditions all refractory metals will undergo a transition in fracture behavior. Chromium, molybdenum, and tungsten (group VIA) are more susceptible to brittle fracture than are vanadium, niobium, and tantalum (group VA).

The postirradiation properties of pure molybdenum and niobium have received some attention, with molybdenum having been studied the more extensively. Bruch, McHugh, and Hockenbury [3] studied the properties of two

heats of molybdenum containing 0.061 and 0.066 wt % C after irradiation at 90°C to an integrated dose of 1.9 to 5.9 × 10^{20} thermal nvt. The transition temperature was increased from −30 to +70°C. The hardness was increased to about 35 BHN units by the irradiation. A further set of specimens were irradiated at 400°C to an integrated dose of 3 × 10^{20} thermal nvt (3 × 10^{19} fast nvt). These specimens increased in hardness indicating that defects were being introduced by irradiation at a rate greater than the annealing rate at 400°C.

Makin and Gillies[4] investigated the behavior of pure molybdenum in the form of 0.040-in.-diam wire after irradiation at 100°C to a dose of 5 × 10^{19} thermal nvt (ratio of thermal to fast 1:1). Bend tests were run and it was found that the transition temperature was increased from −136 to −73°C. Tensile tests were run over the temperature range of 20 to 200°C, and it was concluded that the strength was increased and the ductility slightly decreased. Since quite large yield points were produced by postirradiation annealing at 200°C, it was concluded that the defects were mobile at this temperature. The important defects were throught to be vacancies. This was based on experimental observations by Kinchin and Thompson[5] that cold-worked molybdenum after exposure to a radiation field exhibited a recovery stage at 150°C. The activation energy of the process was found to be 1.3 ev and was attributed to vacancy migration.

Johnson[6] examined the influence of irradiation on the tensile properties of molybdenum specimens having different grain sizes. The intent was to determine whether both or only one of the parameters in the Petch equation[7] ($\sigma_{ys} = \sigma_i + k_y d^{-1/2}$) were changed by irradiation. Hull and Mogford[8] had found that in EN-2 steels irradiated to 9 × 10^{18} nvt only σ_i was increased and k_y was not significantly affected. Johnson found that molybdenum

irradiated to 2×10^{19} fast nvt did not obey the Petch relationship and that the results could not be described by single values of σ_i and k_y. Neither could a satisfactory correlation be made based on the subgrain size. Chow[9] has cleared the picture somewhat by compiling the results for iron and iron-carbon alloys and showing that both of the Petch parameters are changed by irradiation. Changes in σ_i are produced at relatively low exposures, whereas changes in k_y are seen only at higher exposures. Increasing carbon content increases the exposure level necessary to cause a change in k_y.

Mastel et al.[10] have examined irradiated molybdenum foils containing 10, 150, and 450 ppm C. The foils were annealed at 1900°C and irradiated at 40°C to an integrated dose of 10^{19} fast nvt. Uniformly distributed defect clusters in the form of black spots 75 to 100 A in diameter were observed in all of the irradiated foils. Clearly resolved loops were observed only in foils containing more than 150 ppm C. It was found in deformed specimens that moving dislocations could sweep out the defects and leave defect-free channels. It was also reported that irradiation reduced the number of active slip systems and that slip occurred only on {110} and {112} in <111>.

Makin and Minter[11] investigated the postirradiation tensile properties of niobium after irradiation at 16°C to a dose of 1×10^{20} nvt (>0.6 Mev). Only a moderate increase in yield strength occurred immediately after irradition, but annealing in the temperature range of 125 to 175°C resulted in further strengthening. The activation energy associated with a yield drop was found to be 1.30 ±0.1 ev and was associated with condensation of vacancies on dislocations. Recovery of preirradiation properties began at 350°C and was completed after 1 hr at 600°C. The uniform elongation was significantly reduced by irradiation, although the reduction in area at the fracture remained large. This was due to the inability of the irradiated material to work harden.

Evans et al.[12] studied the influence of irradiation on the strength and ductility of high-purity niobium as a function of dose, grain size, and irradiation temperature. Two groups of specimens were irradiated to a dose of 9.1×10^{17} nvt (>1 Mev) at temperatures of 77 and 330°C. The yield strength was increased and the fracture ductility decreased, the effect being larger at the higher irradiation temperature. The data were found to correlate by the Petch equation with only σ_i being changed by irradiation. A third group of specimens was irradiated at 330°C to a dose of 2.5×10^{19} fast nvt. All of the specimens were brittle at room temperature following irradiation. The possibility of oxygen contamination was considered.

In the present study the postirradiation properties of Mo–0.5% Ti, Nb–1% Zr, and D-43 alloys were determined. The irradiation conditions will be described in detail. The Mo–0.5% Ti alloy was studied in the recrystallized condition; the Nb–1% Zr alloy was examined in the cold-worked, recrystallized, and welded conditions; and the D-43 alloy was studied in the annealed and cold-worked conditions. The materials were given various postirradiation anneals to determine the kinetics of annealing of the irradiation-induced defects. Bend testing was the technique used for evaluating the pre- and postirradiation properties of the specimens. Limited metallographic studies were also carried out.

EXPERIMENTAL DETAILS

The test materials used in this study are described in Table 1. All of the material was received in the form of sheet. The Mo–0.5% Ti alloy was in the warm-worked form and was annealed 1 hr at 1350°C prior to test. The Nb–1% Zr alloy sheet had been warm worked followed by 40% cold working. It was tested in both the cold-worked state and after annealing 1 hr at 1350°C.

Table 1. Test Materials

Description of Material	Chemical Analysis (wt %)											
	C	O	N	H	Mo	Nb	Ti	Zr	Ni	Si	Fe	W
0.050-in.-Mo–0.5% Ti-sheet	0.020	0.0018	0.0012	0.0003	base		0.470		0.001	0.001	0.001	
0.063-in.-Nb–1% Zr-sheet	0.011	0.0150	0.0055	0.0004		base		0.93				
0.050-in.-D-43-sheet	0.093	0.0032	0.0032	0.0008		base		0.95				9.4

welds were made in this material by fusing two strips of annealed Nb–1% Zr alloy together. The welds were made in an argon atmosphere and no filler metal was added. The details of the welding procedure have been described previously.[13] The welds were oriented so that the bend was made normal to the weld axis. The D-43 alloy had been stress relieved at 1200°C prior to delivery. All of the material was annealed at 1350°C and was tested both in the annealed state and after being cold worked 20%.

The test specimens were nominally 1.188 × 0.750 in. The 1.188-in. dimension was sheared to ±0.033 in. and the 0.750-in. dimension was ground to ±0.001 in. The bend was made along the 0.750-in. dimension.

The specimens were irradiated in the poolside of the Oak Ridge Research Reactor (ORR). In experiment 101 the range of thermal flux in the facility was 4.0 to 1.5×10^{13} neutrons/cm^2·sec and the fast flux (>2.9 Mev) was 2.5 to 0.6×10^{12} neutrons/cm^2·sec. The integrated thermal dose accrued by the specimens ranged from 1.8 to 0.54×10^{20} nvt and the fast dose (>2.9 Mev) ranged from 1.1 to 0.27×10^{19}. The irradiation temperature was from 60 to 82°C. In experiment 107 the thermal flux ranged from 8.0 to 2.0×10^{13} neutrons/cm^2·sec and the fast flux (>2.9 Mev) from 8.0 to 0.80×10^{12} The integrated flux ranged from 6.4 to 1.6×10^{20} thermal and 6.4 to 0.64×10^{19} fast. The irradiation temperature varied from 157 to 60°C.

The specimens were tested in the bend fixture shown in Fig. 1. The specimen is supported by two pins 3/32 in. in diameter and 3/4 in. between centers. The load is applied along a line midway between the two support rods by a ram having a 3/32-in. radius. The specimen is deflected at a rate of 1/2 in./min. The temperature is measured by a Copper/Constantan thermocouple which passes through the thermocouple well into the block on which the specimen is tested. Figure 2 shows the bend apparatus attached to the

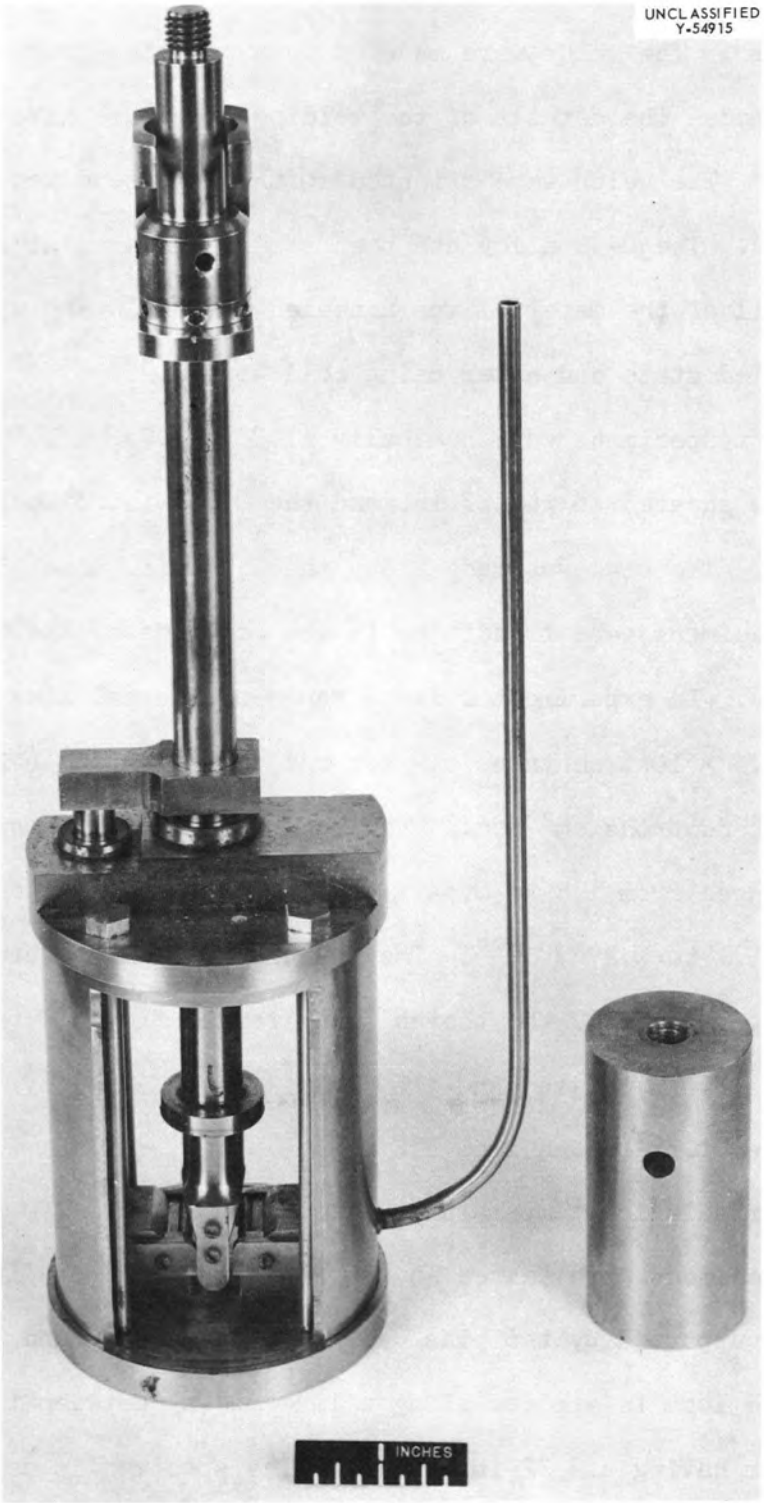

Fig. 1. Fixture for Performing Bend Tests.

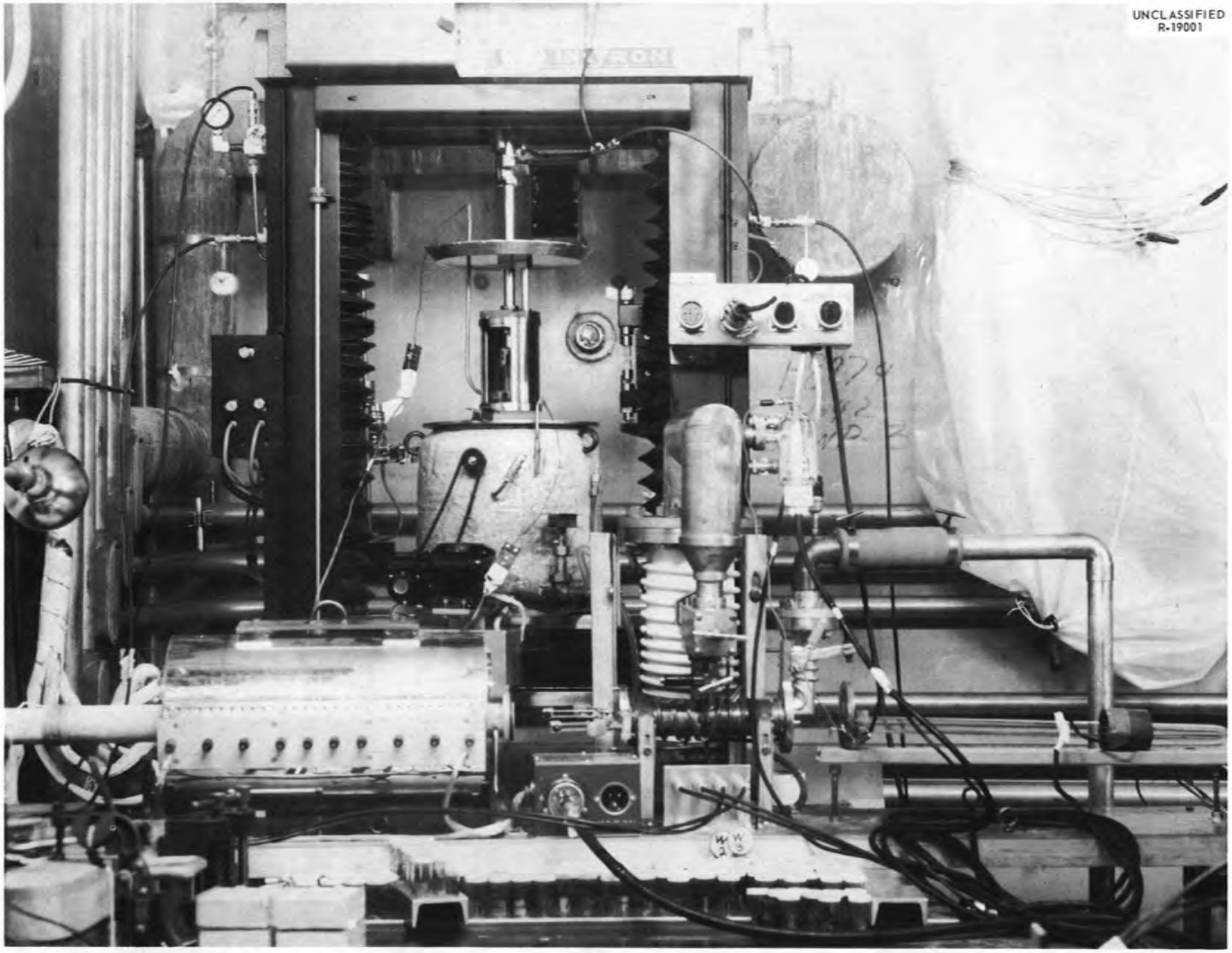

Fig. 2. Background: Bend Fixture Assembled for Running Tests; Foreground: Vacuum Furnace for Postirradiation Annealing.

Instron machine along with the vessel that provided the controlled temperature bath. As shown, the entire apparatus "opened up" to allow easy access to the testing stage. After a specimen was put in place, the crosshead was moved up until the plunger rested on the specimen, but no additional load had been applied. The temperature was adjusted before the test was started. For heating, the vessel was filled with Dow Corning-550 fluid which was heated by Calrod heaters welded to the side of the bath container. The motor and pulleys were attached to a stirrer that equalized the temperature distribution. For moderate cooling the vessel was filled with ethyl alcohol and dry ice was added. Further cooling was obtained by using liquid nitrogen as a bath.

The furnace and associated vacuum equipment in front of the tensile machine were used for postirradiation annealing. The system was capable of a vacuum of 5×10^{-8} torr at 1000°C. The upper temperature limit of 1200°C was imposed by the furnace.

The results of the bend tests are recorded in terms of load-deflection curves. These are analyzed by standard equations based on elastic theory and presented by Marin.[14] The yield strength used in this paper is defined by the intersection of the experimental curve with a line offset by 0.025 in. along the deflection axis and parallel to the elastic portion of the curve. The maximum deflection used was 0.375 in., which bends the specimen slightly in excess of 100°.

The metallography was carried out by fairly standard techniques. Syntron polishing was used and the details have been described previously.[15] The hardness measurements were made on a Kentron machine with a load of 500 g. Five impressions were made and both the average and hardness range are given for each specimen.

RESULTS

Mo—0.5% Ti Alloy

The bend properties of Mo—0.5% Ti alloy after annealing 1 hr at 1350°C in vacuum are illustrated in Fig. 3. The transition temperature is increased slightly as the temperature of the second anneal (equivalent to postirradiation anneal) is increased. After irradiation in ORR experiment 101, the transition temperature was increased significantly (Fig. 4). The transition temperature in the as-irradiated state was 138°C as compared with 19°C for the as-annealed material. Postirradiation anneals of 1 hr at 500, 1000, and 1200°C progressively decreased the transition temperature. However, the preirradiation properties are not recovered after annealing at 1200°C. Results of a similar series of tests run on specimens irradiated in ORR experiment 107 are illustrated in Fig. 5. Although the experimental scatter makes the results difficult to analyze, it appears that the somewhat higher flux and irradiation temperature of ORR experiment 107 did not result in a significantly different transition temperature than did the conditions of experiment 101. Although the ductility was reduced by irradiation, the yield strength was not changed significantly. (See Fig. 6)

Several of the test specimens were mounted and examined metallographically. Hardness measurements were made on most of the specimens and these results are given in Table 2. Irradiation in experiments 101 and 107 increased the hardness from its base value of 174 DPH to 230 DPH and 240 DPH, respectively. Postirradiation annealing at 500°C slightly increased the hardness, but this is partially offset by an increase in hardness of the unirradiated material. Hence it seems that the hardness recovers very little by annealing at 500°C. Annealing

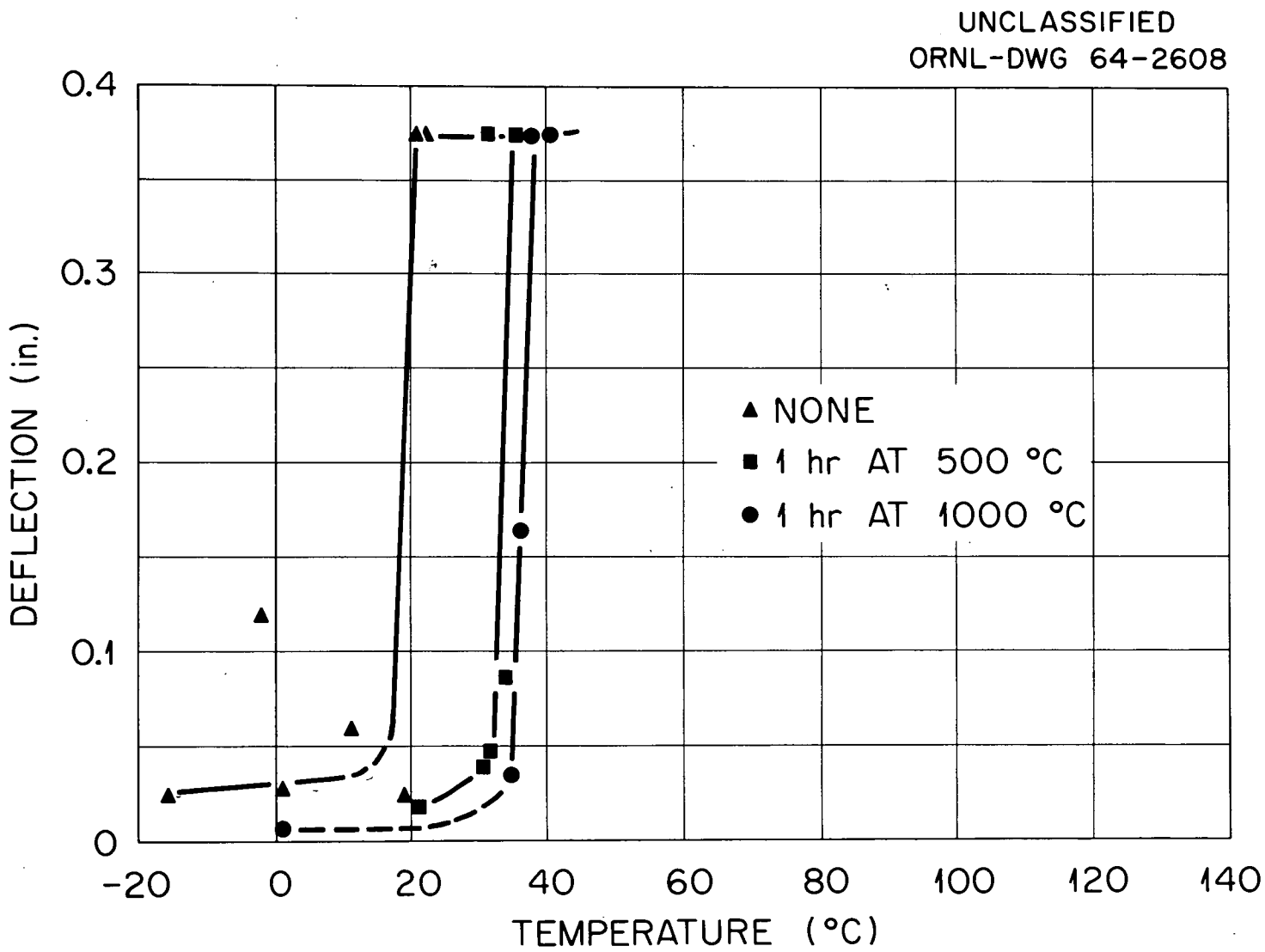

Fig. 3. Bend Properties of Recrystallized Unirradiated Mo–0.5% Ti.

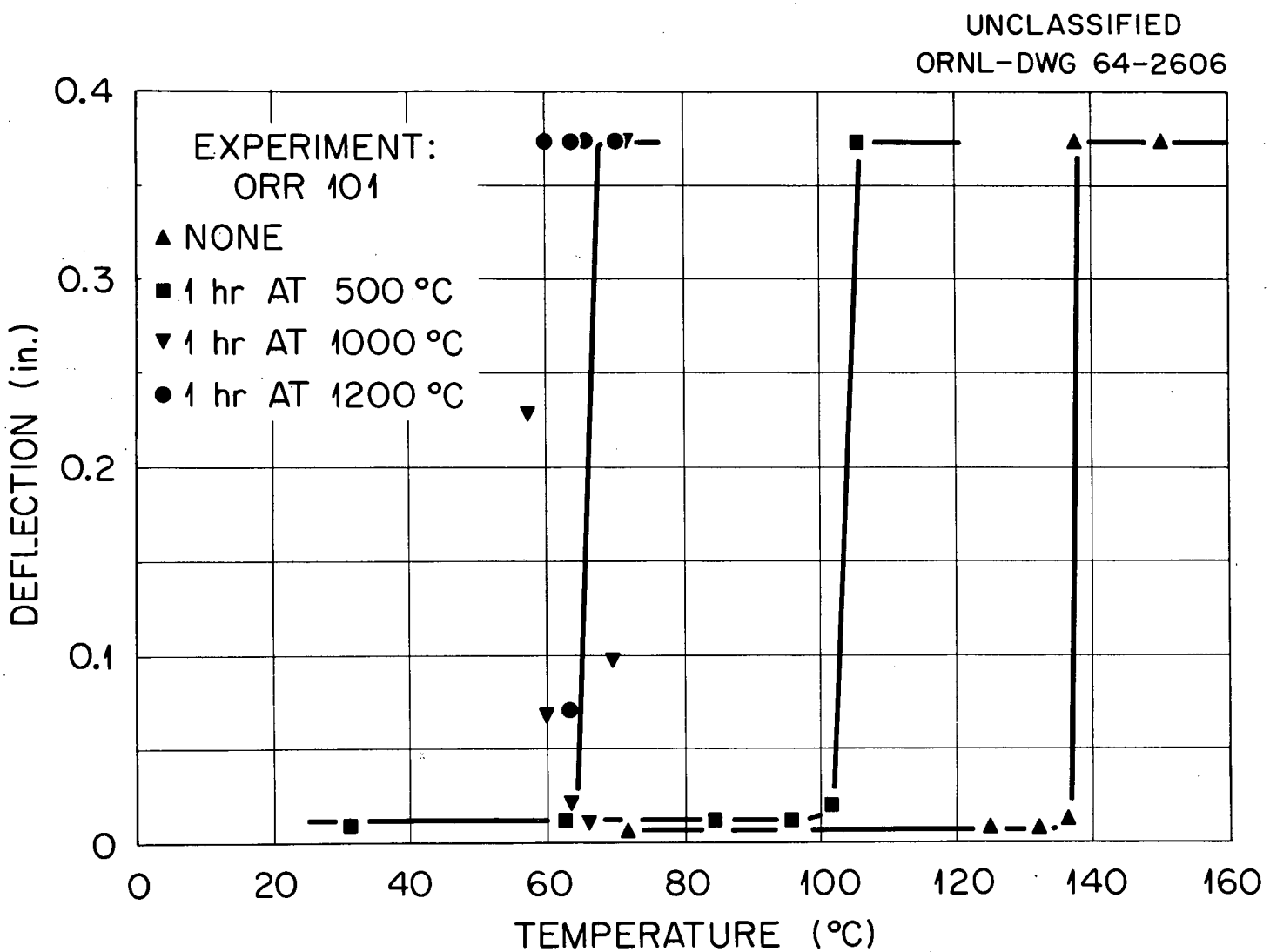

Fig. 4. Bend Properties of Recrystallized Mo–0.5% Ti After Irradiation.

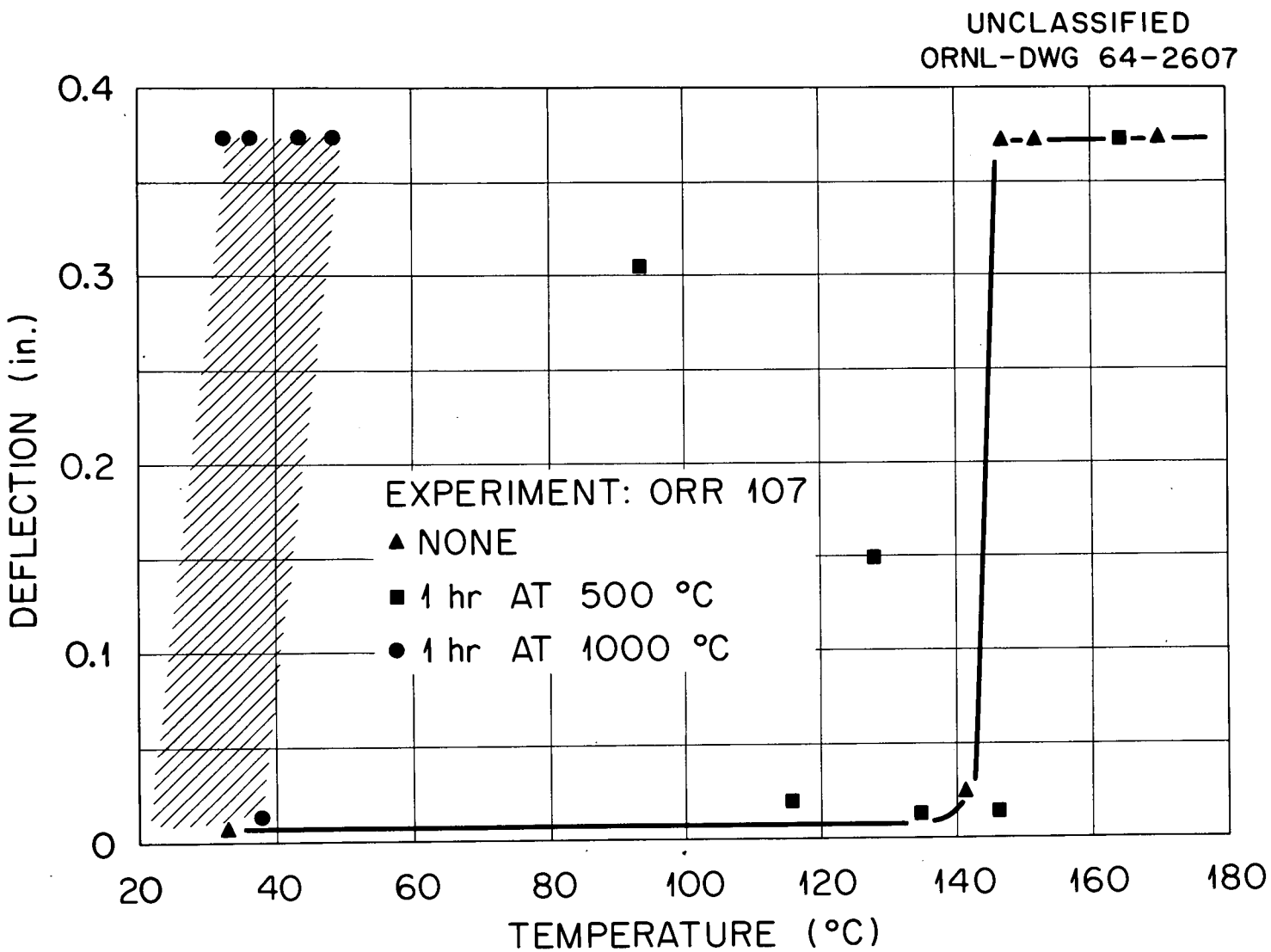

Fig. 5. Bend Properties of Recrystallized Mo–0.5% Ti After Irradiation.

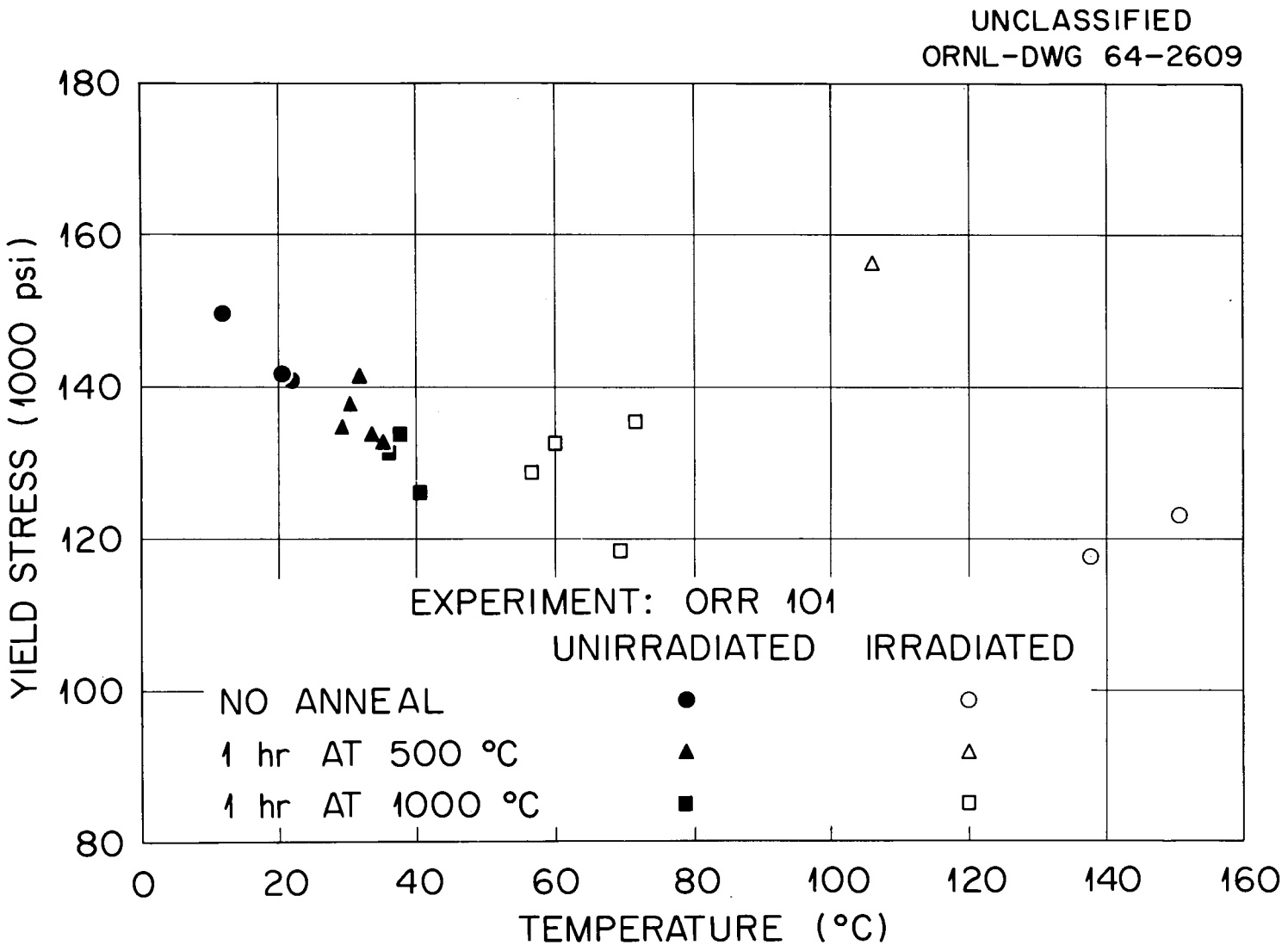

Fig. 6. Bend Properties of Recrystallized Mo-0.5% Ti Before and After Irradiation.

Table 2. Results of Hardness Measurements on the Mo–0.5% Ti Alloy

Post Irradiation Anneal	Hardness, DPH, Range (av)		
	Unirradiated	Irradiated, ORR 101	Irradiated, ORR 107
none	168–178 (174)	219–250 (230)	226–258 (240)
1 hr at 500°C	171–182 (177)	229–239 (233)	226–234 (228)
1 hr at 1000°C	175–182 (178)	189–210 (196)	
1 hr at 1200°C		171–182 (180)	

at 1000°C brings about a large decrease in hardness, with 1200°C causing a further hardness reduction. The fractures of unirradiated and irradiated specimens tested just below the transition temperature are shown in Figs. 7a and 8a, respectively. Both of the fractures are transgranular and there are no significant differences in microstructure observable at 1000 magnification. However, a difference was noted concerning deformation around hardness impressions. This is illustrated in Figs. 7b and 8b. The plastic strain imposed by the indenter was absorbed by the unirradiated material without visible slip-band formation around the impression (Fig. 7b), whereas very coarse slip markings are present around the impression in the irradiated material (Fig. 8b)

Nb–1% Zr Alloy

Since the Nb–1% Zr alloy specimens were found to be ductile at the lowest attainable test temperature, the results have been analyzed in terms of the yield strength. Figure 9 shows a comparison of the yield strength of Nb–1% Zr before and after irradiation in experiment 101. The room-temperature yield strength changes from 61,500 to 93,500, a change of 52%, during irradiation. Postirradiation anneals of 1 hr at 300 and 500°C both produce further strengthening with a resultant room-temperature yield strength of 103,000 psi. A postirradiation anneal of 1 hr at 700°C reduces the strength, and annealing at 1000°C returns the material to its preirradiation strength.

The results obtained from a similar series of specimens from ORR 107 show the same trends, as shown in Fig. 10. However, the conditions of experiment 107 produced greater strengthening than did those of experiment 101.

The microhardnesses of several specimens were measured and are presented in Table 3. The hardness values indicate the same trends as did the yield strengths. Irradiation under the conditions of experiments 101 and 107

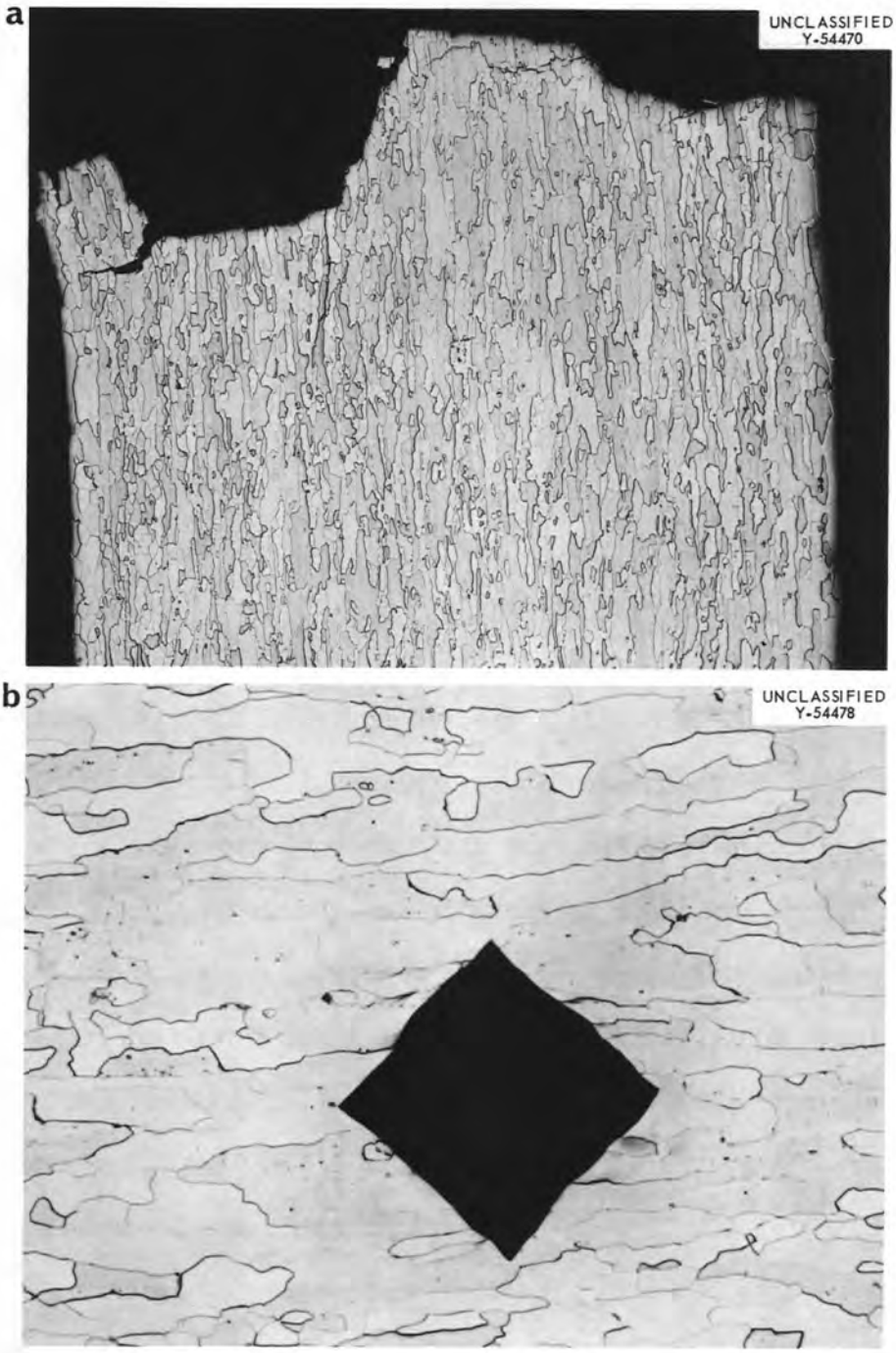

Fig. 7. (a) Photomicrograph of Unirradiated Mo–0.5% Ti Tested After Annealing 1 hr at 1350°C; Tested at 19°C. Etchant: NH_4OH, H_2O. 100×. (b) Hardness impression in same specimen. Etchant: NH_4OH, H_2O. 500×. Reduced 17%.

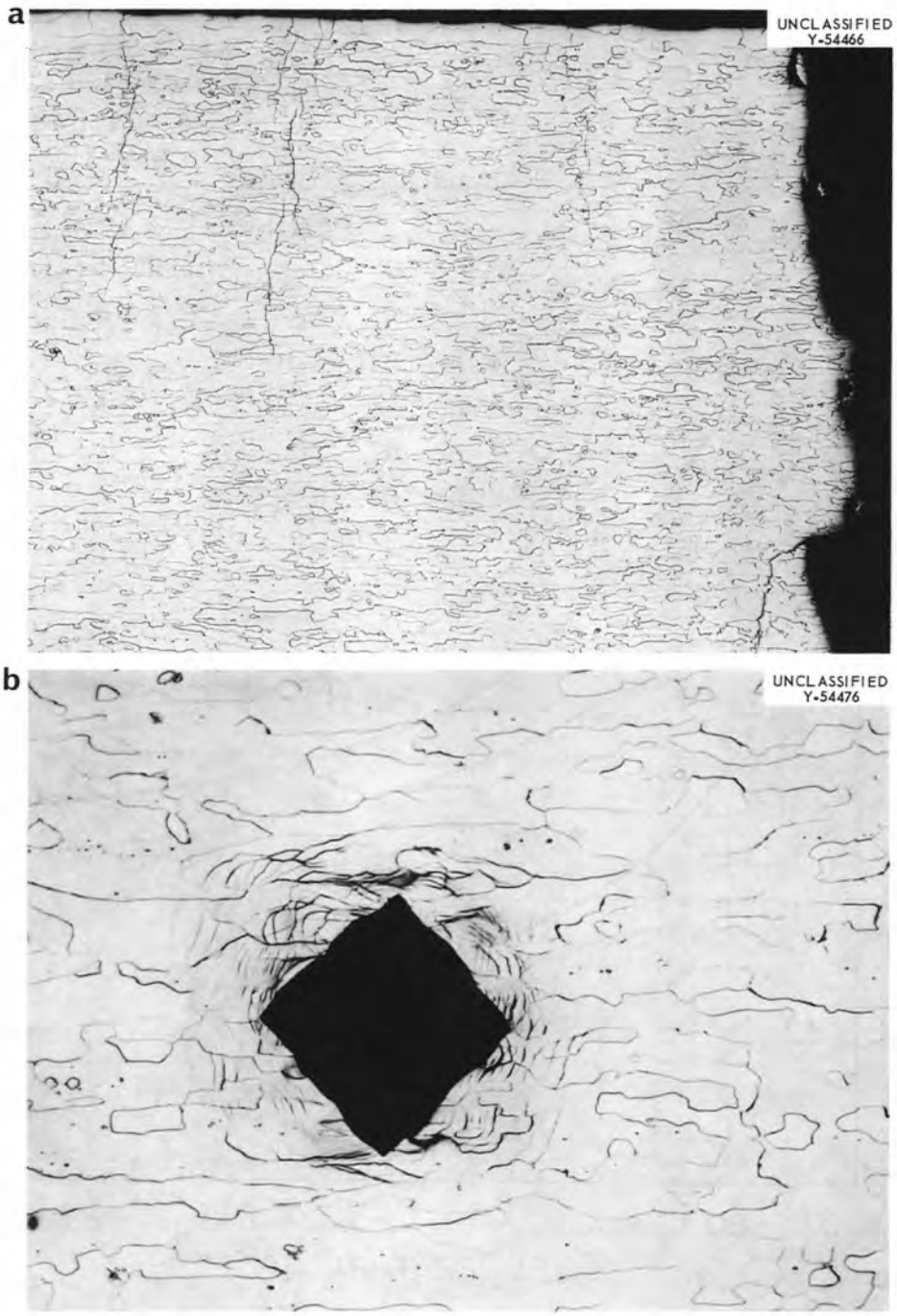

Fig. 8. (a) Photomicrograph of Mo–0.5% Ti Specimen Irradiated in ORR Experiment 101 and Tested at 125°C. Etchant: NH_4OH, H_2O. 100×. (b) Hardness impression in same specimen. Etchant: NH_4OH, H_2O. 500×. Reduced 21%.

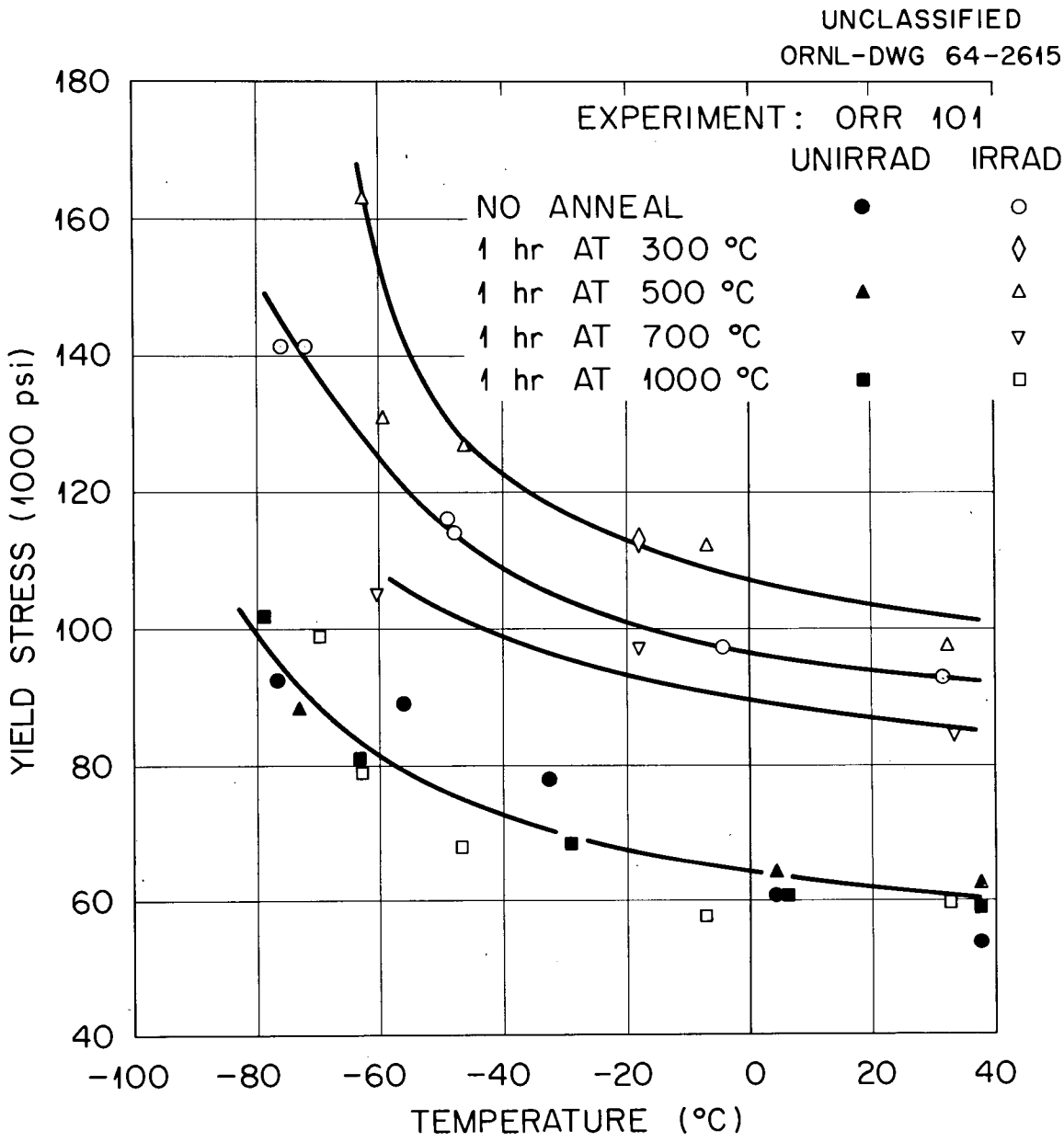

Fig. 9. Bend Properties of Recrystallized Nb–1% Zr Before and After Irradiation.

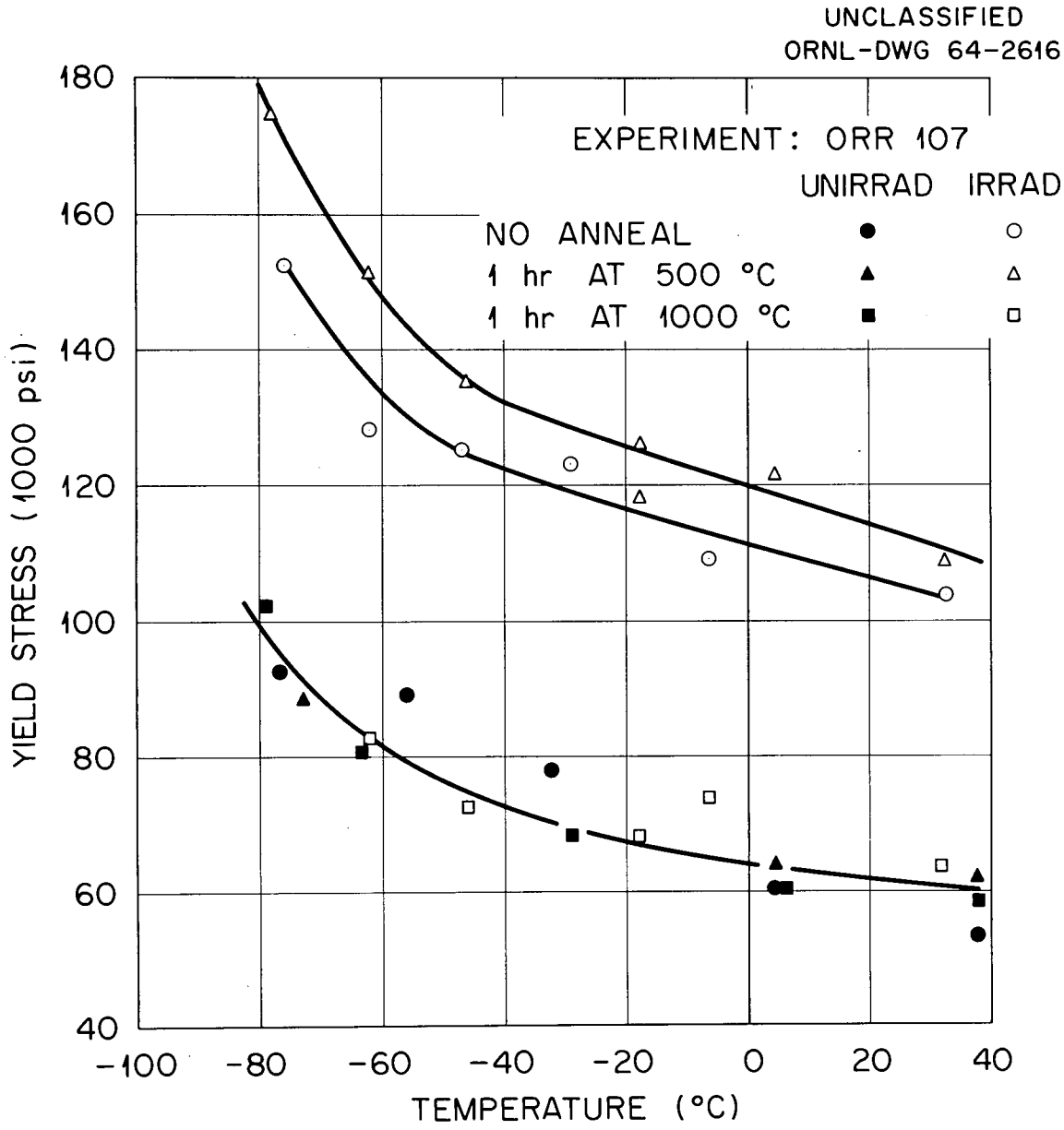

Fig. 10. Bend Properties of Recrystallized Nb–1% Zr Before and After Irradiation.

Table 3. Results of Hardness Measurements on the Nb–1% Zr Alloy

Post Irradiation Anneal	Hardness, DPH, Range (av)		
	Unirradiated	Irradiated, ORR 101	Irradiated, ORR 107
none	101–105 (103)	131–135 (133)	141–141 (141)
1 hr at 300°C		141–148 (146)	
1 hr at 500°C	95–105 (100)	152–160 (156)	
1 hr at 700°C		128–131 (130)	
1 hr at 1000°C	95–107 (99)	117–122 (120)	

produced changes in the hardness from 103 to 133 and 141, respectively. Postirradiation annealing at 300 and 500°C brought about further hardening. Annealing at 700°C reduced the hardness and 1 hr at 1000°C almost returned the material to its preirradiation hardness.

Several of the specimens were examined metallographically. No significant differences were noted in the test specimens irradiated and those unirradiated. Figures 11 (specimen No. 65) and 12 (specimen No. 2) illustrate the bend areas of unirradiated and irradiated material, respectively.

The as-received cold-worked Nb–1% Zr alloy was irradiated in experiment 107 and the results of tests on this material are given in Fig. 13. The room-temperature yield strength was increased from 108,000 to 136,000, an increase of 22%. A postirradiation anneal of 1 hr at 500°C brought about a further increase of the yield strength to 148,000 psi. Annealing for 1 hr at 1000°C reduced the strength significantly to a level comparable to the control material. The hardness data in Table 4 reflect a hardness increase of the cold-worked material during irradiation of 26 points from 145 to 171 DPH. Annealing of the unirradiated cold-worked material at 500 and 1000°C resulted in substantial decreases in the hardness. Figures 14 and 15 compare the microstructures of the irradiated and unirradiated bend specimens after testing.

Several welded Nb–1% Zr alloy specimens were irradiated in experiment 107 and tested to determine the influence of neutron irradiation. One series of specimens was annealed 1 hr at 1350°C after welding, and the other series was irradiated in the as-welded condition. The results of tests on the as-welded series are given in Fig. 16. The properties of this material both before and after irradiation are quite similar to that of the base metal (see Fig. 10). Notable exceptions are that the weld specimens are slightly stronger than the base metal, the weld specimens were not ductile at −196°C, and the preirradiatic

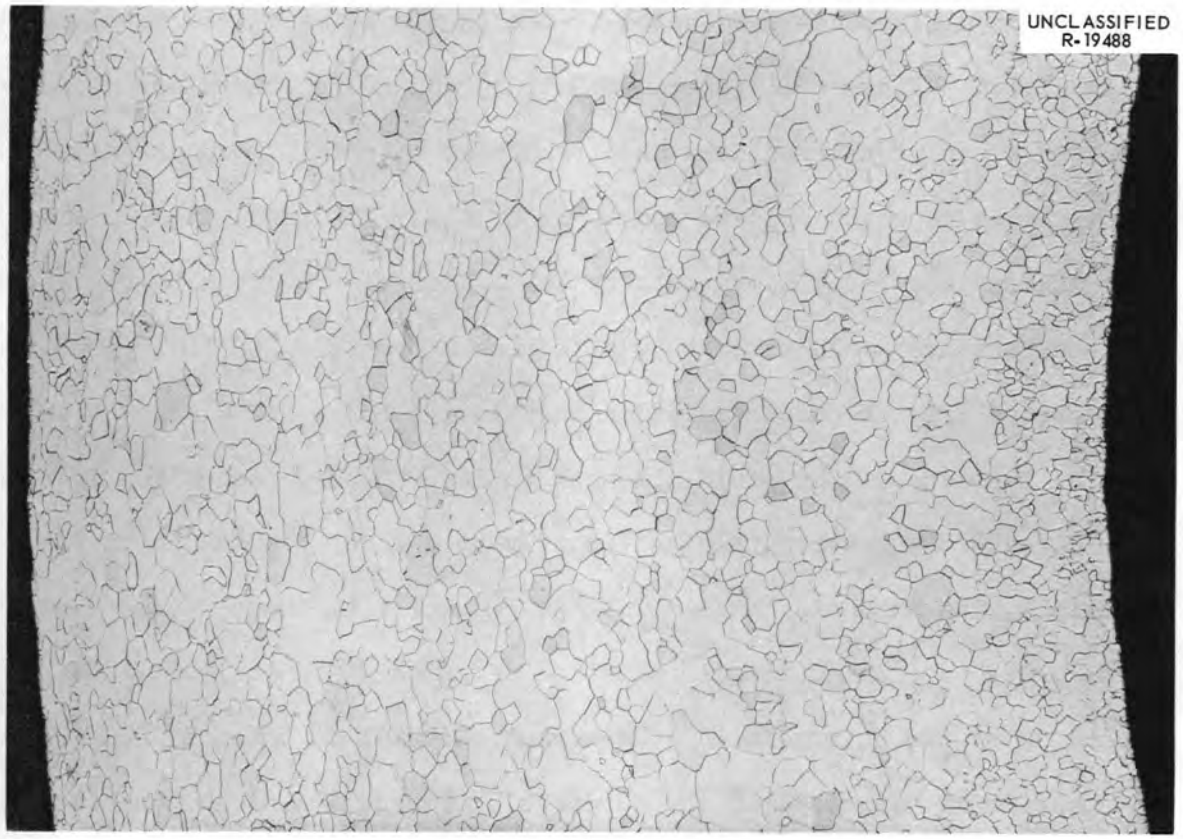

Fig. 11. Microstructure of a Nb–1% Zr Specimen Annealed 1 hr at 1350°C and Tested at 38°C. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×. Reduced 5%.

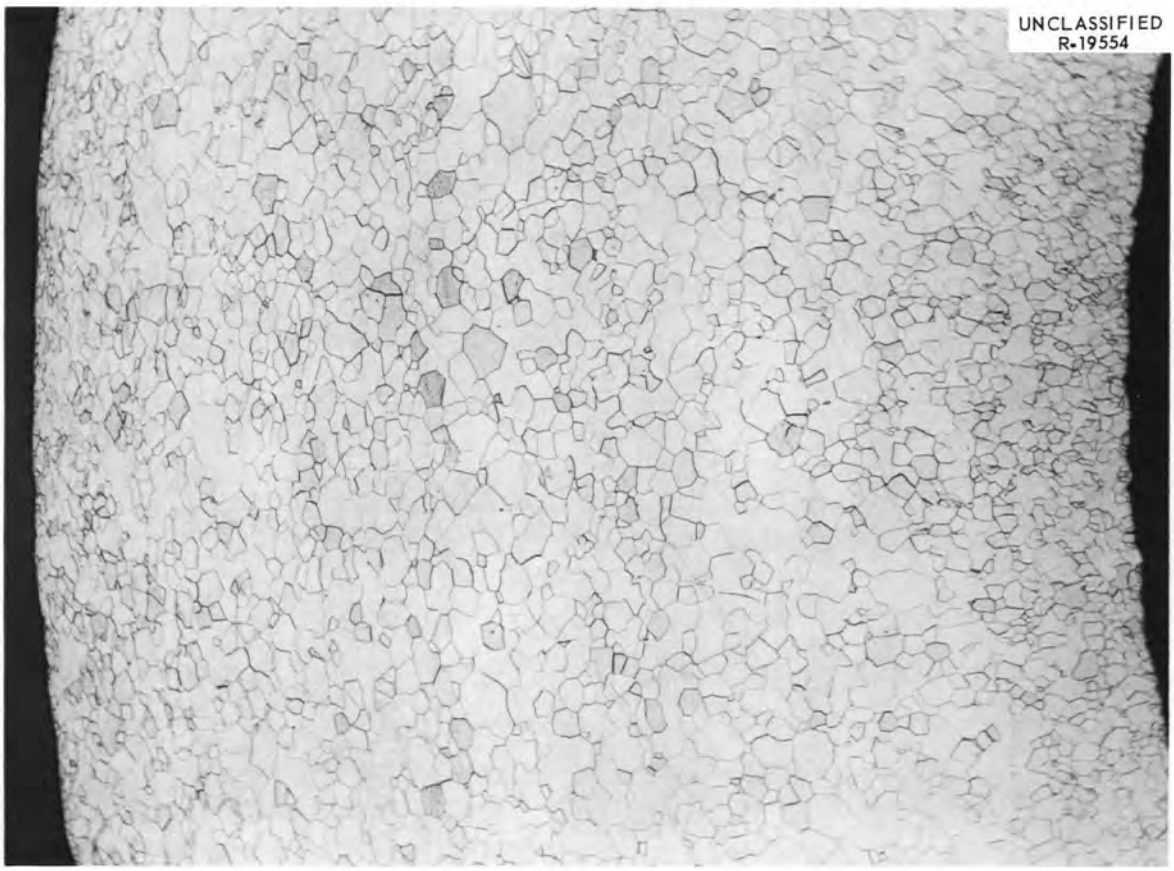

Fig. 12. Microstructure of a Nb–1% Zr Specimen Annealed 1 hr at 1350°C, Irradiated in Experiment 101, and Tested at 31°C. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×. Reduced 4%.

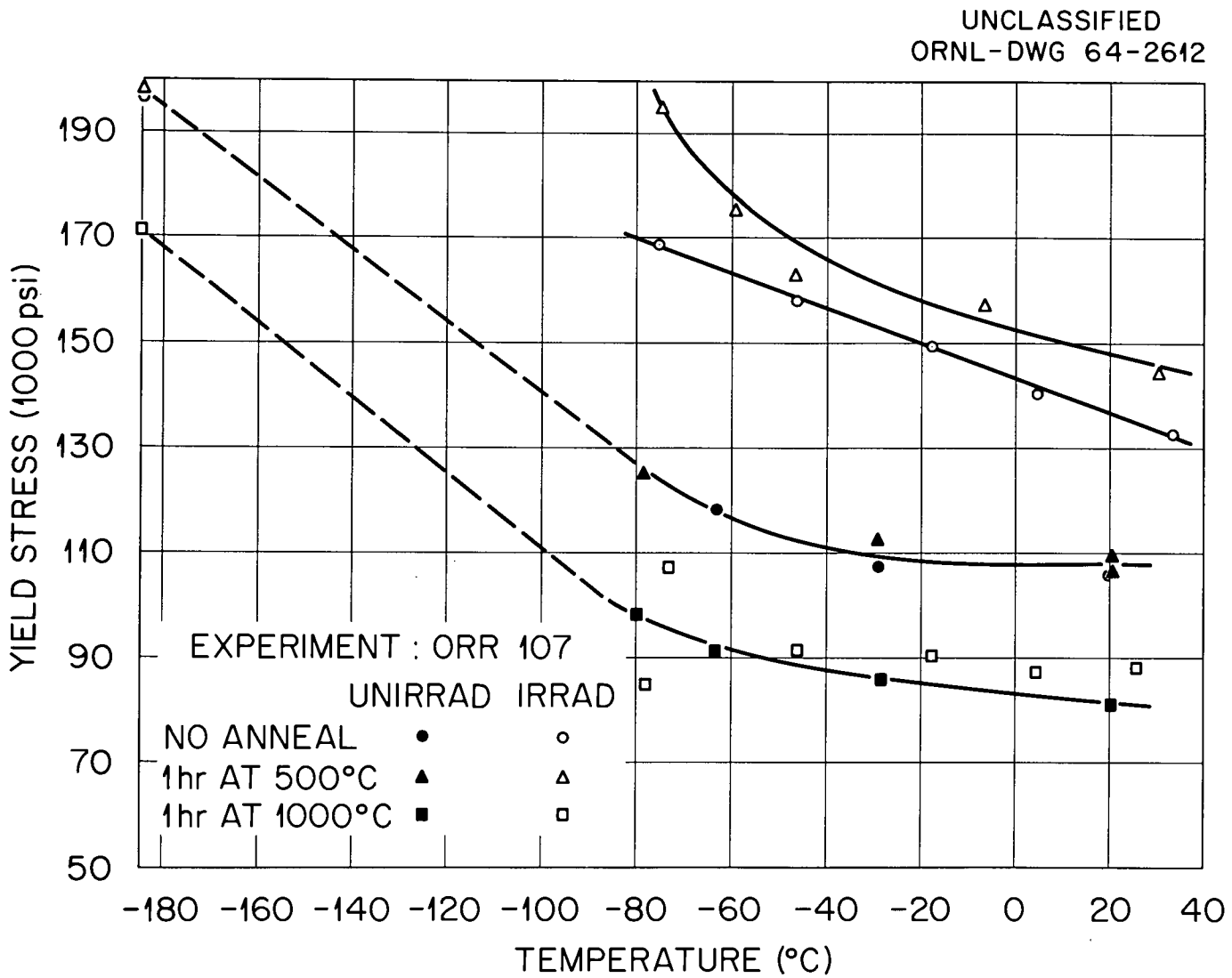

Fig. 13. Bend Properties of Cold-Worked Nb–1% Zr Before and After Irradiation.

Table 4. Results of Hardness Measurements on Cold-Worked Nb–1% Zr Alloy

Post Irradiation Anneal	Hardness, DPH, Range (av)	
	Unirradiated	Irradiated
none	141–152 (145)	164–173 (171)
1 hr at 500°C	134–141 (139)	
1 hr at 1000°C	117–122 (121)	

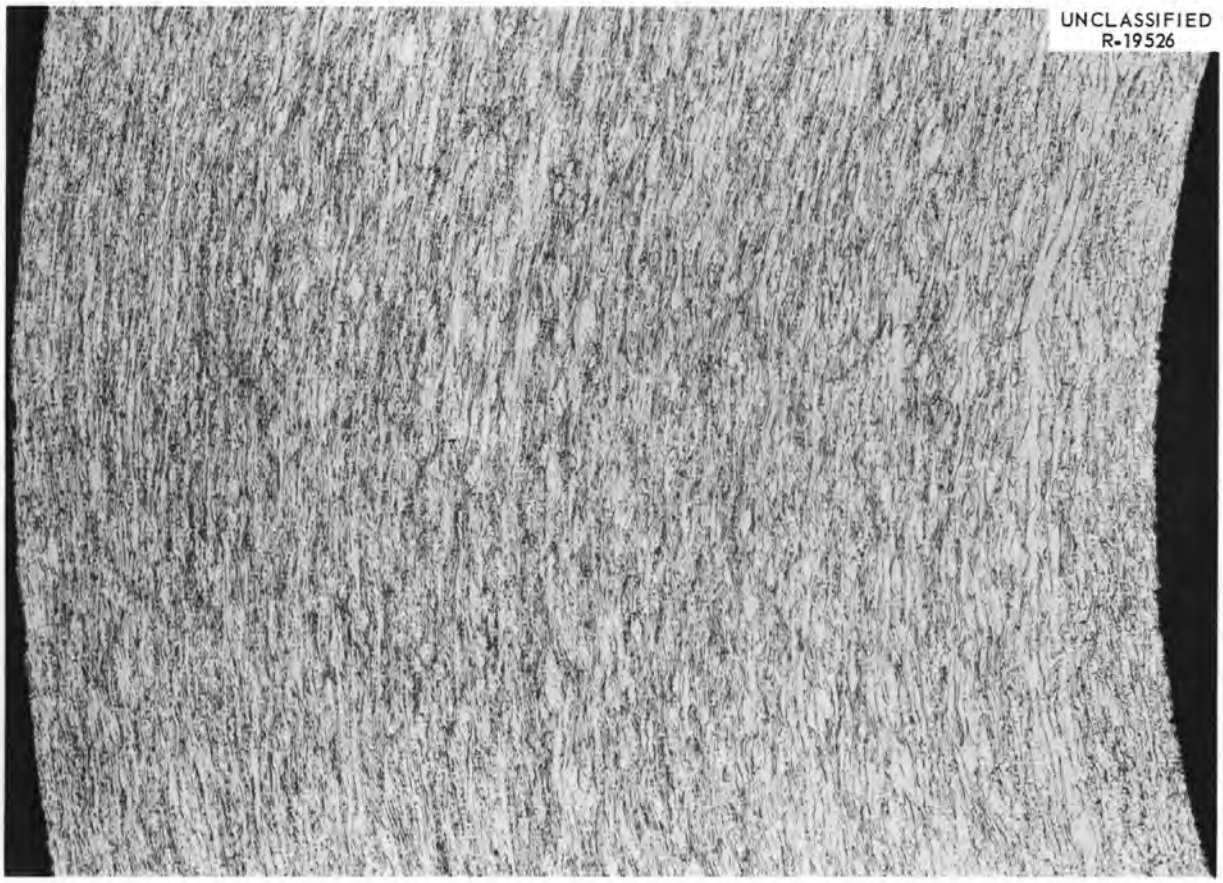

Fig. 14. Photomicrograph of the Bend Area of a Nb–1% Zr Specimen (No. 218) Tested in the Cold-Worked Condition; Tested at 20°C. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×.

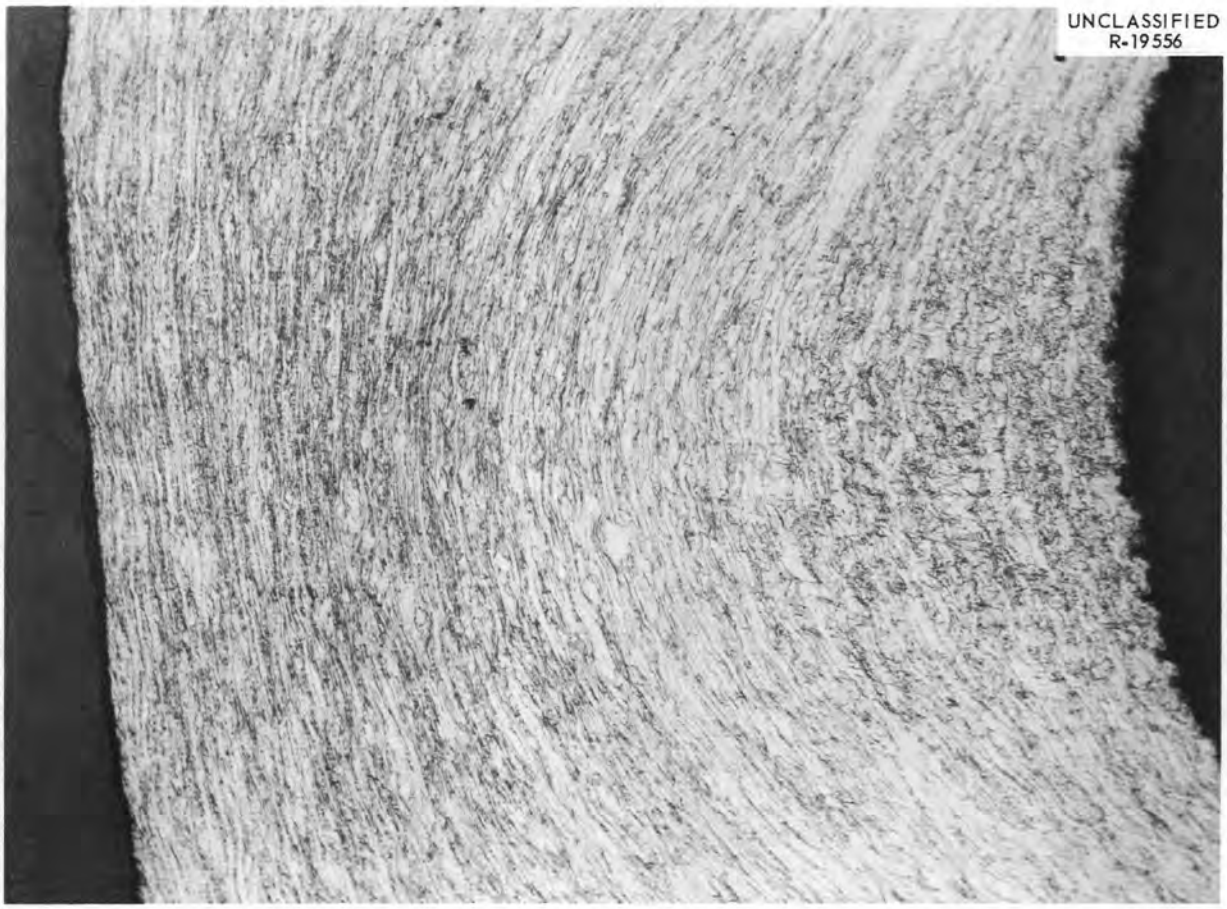

Fig. 15. Photomicrograph of the Bend Area of a Cold-Worked Nb–1% Zr Specimen (No. 196) After Irradiation in Experiment 107; Tested at 33°C. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×.

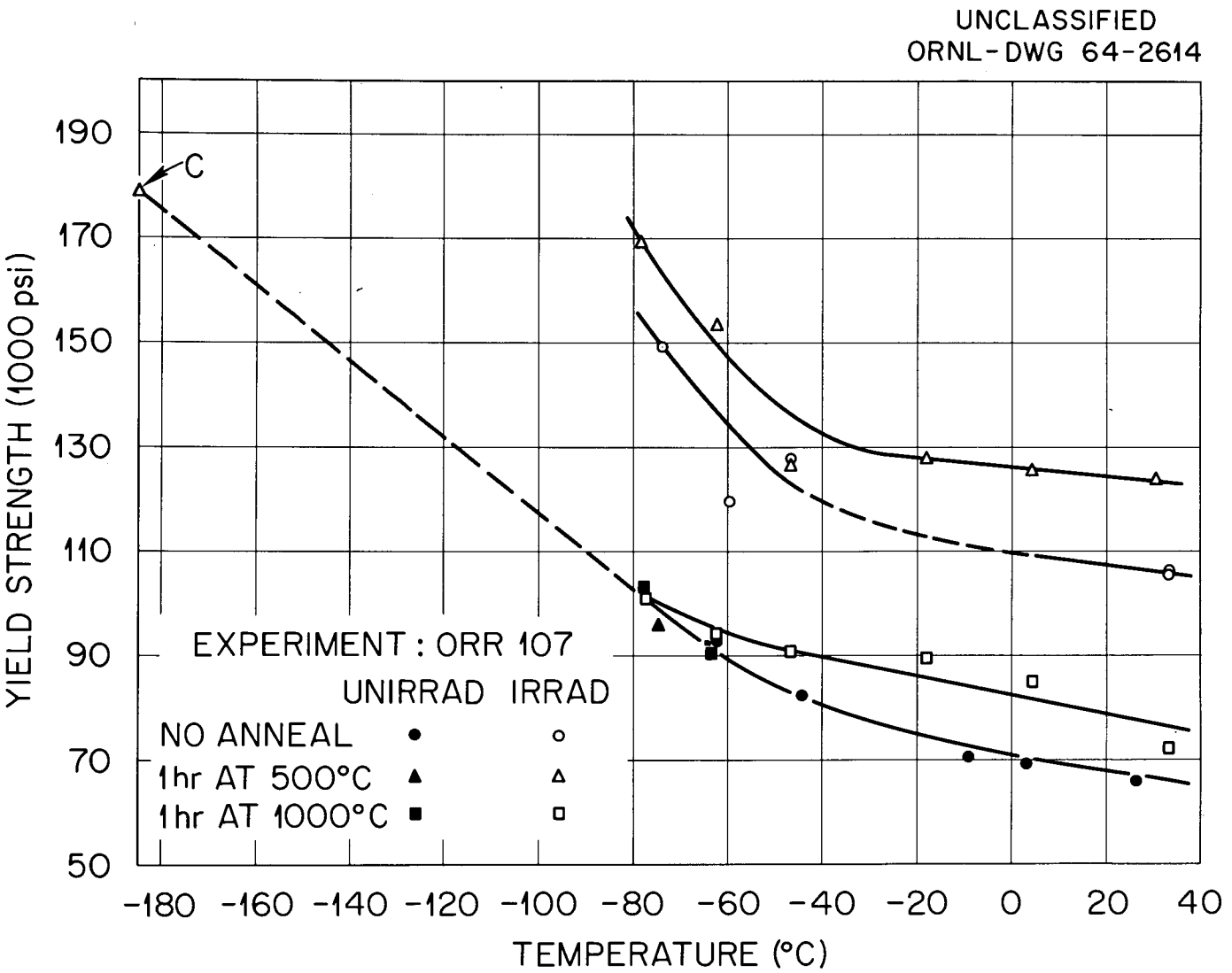

Fig. 16. Bend Properties of Welded Nb–1% Zr Before and After Irradiation.

properties are not completely recovered by annealing 1 hr at 1000°C. The series of specimens annealed at 1350°C after welding (Fig. 17) was found to be weaker than the unannealed material (Fig. 16) and quite comparable in strength to the base metal (Fig. 10). However, the strength of the material after irradiation was similar for the annealed and unannealed welds and the base metal.

Hardness measurements were made on several of the weld specimens and are listed in Table 5. The weld metal was slightly harder than the base metal, possibly due to slight contamination during welding. Both the weld and base metal increased in hardness during irradiation by 30 and 39 points, respectively; both values are comparable with that observed for the base metal in unwelded samples (see Table 3). Hardness measurements on the weld metal of unirradiated specimens showed an increase with increasing annealing temperature, indicative of possible aging.

The microstructure of a specimen welded and annealed 1 hr at 1350°C and tested at 33°C is shown in Fig. 18. The grain size of the weld metal is quite large. Figure 19 shows the weld and base metal in the deformed area of a weld specimen tested at −196°C. Note that numerous deformation twins have formed near the crack. Figure 20 shows the structure of the weld metal in a specimen that was annealed 1 hr at 1000°C. The fine precipitate is a further indication of aging.

D-43 Alloy

The alloy D-43 was studied in the annealed and cold-worked conditions. The results of a series of tests on the annealed material are given in Fig. 21. The room-temperature yield strength is increased by irradiation from 117,000

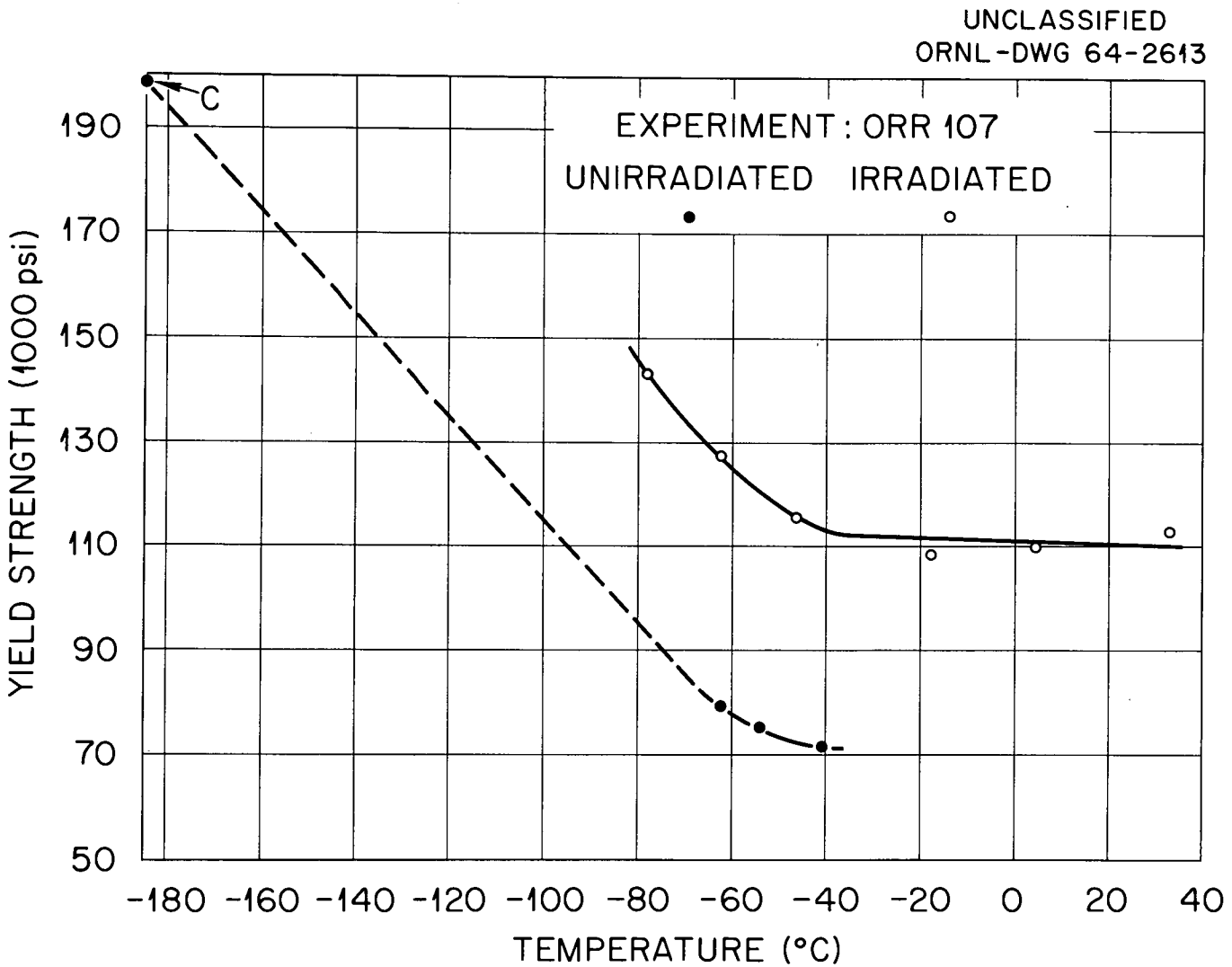

Fig. 17. Bend Properties of Welded Nb–1% Zr Before and After Irradiation; Annealed at 1350°C After Welding.

Table 5. Results of Hardness Measurements on Welded Nb–1% Zr Alloy

Post Irradiation Anneal	Hardness, DPH, Range (av)			
	Unirradiated		Irradiated	
	Weld Metal	Base Metal	Weld Metal	Base Metal
none[a]	114–117 (115)	105–110 (107)	141–152 (145)	144–148 (146)
none	110–117 (114)	107–112 (110)		
1 hr at 500°C	122–125 (124)	107–110 (109)		
1 hr at 1000°C	141–148 (145)	114–116 (115)		

[a]This material annealed 1 hr at 1350°C after welding. Other material tested in as-welded condition.

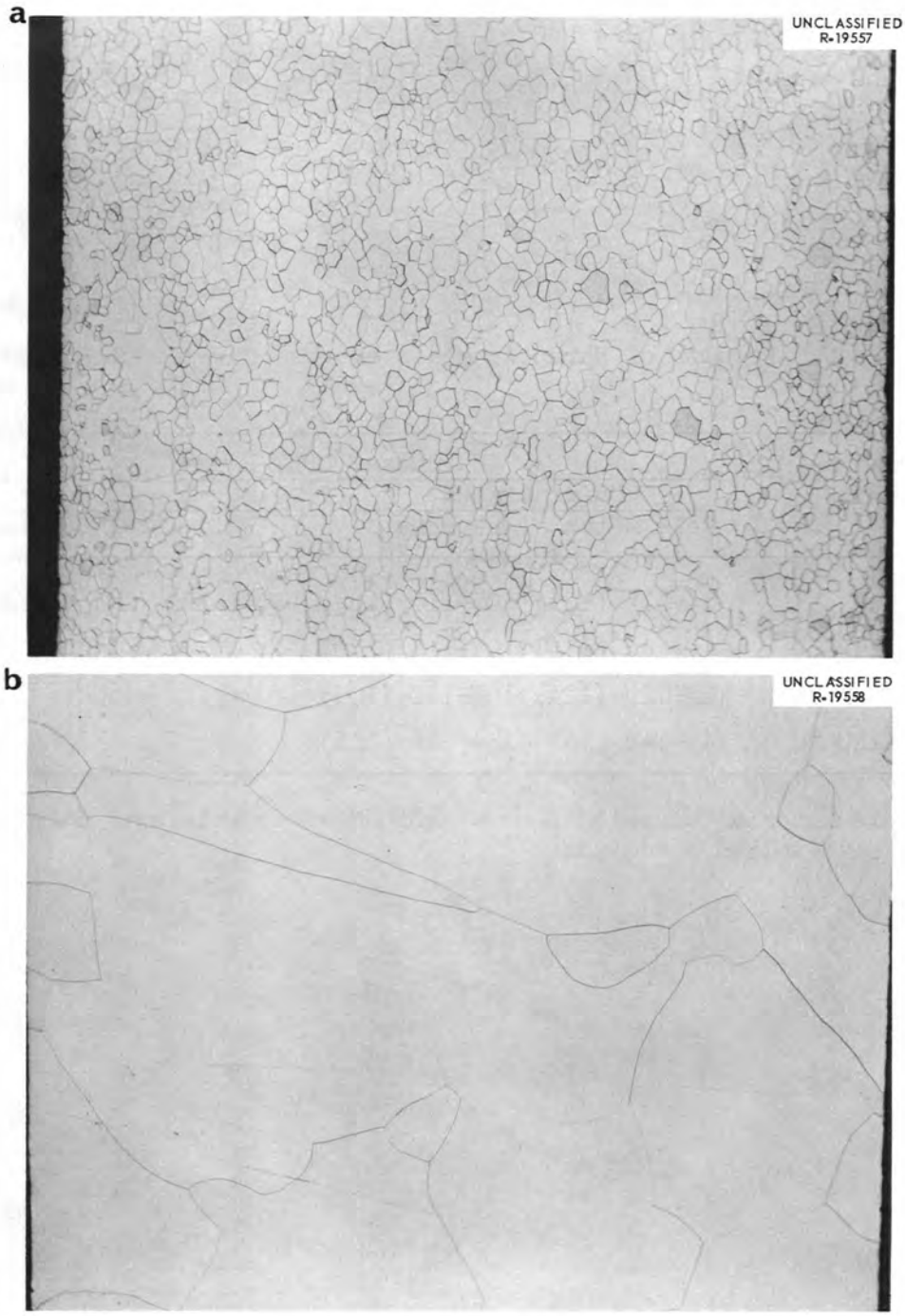

Fig. 18. Microstructure of Nb–1% Zr Weld Specimen (No. 154) Tested After Irradiation in Experiment 107; Annealed 1 hr at 1350°C After Welding; Tested at −33°C. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×. (a) Base metal. (b) Weld metal. Reduced 24%.

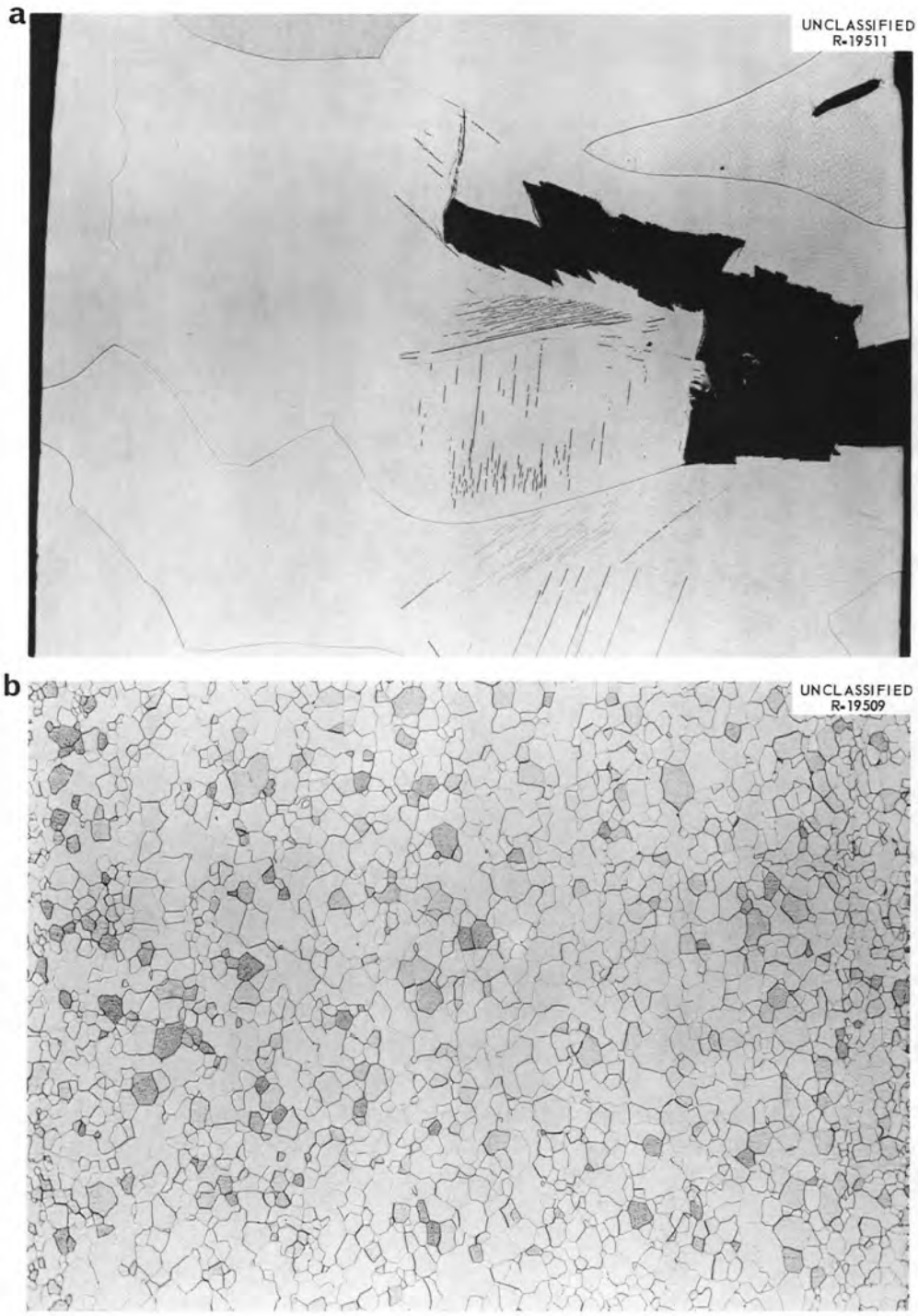

Fig. 19. Microstructure of Welded Nb–1% Zr Specimen (No. 169) After Testing at −196°C; Annealed 1 hr at 500°C Prior to Testing. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×. (a) Base metal. (b) Weld metal. Reduced 19%.

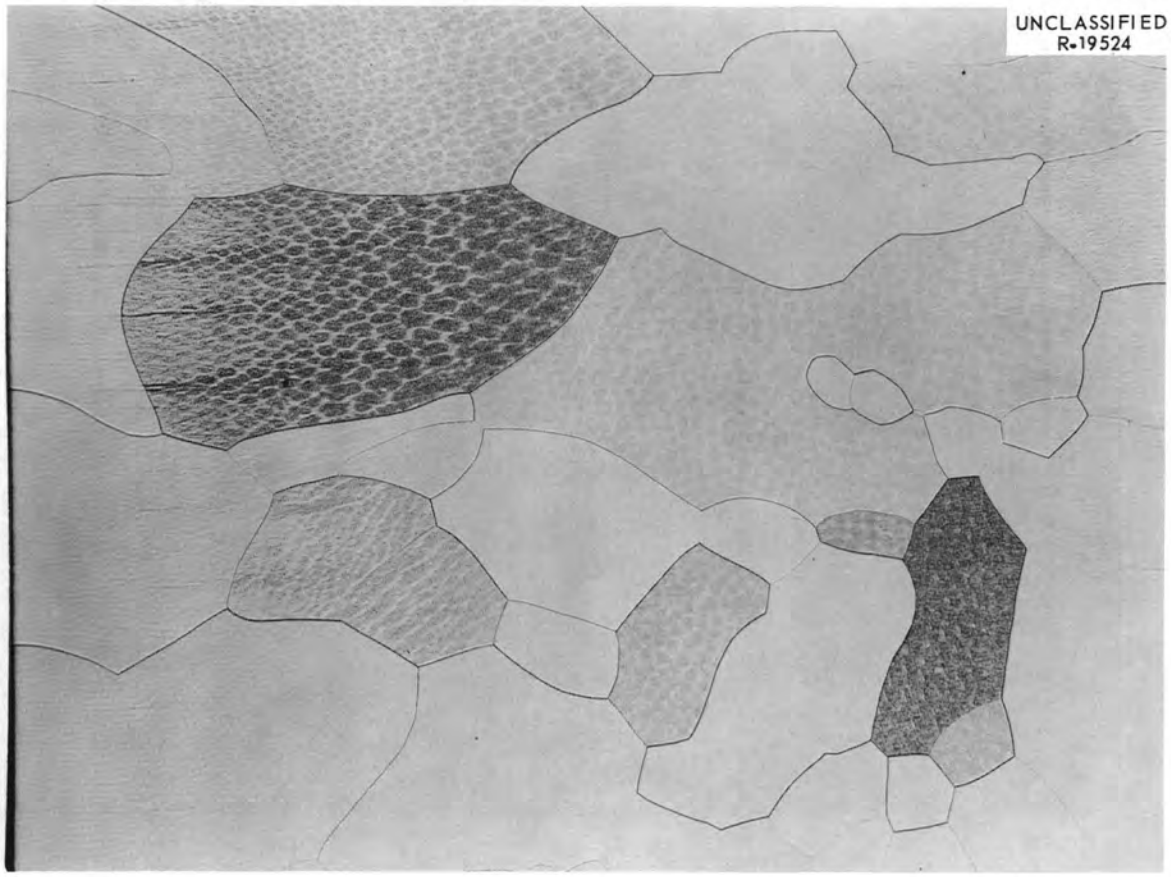

Fig. 20. Microstructure of Welded Nb–1% Zr Specimen (No. 187) After Testing at 28°C; Annealed 1 hr at 1000°C Prior to Testing; Weld Metal. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×.

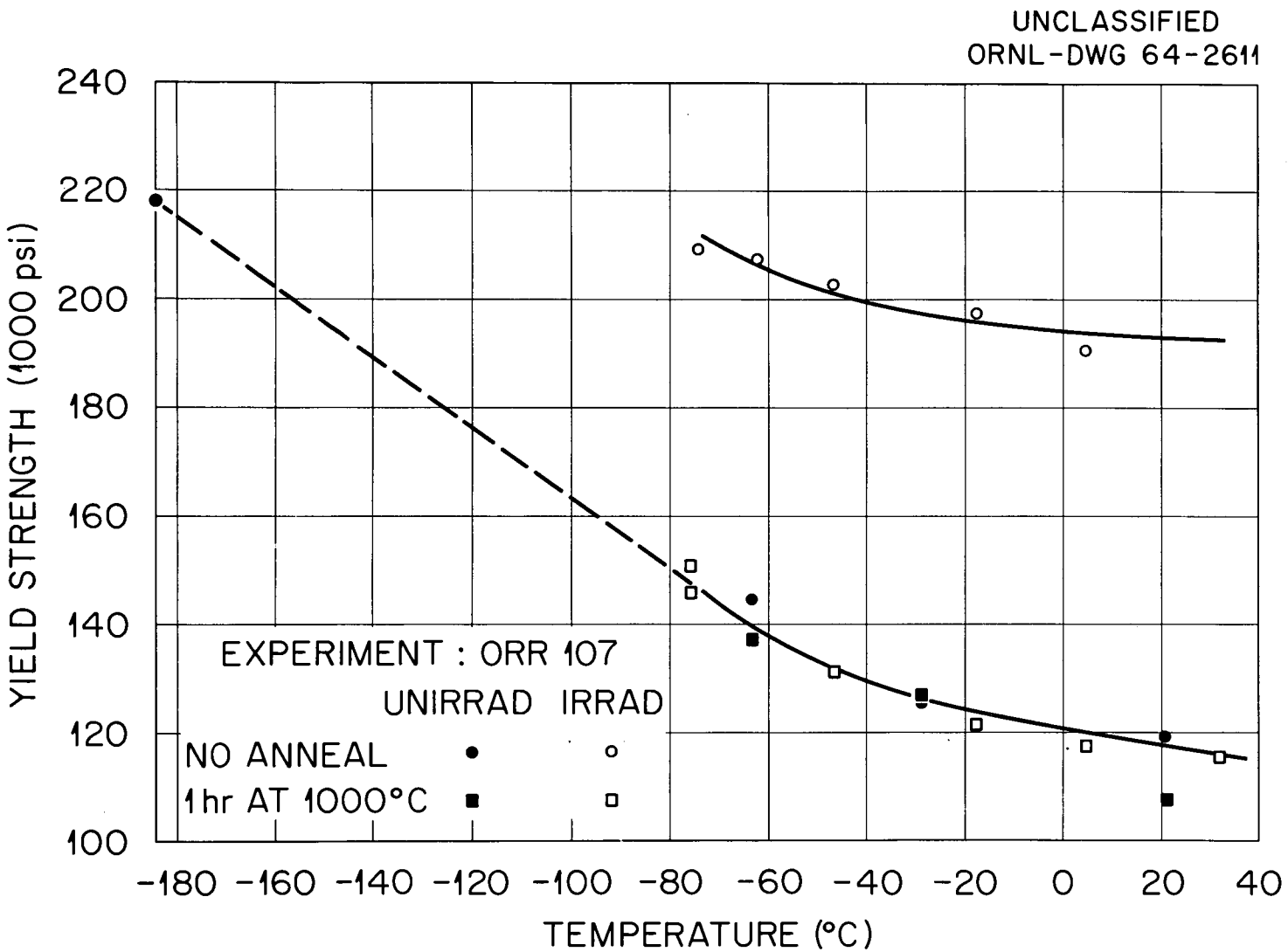

Fig. 21. Bend Properties of Recrystallized D-43 Alloy Before and After Irradiation.

to 193,000, an increase of 65%. Annealing for 1 hr at 1000°C following irradiation recovered the preirradiation properties of the alloy. Cold working the material 20% increased the strength (Fig. 22). Although irradiation produced similar yield stresses at room temperature in the annealed and cold-worked material, the strength at lower temperatures is significantly different. The yield strength increases more rapidly with decreasing temperature for the cold-worked material than for the annealed. Annealing 1 hr at 1000°C after irradiation did not recover the preirradiation properties of the alloy.

The hardness of annealed D-43 was increased 87 points from 196 to 240 DPH (Table 6). The hardness of the cold-worked material changed only slightly during irradiation.

Metallographic examination showed that the 1 hr at 1350°C anneal was not sufficient to bring about complete recrystallization. Figure 23 shows the structure of a specimen irradiated and tested at 4.4°C. The material was about 50% recrystallized. A specimen cold worked 20% and irradiated is shown in Fig. 24. The microstructures of the unirradiated and irradiated specimens were not detectably different.

DISCUSSION OF RESULTS

Irradiation was found to increase the transition temperature of the Mo–0.5% Ti alloy from 19 to 138°C. This is in agreement with the observations of Bruch, McHugh, and Hockenbury[3] and Makin and Gillies[4] that the transition of pure molybdenum was increased by comparable doses. This change in ductility was accompanied by an insignificant change in strength in contrast with the observation of Makin and Gillies[4] that the yield strength of pure molybdenum was increased. It was found that the irradiation-induced defects present in

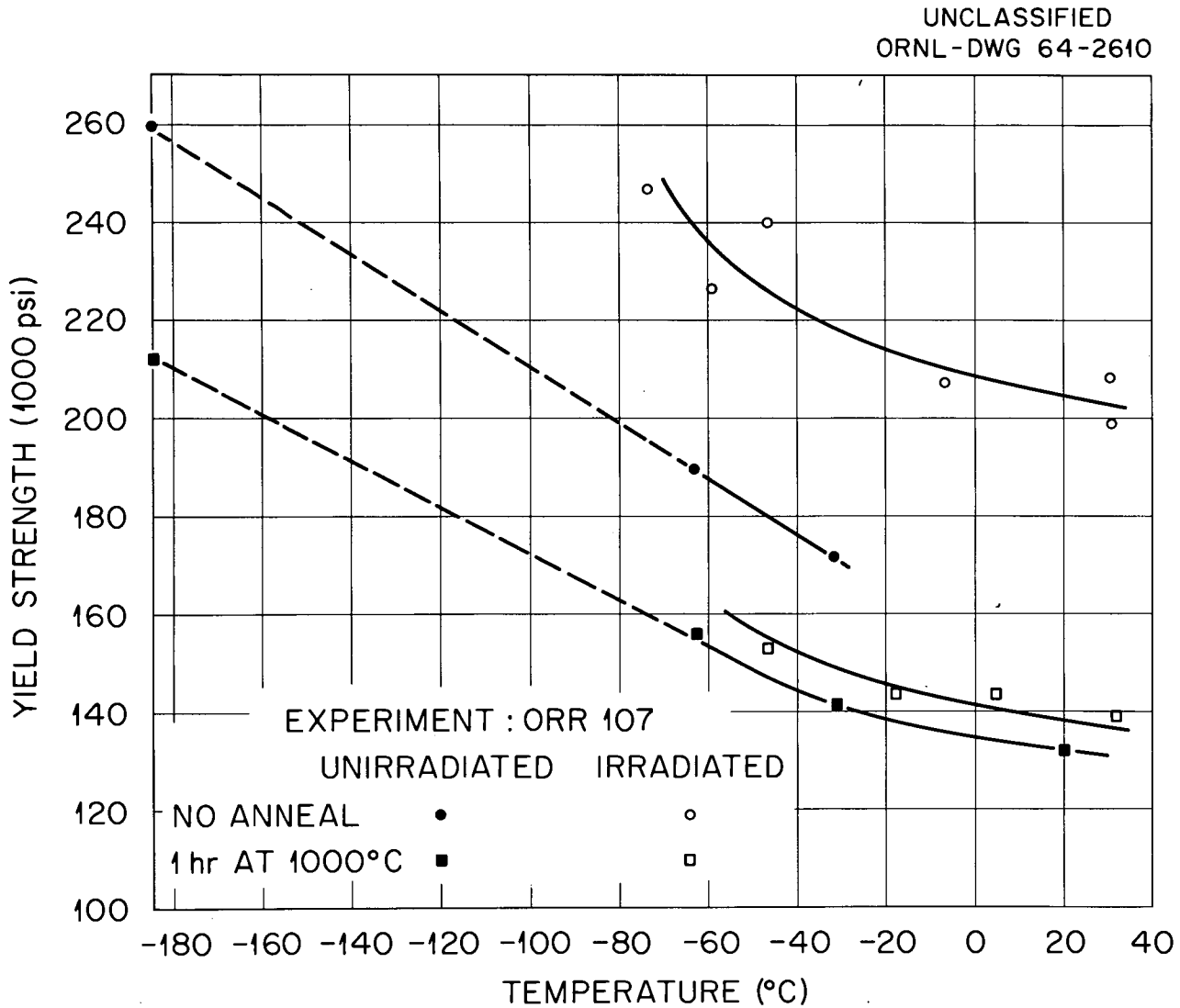

Fig. 22. Bend Properties of Cold-Worked D-43 Alloy Before and After Irradiation.

Table 6. Results of Hardness Measurements on D-43 Alloy

Post Irradiation Anneal	Hardness, DPH, Range (av)			
	Annealed		Cold-Worked 20%	
	Unirradiated	Irradiated	Unirradiated	Irradiated
none	184–213 (196)	226–249 (240)	218–249 (233)	249–258 (254)
1 hr at 1000°C	184–207 (198)		226–226 (226)	

Fig. 23. Microstructure of D-43 Alloy Specimen (No. 248) After Bending at 4.4°C; Annealed 1 hr at 1350°C Prior to Irradiation in Experiment 107. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×.

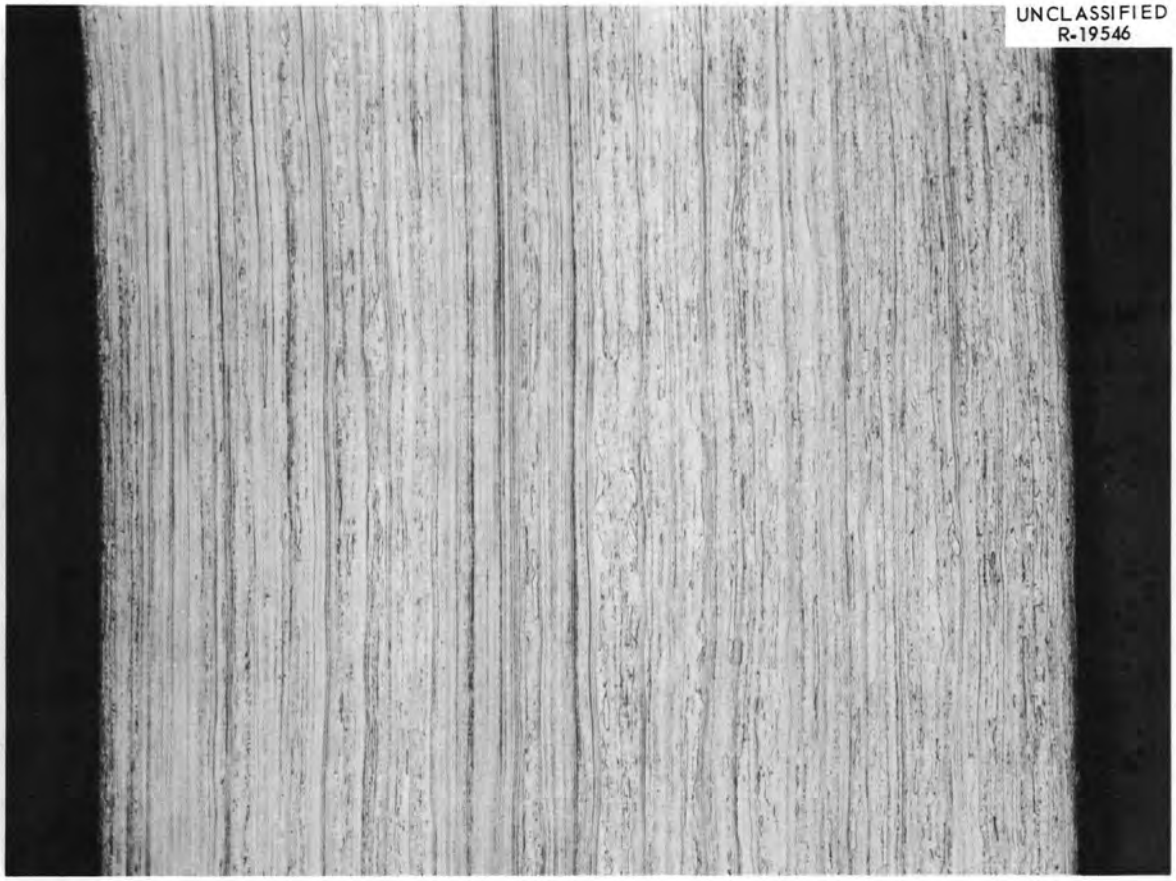

Fig. 24. Microstructure of D-43 Alloy Specimen (No. 272) After Bending at 31°C; Cold Worked 20% Prior to Irradiation in Experiment 107. Etchant: H_2O, HNO_3, HF, H_2SO_4. 100×.

the Mo–0.5% Ti alloy were only partially annealed after heating for 1 hr at 500°C. Since Kinchin and Thompson[5] observed that single vacancies were mobile in molybdenum at 150°C, it would be reasonable to assume that single defects would be removed in the Mo–0.5% Ti alloy by annealing at approximately the same temperature. Mastel et al.[10] observed two types of defects in irradiated molybdenum containing 150 ppm C: (1) clusters, 75 to 100 A in diameter, and (2) loops. Hence, although the defects present in the Mo–0.5% Ti alloy specimens were not observed directly, it seems probable that many of them are considerably more complex than single defects. Annealing studies indicate that these defects are not completely annealed after 1 hr at 1200°C.

Irradiation experiments on the Nb–1% Zr alloy in the annealed state showed that the strength of this material was increased but that sufficient ductility was retained at quite low test temperatures. It was found that annealing at 300 and 500°C increased the strength further. Annealing at 700°C brought about some recovery, and annealing at 1000°C resulted in complete recovery. Makin and Minter[11] irradiated pure niobium at 16°C and found further strengthening after annealing in the 125 to 175°C range, beginning of recovery at 350°C, and complete recovery after 1 hr at 600°C. These observations are quite compatible with those presently made on the alloy. Makin and Minter[11] reported the appearance of yield points after postirradiation annealing which was associated with vacancy diffusion. Although yield points were not observed in any of the Nb–1% Zr test specimens in the present study, this may have been due to the stress distribution across the bend specimen.

Although the authors know of no studies that give insight into the nature of the defects present, it would seem that they must be quite complex. Kinchin and Thompson[5] observed breaks in the annealing spectrum of irradiated

molybdenum due to vacancy diffusion at 150°C and for the same process in tungsten at 377°C. In view of these observations, vacancies in the Nb–1% Zr alloy would be expected to anneal at temperatures less than 500 to 700°C. Hence, the defects in this material are likely vacancy clusters and possibly loops.

The cold-worked Nb–1% Zr alloy was further strengthened and hardened by irradiation. Although the percent change in strength was not as great for the cold-worked as for the annealed material, the hardness of both materials increased by about 30 DPH points. Again further hardening occurred with a postirradiation anneal of 1 hr at 500°C and complete recovery resulted from a 1000°C anneal.

The results from the welded Nb–1% Zr alloy specimens indicate that welds in this material are influenced by irradiation much as the base metal. The occurrence of aging in the unirradiated weld metal after subsequent annealing is not surprising and has been reported by Franco-Ferreira and Slaughter.[13]

Although the D-43 alloy was not as extensively studied as was the Nb–1% Zr alloy, both appear quite similar in behavior. However, the D-43 alloy is strengthened significantly more than the Nb–1% Zr alloy after comparable irradiation doses. The D-43 alloy contains a dispersed carbide phase that may influence the distribution of the irradiation-induced defects. It was found that cold-worked material was affected to a lesser extent but in a similar manner.

Specimens of each material were examined metallographically and, although numerous interesting details were observed, the irradiated and unirradiated materials appeared identical. One point of difference was with

respect to the hardness impressions. The impressions in the unirradiated material were even and no adjacent markings were present. In the irradiated material large slip steps were present in the material adjacent to the impression. This effect, while most prominent in the Mo–0.5% Ti alloy, was present in the other materials and has also been noted in tungsten.[16] Such behavior can be explained in terms of the observations of Mastel et al.[10] which suggest that slip occurs in irradiated molybdenum only in bands where the defects were swept out. Hence, deformation is confined to certain zones which gives rise to large slip steps.

Although the Nb–1% Zr alloy was irradiated under two conditions of dose and temperature, it is difficult to say which variable resulted in the difference in properties. The problem is further complicated by the fact that increasing dose and temperature may have compensating effects.

It is indeed unfortunate that slightly lower test temperatures could not be obtained for the irradiated specimens. Since the slopes of the yield strength versus temperature curves are changing rapidly at the lower test temperature, the transition temperature would probably be reached at slightly lower temperatures.

In rationalizing the influence of irradiation on the deformation characteristics of the alloys studied, it is necessary to consider the current thinking relative to the plastic or brittle behavior of body-centered cubic metals. Stroh[17] feels that the important factor concerning the transition behavior is whether conditions are such that dislocations can escape a barrier. At relatively high temperatures thermal fluctuations will free the dislocations and high stress concentrations will not develop. The material will be ductile. As the temperature is lowered, thermal fluctuations will become insufficient

to free dislocations, and dislocation pileups with resulting high stress concentrations can develop. If the applied stress is high enough, brittle fracture will result. Cottrell[18] and Petch[19] have independently proposed models to account for the ductile-brittle transition which are quite similar. They both involve the production of a crack by dislocation coalescence and the propagation of this crack. The first requires a certain resolved shear stress on unlocked dislocations, and the second depends upon a tensile force. If conditions are such that the crack can propagate, the material will be brittle. If the crack propagation is limited, the material will continue to deform until several cracks bridge together and ductile failure occurs. Both of the models are supported by certain experimental observations. With respect to the important processes in yielding, the impurity "atmosphere" model of Cottrell[20] and the somewhat newer model of Hahn[21] based on dislocation multiplication and velocity characteristics represent the current schools of thought.

It would seem that the defects produced by irradiation would help to nucleate cracks by the models of Stroh,[17] Cottrell,[18] and Petch.[19] These defects could either act as a barrier to dislocations, as required by Stroh, or could coalesce to form a crack as required by Petch and Cottrell. However, the crack nuclei must be capable of propagation for fracture to occur. This in turn requires, by the models of Cottrell or Hahn, that either the dislocations be anchored or that the dislocation sources be inoperative. The fact that higher yield stresses were obtained for the irradiated specimens indicate that indeed the yielding processes are hindered by the irradiation-induced defects. By yielding being hampered, the stress in a test specimen continues to increase until yielding occurs or until the cracks are able to

propagate with resulting brittle fracture. The observation of defect-free areas in strained molybdenum specimens by Mastel et al.[10] and the observation of low strain hardening in niobium by Evans et al.[12] lend support to this mechanism. The stresses necessary to cause initial yielding are so high that, once dislocations have swept clean a small band of material, failure results in this low strain hardening material with high local and low uniform deformation.

SUMMARY AND CONCLUSIONS

The bend properties of Mo—0.5% Ti, Nb—1% Zr, and D-43 alloys have been evaluated before and after irradiation. Several significant observations were made.

1. The transition temperature of the Mo—0.5% Ti alloy was increased from 19 to 138°C. This was accompanied by hardening and an insignificant change in strength. Postirradiation annealing at 500°C caused partial recovery of properties, but annealing at 1200°C did not bring about full recovery.

2. The yield strength of annealed Nb—1% Zr alloy was increased by irradiation, but good ductility was retained at —75°C. An increase in hardness was also observed. Postirradiation anneals of 1 hr at 300 and 500°C resulted in further strengthening. A 700°C anneal gave partial recovery and 1 hr at 1000°C gave full recovery.

3. Cold-worked Nb—1% Zr alloy was strengthened but not to the same extent as the annealed material. Similar annealing kinetics were observed.

4. The Nb—1% Zr alloy weld specimens were found to respond to irradiation in a manner analogous with the base metal. The welds aged during postirradiation annealing at 500 and 1000°C.

5. Annealed D-43 alloy was strengthened by irradiation to a greater extent than Nb–1% Zr. Annealing at 1000°C completely recovered the properties.

6. Cold-worked D-43 alloy was strengthened to a lesser extent than annealed D-43. Annealing at 1000°C did not completely recover the pre-irradiation properties.

7. Because of the observed annealing kinetics, it is felt that the irradiation-induced defects are quite complex. The observed property changes are felt to be explained in terms of the interference of the irradiation-induced defects with normal yielding processes in the metal.

ACKNOWLEDGMENT

The bend experiments reported in this study were conducted by B. C. Williams. The welded Nb–1% Zr specimens were prepared by R. G. Donnelly of the Welding Group. The metallographic results were obtained by H. R. Tinch, E. H. Lee, and L. G. Shrader. The authors also gratefully acknowledge the assistance of the Metals and Ceramics Division Reports Office and the Graphic Arts Department in preparing this document. Appreciation is also expressed for the help of W. C. Thurber and D. A. Douglas in reviewing this manuscript.

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