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RADIATION HARDENING AND RADIATION-ANNEAL HARDENING IN VANADIUM-OXYGEN ALLOYS

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Abstract

Tensile tests were performed on high-purity vanadium containing 60-3160 weight ppm oxygen. Portions of the polycrystalline specimens were irradiated to 1.2×10^{19} neutrons/cm^2 (E>1MeV) at 105°C and to 1.5×10^{19} neutrons/cm^2 at 85°C. Two types of investigations were conducted: (1) a study of radiation-anneal hardening (RAH) in which the additional hardening upon post-irradiation annealing between 100°C and 800°C was observed, as well as the recovery to pre-irradiation properties, and (2) the effect of irradiation on the temperature dependence of yielding in unirradiated, as-irradiated, and radiation-annealed specimens.

The RAH (yield stress increase) occurs over the annealing temperature range of 100-400°C, and appears to increase in magnitude with increasing oxygen concentration. The recovery to pre-irradiation yield stress values takes place at 500-700°C. The mechanism proposed is that interstitial oxygen migrates to radiation-produced defect clusters, thus strengthening them as barriers to slip dislocation motion.

The temperature dependence of the yield stress was observed over the range of test temperatures from 4°K to 400°K. Radiation hardening increased with increasing oxygen concentrations for tests at 195-400°K. This was interpreted tentatively in terms of the formation of stronger dispersed barriers for higher oxygen alloys due to some trapping of oxygen at defect clusters during irradiation. At 4°K and 77°K, where twinning occurs, the radiation hardening diminished with increasing oxygen concentration. Radiation softening did not occur, when analyzed in terms of the total yield stress.

Introduction

Vanadium and vanadium alloys are of practical interest because of their possible application to advanced reactor systems of fission and fusion types. In such applications vanadium materials are subjected to exposure to high-energy neutrons. This has stimulat efforts to achieve a better fundamental understanding of the effect of radiation-produced defects on the mechanical properties of vanadium as a function of controlled additions of important solute additions.

Progress in research on vanadium was impeded in former years by difficulties in the preparation of specimens of adequate purity. However, considerable advances have been made more recently in vanadium purification by such processes as iodide refining (1), electrorefining (2), electron-beam zone melting and vacuum purification (3-5), and electrotransport purification (6). Techniques have also improved for doping with impurities, particularly interstitial impurities, and in the analysis and characterization of vanadium specimen materials. As a result, a number of papers are now available on various aspects of flow and fracture of high-purity vanadium and vanadium doped with oxygen, nitrogen, carbon, and hydrogen (see, for example, 7-19). It is known that the mechanical properties of vanadium are strongly influenced by interstitial impurities. The presence of interstitial impurities increases the ductile-brittle transition temperature, is largely responsible for strain aging, and inhibits low-temperature deformation twinning. However, it is not yet well established how large a role interstitial impurities play in the large temperature and strain-rate dependence of yielding in vanadium and BCC transition metals generally. For example, Lachenmann and Schultz (20) measured the critical resolved shear stress in tantalum single crystals purified to below 3.5 at. ppm, total, of interstitial impurities and in samples containing increasing amounts of nitrogen. While the nitrogen additions increased both the thermal and athermal components of the critical resolved shear stress, considerable temperature dependence remained even for the material believed to contain as little as 3.5 at. ppm of interstitial impurities.

The combined effects on the temperature dependence of yielding in vanadium of an interstitial impurity (oxygen) and radiation-produced defects have been studied by Arsenault and Pink (21). The athermal component of yield stress, σ_μ, was observed to increase with increasing oxygen concentration. Also, σ_μ increased upon irradiation to a greater extent for the higher oxygen materials. However, the results for the thermal component of yield stress, σ^*, were somewhat more complicated in that σ^* was observed to increase upon irradiation for low oxygen concentrations, to decrease for intermediate oxygen concentrations, and to be unchanged for high oxygen concentrations.

It has been suggested as a result of yield stress measurements on unirradiated and neutron-irradiated niobium (22) that the additional increase in yield stress (radiation-anneal hardening, RAH) that occurs upon post-irradiation annealing may be due to the motion of interstitial impurities to radiation-produced defect clusters. The defect clus-s may become strengthened as barriers to slip dislocation motion when the interstitial impurity atoms become trapped at the defect clusters, and this causes an increase in yield stress (23). RAH has been observed in neutron-irradiated vanadium by Smolik and Chen (24), Venetch et al. (25), Smidt (26), and Shiraishi et al. (27). In the present work, the influence of oxygen impurity atoms on RAH is being investigated. It will be seen that RAH appears to increase with increasing oxygen concentration.

Additional evidence for attributing RAH peaks to interstitial impurities stems from studies of internal friction and electrical resistivity in irradiated vanadium (28-30) and niobium (31, 32). The internal friction specifically due to interstitial oxygen impurities (Snoek damping) decreases upon annealing in the same temperature range (100-200°C) as is observed for the onset of RAH. A resistivity annealing step is also observed at these temperatures whose magnitude increases with oxygen concentration. On the other hand, it is possible to argue that these annealing effects are due to the motion of intrinsic radiation-produced defects, and the question is presently being debated in the technical literature (33-34).

This paper is also concerned with the effect of neutron irradiation and oxygen doping on the temperature dependence of yielding in vanadium. The earlier work of Arsenault and Pink (21) is extended down to test temperatures of 4°K. Furthermore, a separate series of observations is presented for samples given post-irradiation annealing treatment designed to bring the material to strength levels corresponding to the peak on the RAH curve. Tensile tests were used exclusively, whereas most of Arsenault and Pink's tests were in compression. It will be seen that profuse twinning was observed at 77°K and 4°K in our work, and a further point of interest is the effect of irradiation and radiation annealing on deformation twinning at low temperatures. This is a question that has not been widely investigated, although Smolik and Chen (35) reported that twinning is suppressed in vanadium irradiated at 83°K and is recovered upon annealing between 193°K and 212°K.

Experimental Procedure

The vanadium used in this study was obtained in two lots from the U. S. Bureau of Mines at Boulder City, Nevada, in the form of electrorefined dendritic crystals. Lot 1 was used for the RAH studies and Lot 2 for the work on the temperature dependence of yielding. Typical analyses are given in Table 1.

TABLE 1.

Chemical Analysis of Starting Vanadium

Element	Concentration (wt. ppm)		Element	Concentration (wt. ppm)	
	Lot 1	Lot 2		Lot 1	Lot 2
Al		27	Mn	<5	<20
B		<150	Ni		<50
Ca		<30	Si	<15	<40
Cr	50	<80	Ti		<25
Cu	<5	<25	O	13	25
Fe		45	N	1	2
Mg	<3	<20	C	7	9

For Lot 1, three oxygen concentrations were prepared by co-arc melting vanadium metal and fused V_2O_5 powder in a gettered argon atmosphere. The material was melted into 100 gm fingers. For Lot 2 the same procedure was followed for preparing four oxygen concentrations.

The material from both lots was swaged and machined into tensile specimens with a one inch gauge length and approximately 0.08 inch gauge diameter. The tensile specimens were then electropolished and annealed at about $900^{\circ}C$ at a pressure of 10^{-6} torr or better. For the Lot 2 specimens, the two lowest oxygen concentration materials had a mean grain diameter of 0.0057 inches while the next higher oxygen material had a mean grain diameter of 0.0040 inches. Oxygen concentrations were determined by vacuum fusion techniques to be 60, 205, and 640 wt. ppm* for the specimens from Lot 1, and 70, 660, 2100, and 3160 wt. ppm for the specimens from Lot 2. For the Lot 2 irradiation, the tensile specimens were placed in aluminum sample holders that were sealed under a partial pressure of helium. The sample holders were irradiated in a cadmium shielded capsule and a thermocouple monitored the temperature of one of the sample holders.

The irradiation was performed at the neutron convertor facility of the Ames Laboratory Research Reactor at a temperature of $85^{\circ}C$. The samples were irradiated to a fluence of 1.5×10^{19} neutrons/cm^2 (E>1MeV). Following de-encapsulation the samples were observed to have a surface corrosion layer. This was removed using a sodium hydroxide solution.

The Lot 1 specimens were irradiated in contact with the heavy water coolant in the neutron convertor facility of the ALRR at $105^{\circ}C$ to a fluence of 1.2×10^{19} neutrons/cm^2 (E>1MeV). Further details have been described previously (30).

The tensile tests were conducted at strain rates of 1.67×10^{-4} sec^{-1} in an Instro

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*All concentrations are in weight parts-per-million unless otherwise specified.

testing machine. For the study of RAH the test temperature was 300°K. For the study of temperature dependence of yielding, the test temperatures were 4.2°K (liquid helium), 77°K quid nitrogen), 195°K (dry ice and acetone), 300°K (room temperature), and 400°K (heated oil bath).

Results

The lower yield stress was chosen as the principal measure of strength. Fig. 1 shows stress-strain curves for unirradiated and irradiated specimens of the V-2100 (vanadium-2100 wt. ppm oxygen) alloy. The criterion for the lower yield stress is illustrated for cases where a yield drop is exhibited. When no yield drop was apparent, an 0.2 percent offset yield stress criterion was employed. Fig. 1 also shows that the yield stress at 300°K increased from 49 to 80 kpsi upon irradiation. Also shown is the decrease upon irradiation in uniform strain from 15.6 percent to zero and decrease in fracture strain from 23.5 to 8.4 percent.

Fig. 2 shows the yield stress for unirradiated samples as a function of test temperature and oxygen concentration. It is quite apparent that the general level of the yield stress curve decreases with decreasing oxygen concentration. It also appears that there is some decrease in the temperature dependence of the yield stress with decreasing oxygen concentration. However, even for the V-70 alloy considerable temperature dependence remains. These results are quite similar to those for Ta-N alloys (20). It appears that purer material is required to demonstrate whether ultra high-purity BCC metals can achieve a temperature independent yield stress as is characteristic of FCC metals.

The results of the RAH measurements are illustrated in Fig. 3, where each point represents a separate specimen annealed for one hour at the indicated annealing temperature and tested at room temperature. We see that the increase in yield stress upon irradiation is greater for higher oxygen specimens. Furthermore, RAH takes place upon annealing between 100°C and 400°C (0.17 T_m and 0.31 T_m, where T_m is the melting point in °K), and upon annealing from about 500°C to 700°C (0.33 T_m to 0.45 T_m) there is a general recovery to unirradiated values. The shapes of the RAH curves are not greatly affected by the oxygen concentration, and they appear to be qualitatively similar to those obtained by previous investigators (24-27). Several transmission electron microscopy (TEM) studies have been made of the density and size distribution of defect clusters in irradiated and annealed vanadium (26,27,35-37). The defect cluster densities upon irradiation to fluences of 10^{19}-10^{20} neutrons/cm^2 are in the range 10^{16}-10^{17} cm^{-3}, and upon annealing to about 400°C little change in density is observed. However, upon annealing in the range 500°-700°C the defect ster density is reduced and the average defect cluster size is increased.

Figures 4, 5, and 6 illustrate the effect of irradiation at 85°C on the temperature dependence of the yield stress and the subsequent effect of a one hour anneal at 400°C.

This annealing treatment was chosen to correspond to the peaks on the RAH curves shown in Fig. 3. The increase in yield stress upon irradiation (filled symbols) appears to increase with increasing oxygen concentration for tests at higher temperature (200-400°C) in agreement with the results in Fig. 3. However, for tests at lower temperatures (77°K and 4°K) the radiation hardening decreases with increasing oxygen concentration. Nevertheless, for all three oxygen concentrations the curves for the irradiated specimens always lie above the corresponding curves for unirradiated specimens, and in this sense no evidence of radiation softening is obtained. The somewhat reduced radiation hardening at 4°K and 77°K for higher oxygen concentrations is interesting since twinning was prominent at these temperatures as indicated by repeated sudden drops in load during testing, audible clicks, and subsequent metallographic examination.

The half-filled symbols in Figs. 4-6 represent the effect of the anneal at 400°C for one hour. For test temperatures of 300°K and 400°K, the RAH is greater for the V-660 and V-2100 alloys than for the V-70 alloys, but there appears to be little or no difference in RAH at these temperatures between the V-660 and V-2100 alloys. The RAH for one hour anneal at 400°C for the V-660 alloys measured at 300°K is about 12 kpsi (Fig. 5), as compared to about 10 kpsi for the V-640 alloy (Fig. 3). This small difference is probably within experimental error or it may be due to combination of a slightly higher oxygen concentration and radiation fluence and lower irradiation temperature for the V-660 alloy. At lower test temperatures, Figs. 4-6 show little or no effect of the 400°C anneal on the yield stress, with the exception of the measurement at 4°K for the V-70 alloy (Fig. 4). In this case, the radiation-annealed specimen indicated a lower yield stress than the as-irradiated specimen, but still a higher yield stress than for the unirradiated specimen.

As is generally observed for BCC transition metals, the yield stress curves in Figs. 4-6 show a levelling off for higher test temperatures. It has become customary to regard the stationary value as the athermal stress, σ_μ, and the difference, $\sigma-\sigma_\mu$, as the thermal or effective stress, σ^*, As a measure of σ^*, we have plotted in Figs. 7-9 the yield stress at temperature T, $\sigma(T)$, minus the yield stress at 400°K, $\sigma(400^\circ K)$. No consistent pattern as a function of oxygen concentration appears to emerge for higher test temperatures. But, for the twinning temperatures 4°K and 77°K, the thermal stress σ^* upon irradiation appears to show an increase, to be unchanged, and to show a decrease for the V-70, V-660, and V-2100 specimens, respectively.

Discussion

It is of interest to compare our observations on the effect of irradiation on the thermal stress (Figs. 7-9) with the corresponding results on polycrystalline specimens o Arsenault and Pink (21). In both investigations, an increase in thermal stress ($\Delta\sigma^*$) was observed upon irradiation for low-oxygen vanadium, particularly at lower temperatures.

However, the results for the higher oxygen alloys are not so straightforward. For our 660 alloy, Fig. 8 shows some indication of positive values for $\Delta\sigma^*$ at 77°K and 200°K, and a slight negative value at 4°K. These results are somewhat suggestive of Arsenault and Pink's results for vanadium-300 ppm oxygen [see Fig. 6 of (21)], which also shows a peak in $\Delta\sigma^*$ as a function of test temperature. For the high-oxygen vanadium, however, Arsenault and Pink indicate no change in σ^* for V-1800 ppm oxygen tensile tests, whereas our Fig. 9 indicates an increase at 195°K, a slight decrease at 77°K, and a large decrease at 4°K.

A more straightforward approach may be to analyze the effect of irradiation on the total yield stress, σ. The change in yield stress upon irradiation, $\Delta\sigma=\sigma_i-\sigma_u$ is plotted in Fig. 10 as a function of test temperature. We see that in the range of temperatures where the deformation is by slip (195-400°K), $\Delta\sigma$ increases with increasing oxygen concentration, with a tendency for larger $\Delta\sigma$'s for the lower temperatures in this range for V-660 and V-2100 alloys but no dependence on temperature for the V-70 alloys. It may be that some trapping of oxygen at defect clusters occurs during irradiation in the higher oxygen alloys, which strengthens the defect clusters as barriers to slip dislocation motion.

Fig. 10 also shows that at the two temperatures where twinning occurs (4°K and 77°K), $\Delta\sigma$ decreases with increasing oxygen concentration. It has been observed that the presence of interstitial impurities suppresses low temperature twinning by providing sites at which dislocation tangles form or by inducing cross slip, both of which tend to break up the stress concentrations necessary for twinning (38). It may be supposed that radiation-produced defect clusters have the same effect. Some evidence for the suppression of twinning upon irradiation has been observed in vanadium (35), niobium (39), and iron (40), although the promotion of twinning upon irradiation was reported for Nb-40%V (41). Also, for the work on irradiated vanadium (35), the twinning properties were recovered upon annealing at a relatively low temperature (between 193°K and 212°K). Accepting the idea that twinning is suppressed in some cases by both interstitial impurity atoms and radiation-produced defects, we may argue that the effect of irradiation in increasing the twinning stress at low temperatures should be smaller for higher oxygen alloys, where more interstitial impurity atoms are already present to reduce the stress concentrations necessary for twin nucleation and growth. However, it must be recognized that the situation is more complicated than the above brief discussion might suggest. For one thing, significant amounts of slip may precede, accompany, and follow twin formation, as Wasilewski (42) s pointed out.

The observation that RAH is greater in higher oxygen vanadium alloys (Fig. 3) is consistent with a similar observation for Fe-N alloys (43) and with the suggestion made earlier (22,23) that RAH is associated with the motion of interstitial impurity atoms to radiation-produced defect clusters. If this is the case, it is instructive to calculate whether oxygen atoms could be expected to reach the defect clusters for the annealing times employed and the annealing temperatures (100-400°C) at which the RAH occurs. The activation energy, Q, for diffusion of oxygen in vanadium is 1.18 eV and the pre-exponential diffusivity factor, D_0, is 0.013 cm^2/sec (44). The corresponding diffusion distance is

$$\bar{x} = 2(Dt)^{\frac{1}{2}}$$

where

$$D = D_0 \exp(-Q/kT)$$

Using t = 1 hr, we calculate $\bar{x} = 14Å$ for $T = 100^\circ C$ and $5.2 \times 10^4 Å$ for $T = 400^\circ C$. The defect cluster density is likely to be in the range 10^{16}-10^{17} cm^{-3} (26,27,35-37), and thus the average spacing between defect clusters would be about 200-500Å. Hence, oxygen impurity atoms appear to have sufficient mobility to migrate to the defect clusters where they become trapped.

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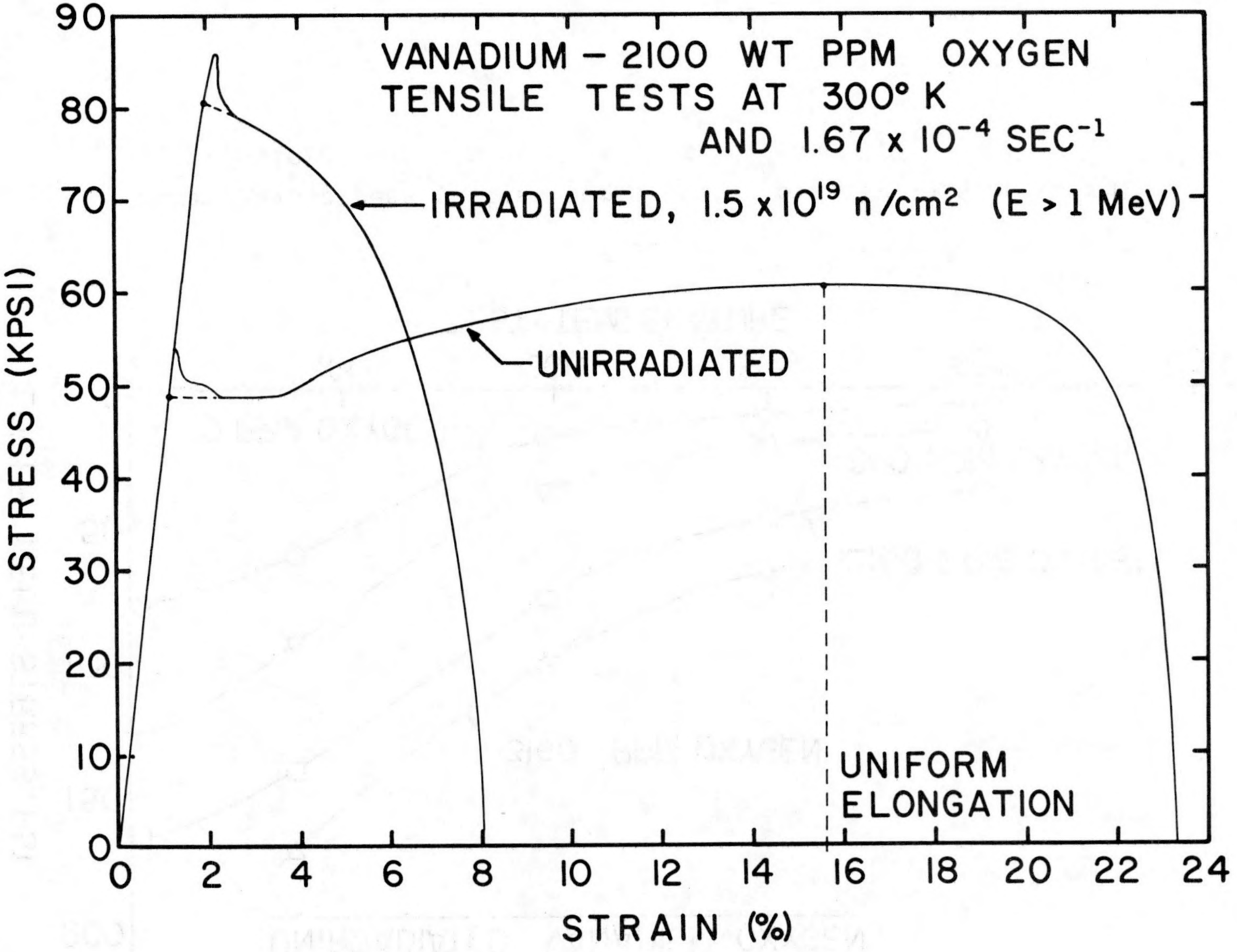

FIG. 1

Comparison of stress-strain curves of unirradiated and irradiated vanadium-2100 wt ppm oxygen tested at 300°K. Irradiation temperature, 85°C.

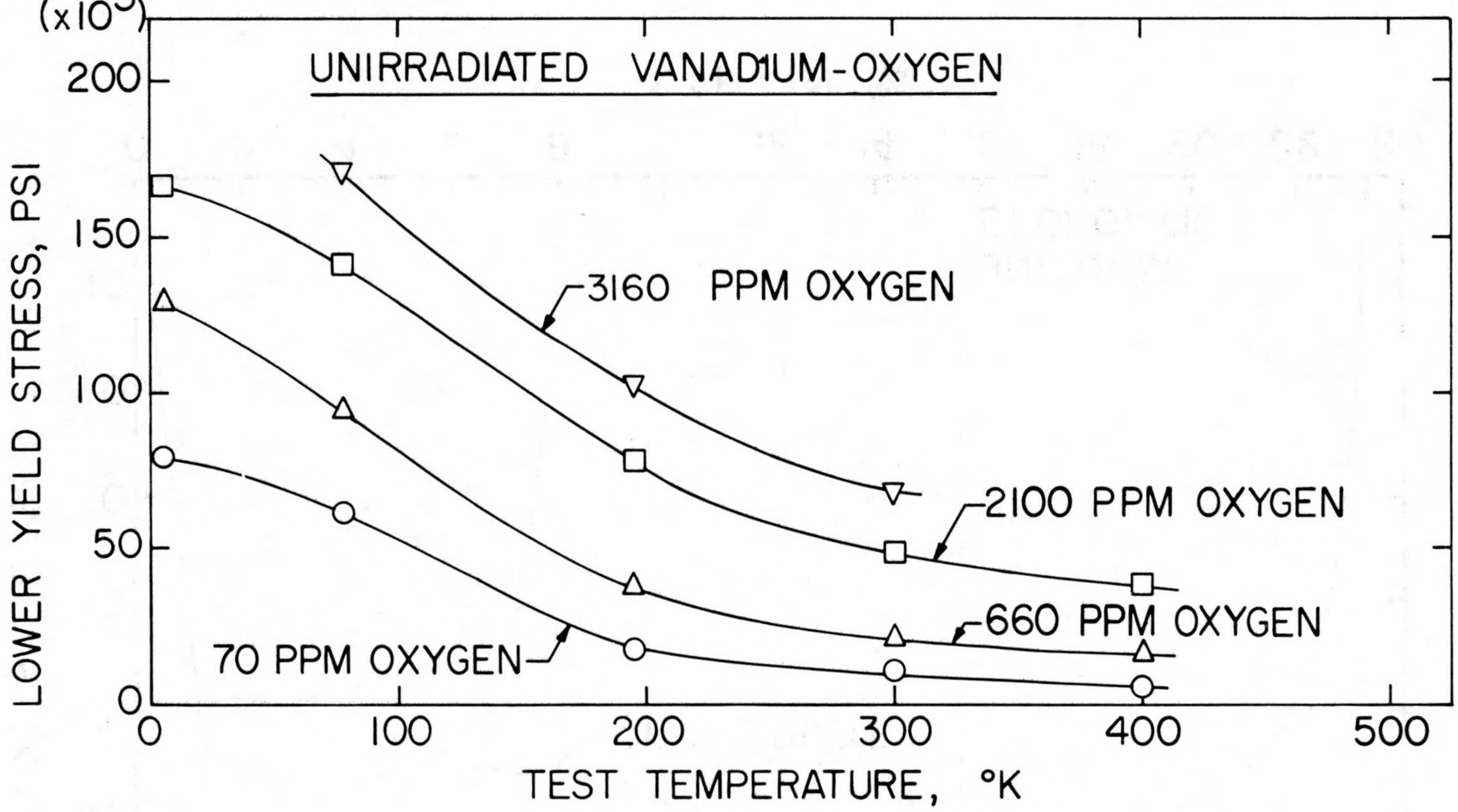

FIG. 2

Lower yield stress vs test temperature for unirradiated vanadium-oxygen alloys. Strain rate, 1.67×10^{-4} sec^{-1}.

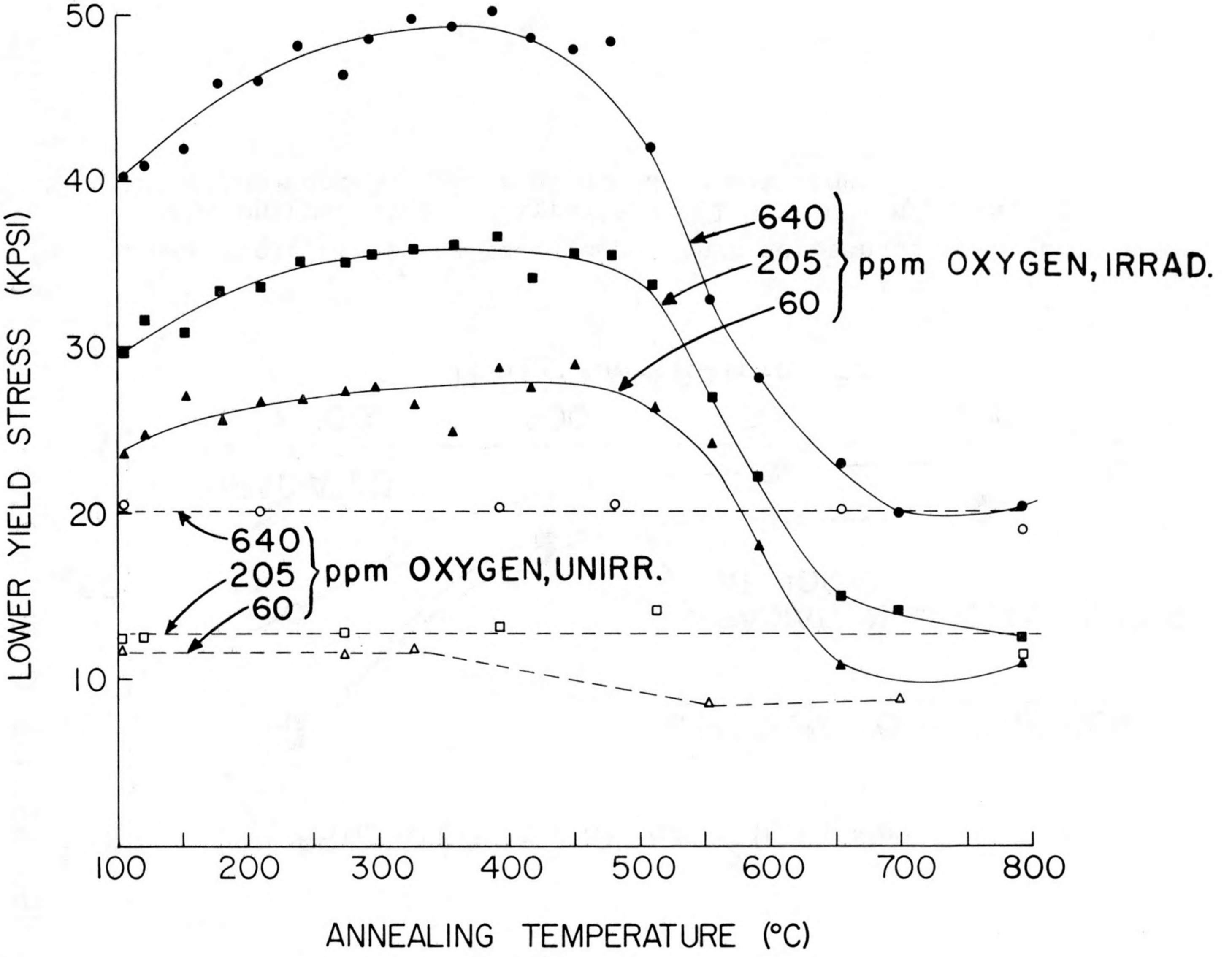

FIG. 3

Lower yield stress vs annealing temperature for unirradiated and irradiated vanadium-oxygen alloys. Irradiation dose and temperature: 1.2 x 10^{19} n/cm^2 (E >1 MeV) at 105°C. Test temperature, 300°K; annealing time, 1 hr.

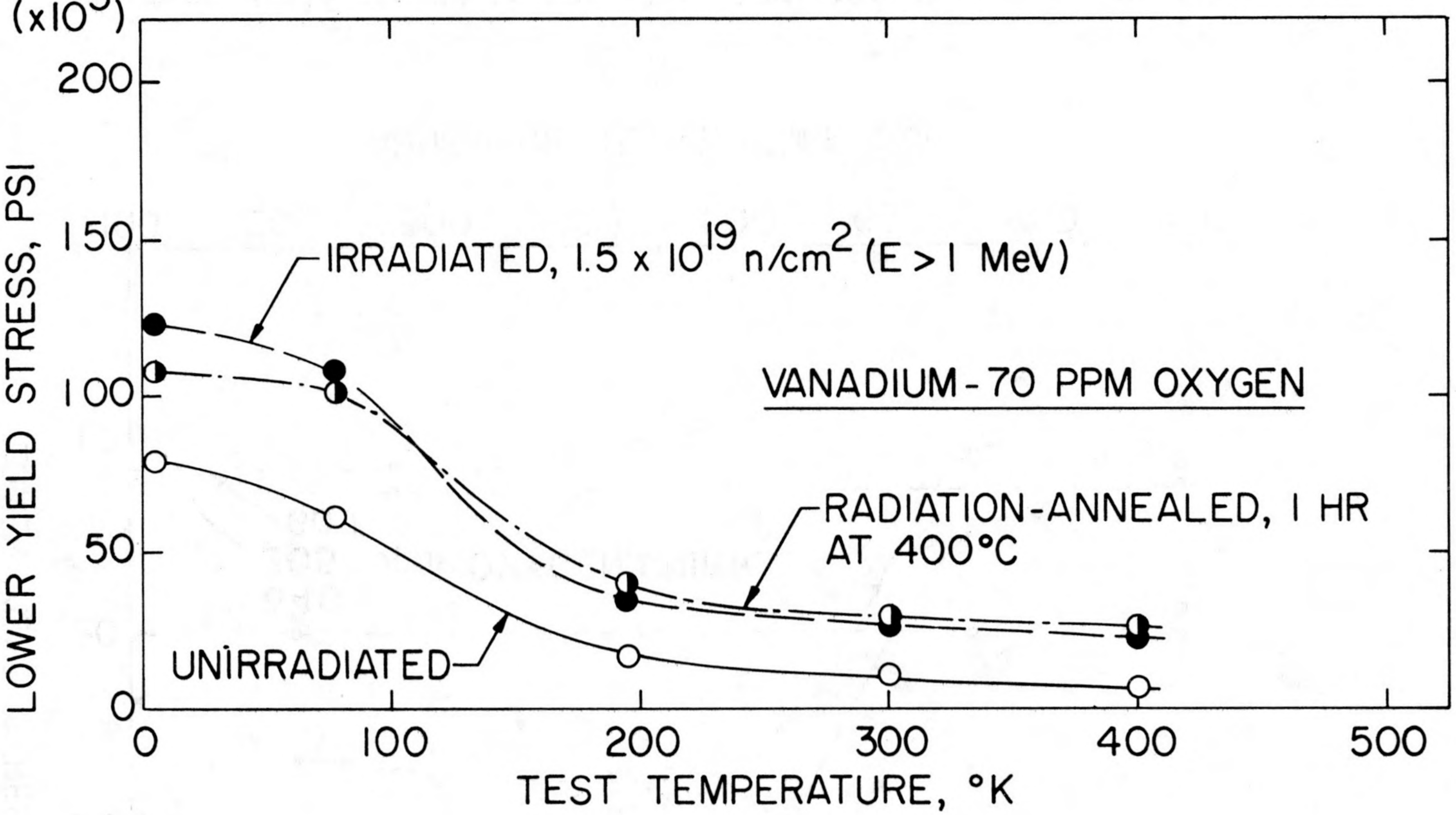

FIG. 4

Lower yield stress vs test temperature for vanadium-70 ppm oxygen in the unirradiated, as-irradiated (1.5 x 10^{19} n/cm^2 at 85°C), and radiation-annealed (1 hr at 400°C) conditions.

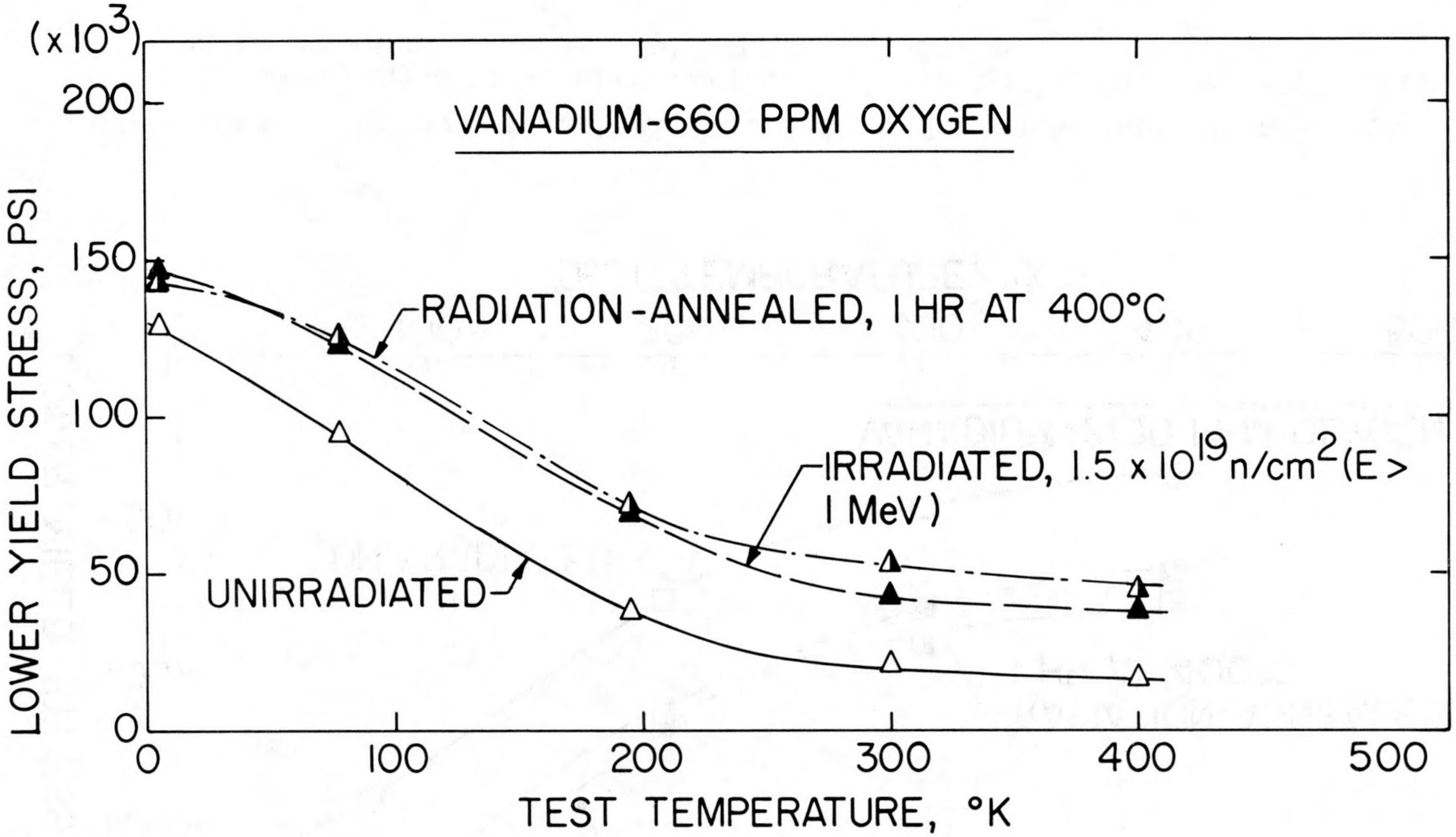

FIG. 5

Lower yield stress vs test temperature for vanadium-660 ppm oxygen in the unirradiated, as-irradiated (1.5 x 10^{19} n/cm^2 at 85°C), and radiation-annealed (1 hr at 400°C) conditions.

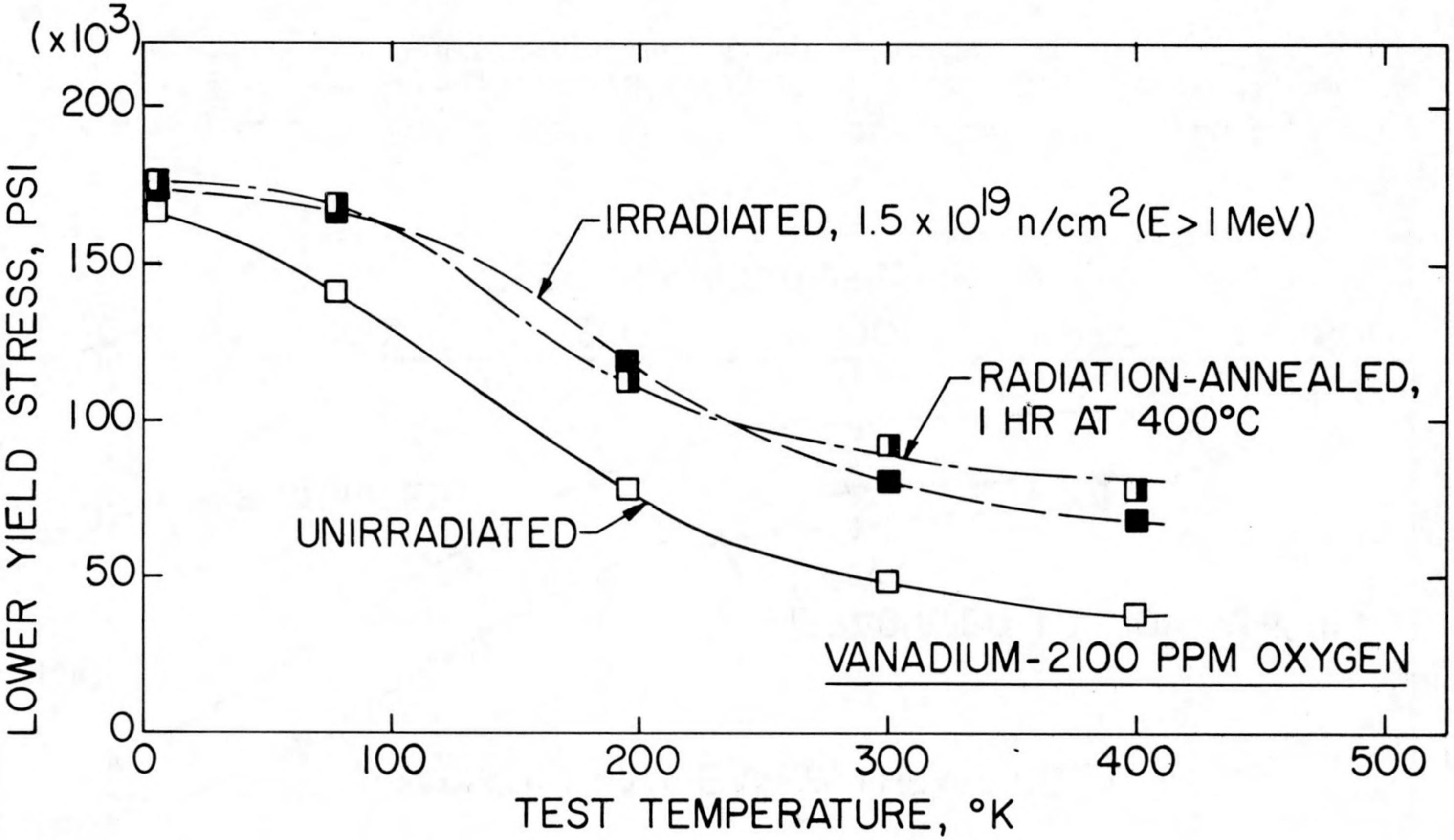

FIG. 6

Lower yield stress vs test temperature for vanadium-2100 ppm oxygen in the unirradiated, as-irradiated (1.5 x 10^{19} n/cm^2 at 85°C), and radiation-annealed (1 hr at 400°C) conditions.

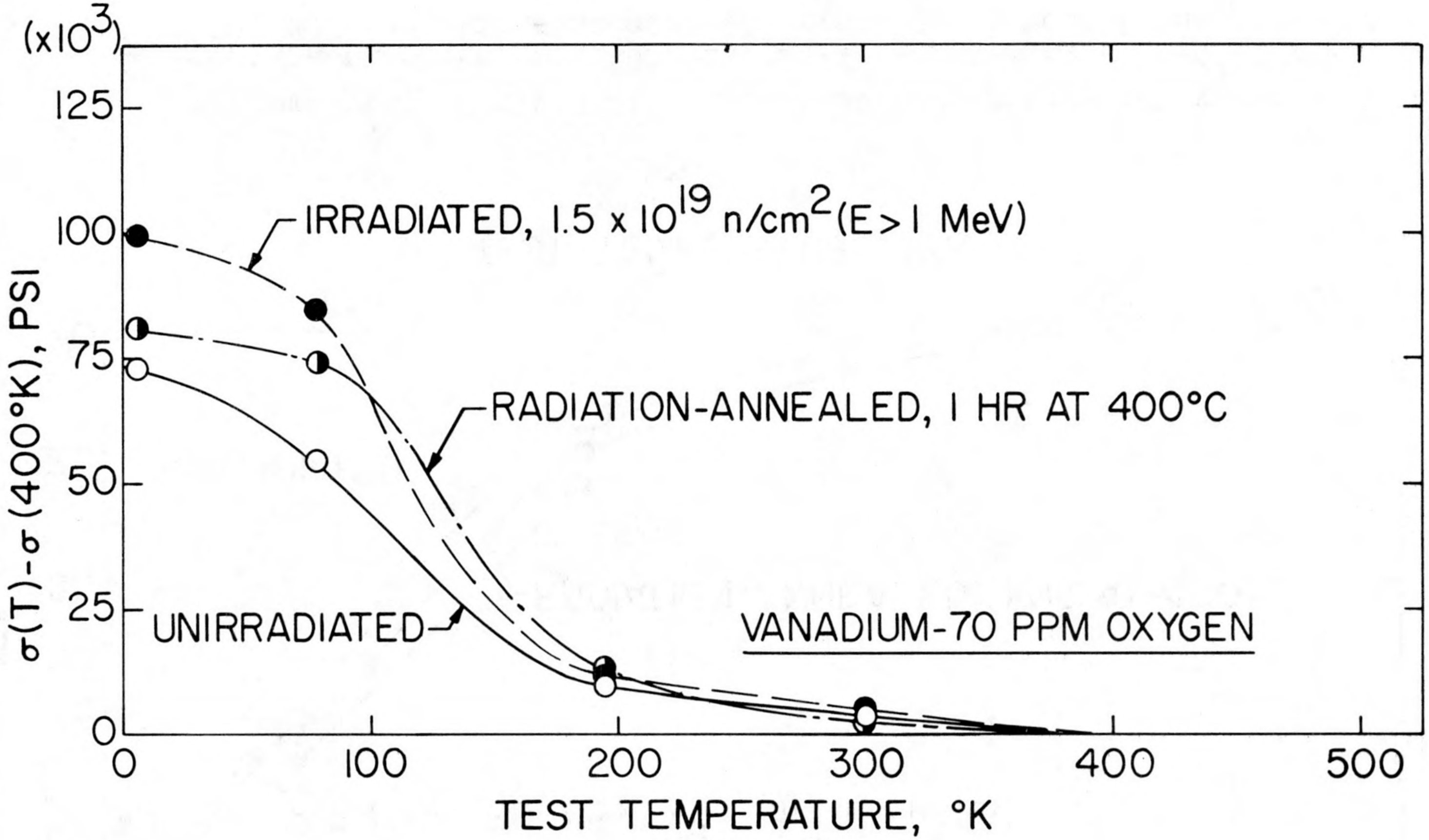

FIG. 7

Thermal component of yield stress vs test temperature for vanadium-70 ppm oxygen in the unirradiated, as-irradiated (1.5×10^{19} n/cm^2 at 85°C), and radiation-annealed (1 hr at 400°C) conditions.

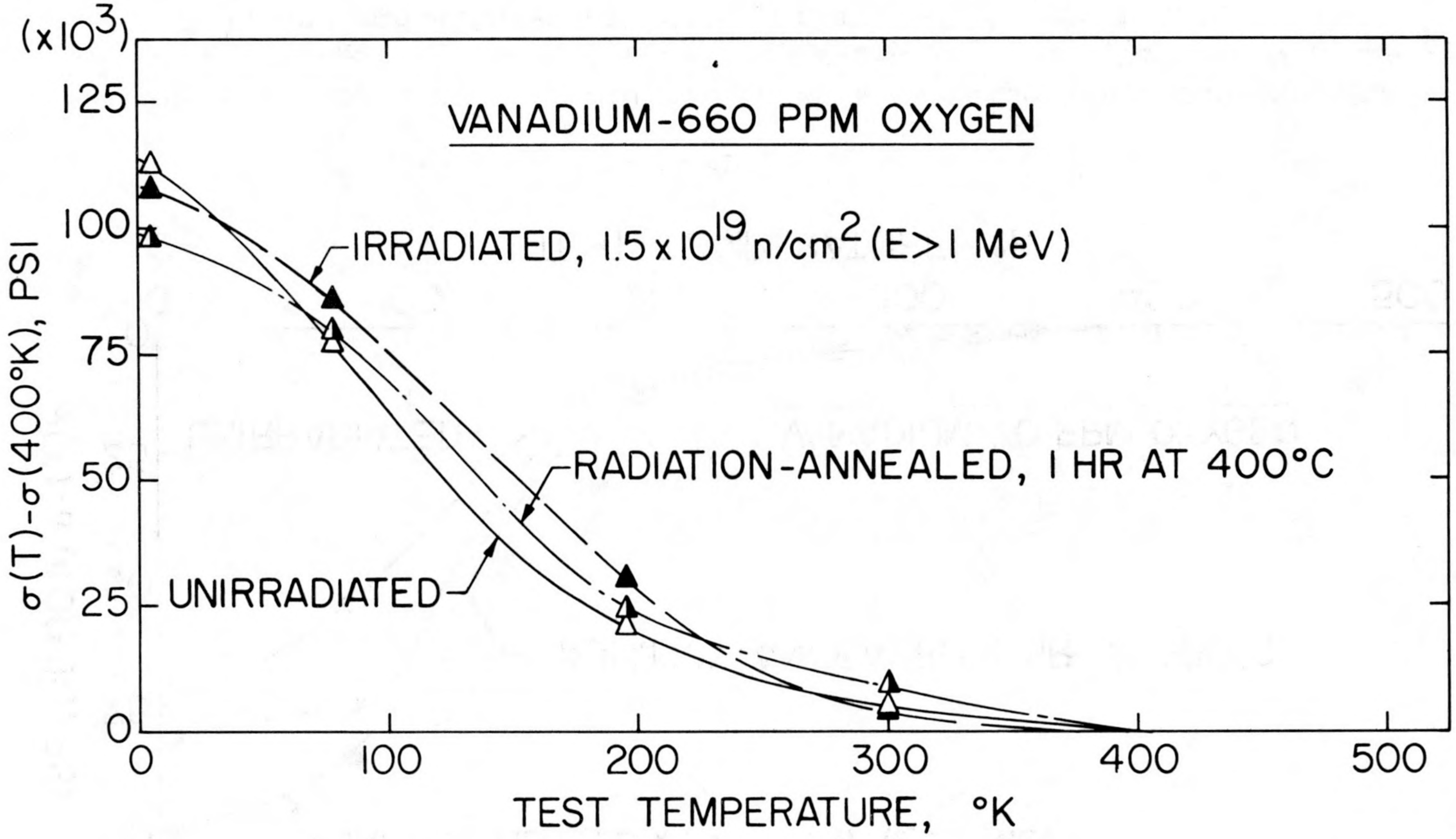

FIG. 8

Thermal component of yield stress vs test temperature for vanadium-660 ppm oxygen in the unirradiated, as-irradiated (1.5 x 10^{19} n/cm^2 at 85°C), and radiation-annealed (1 hr at 400°C) conditions.

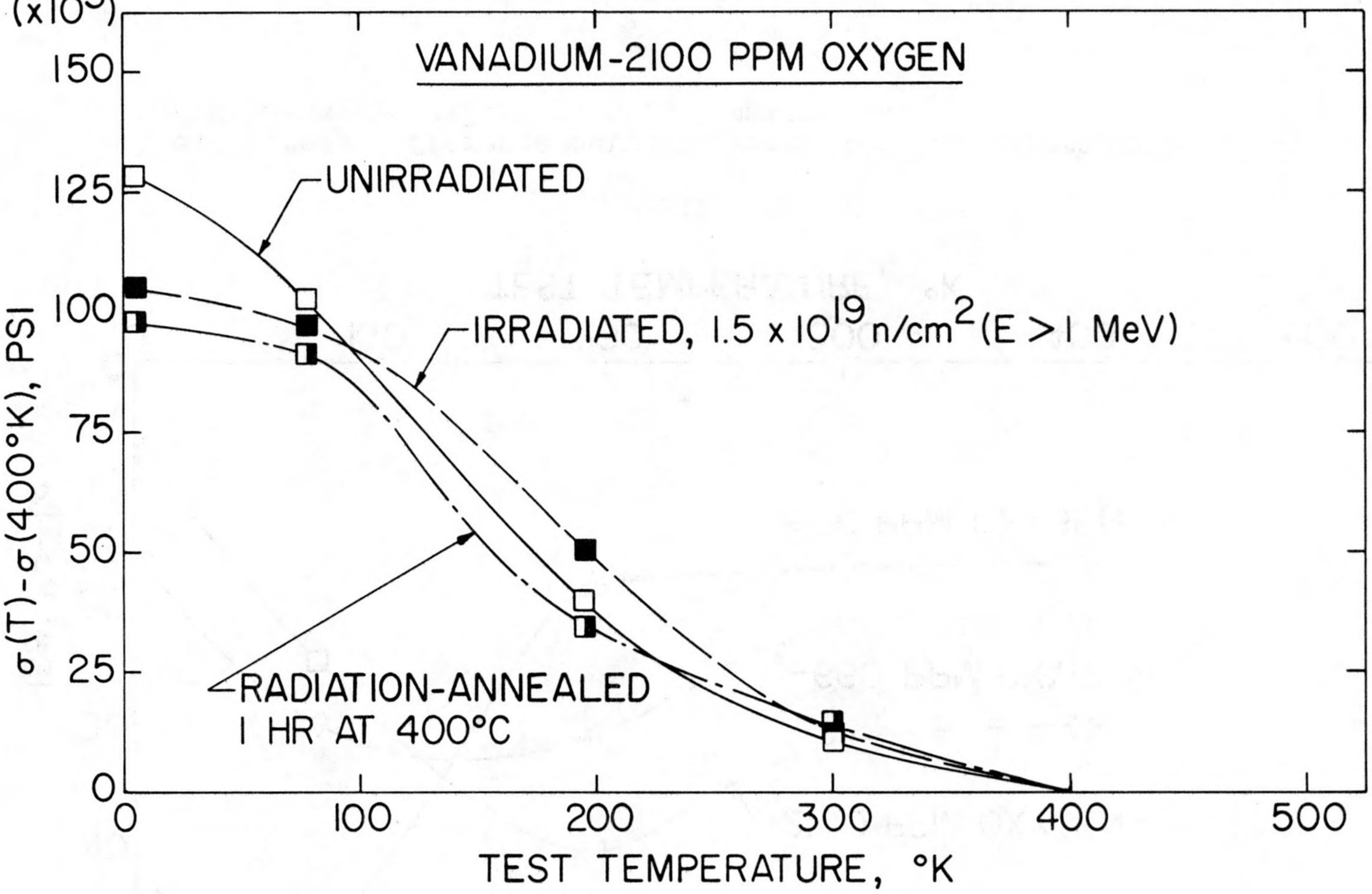

FIG. 9

Thermal component of yield stress vs test temperature for vanadium-2100 ppm oxygen in the unirradiated, as-irradiated (1.5 x 10^{19} n/cm^2 at 85°C), and radiation annealed (1 hr at 400°C) conditions.

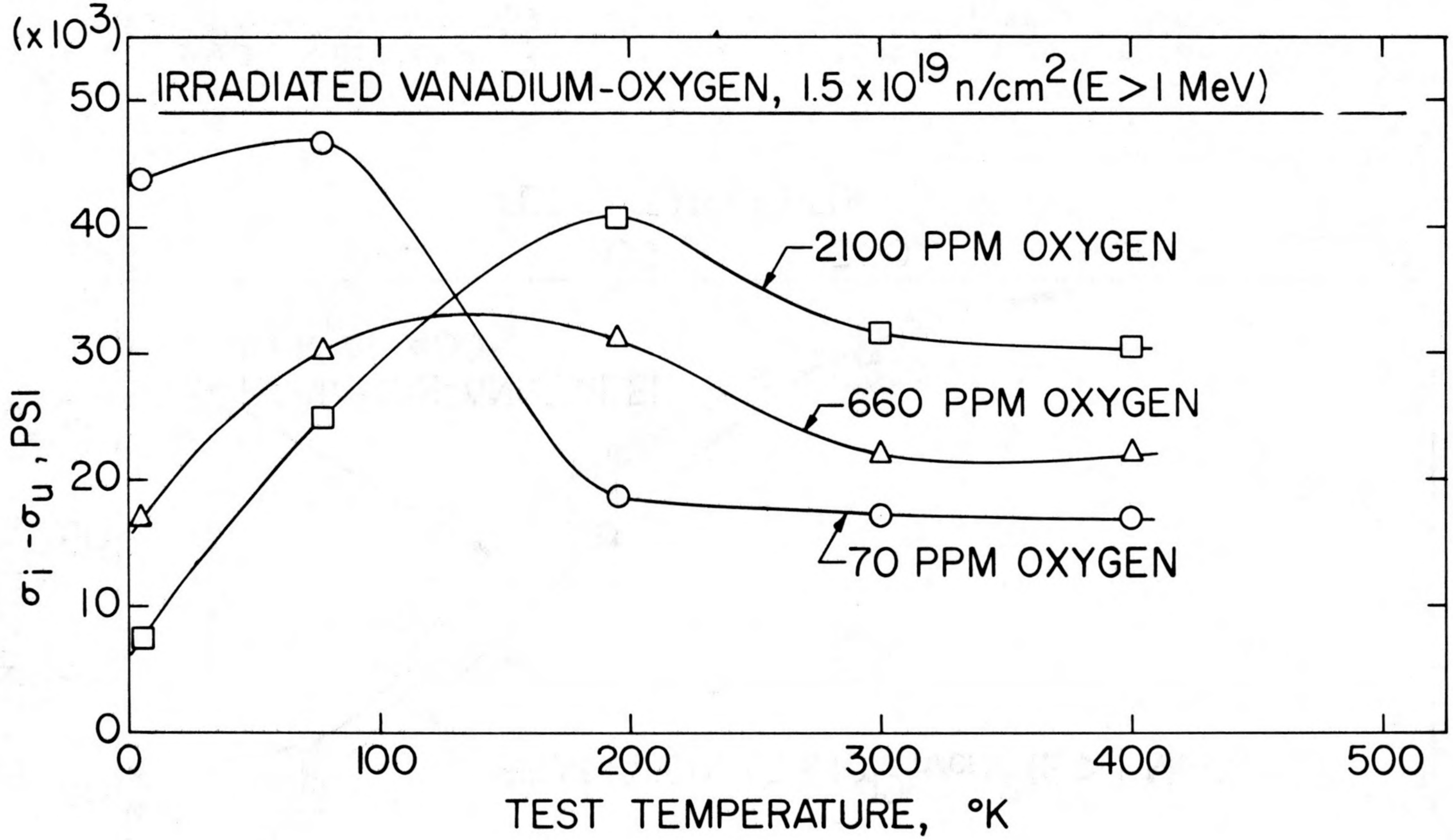

FIG. 10

Yield stress difference between irradiated and unirradiated vanadium-oxygen alloys vs test temperature.