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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

Technical Progress Report

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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

ABSTRACT

During the past contract year, the following projects have been completed and are in various stages of write-up: (a) alloy hardening and softening in body centered cubic metals; (b) carbon-vacancy interactions in austenitic alloys; and (c) Snoek anisotropy in body centered cubic metals. In the same period two new programs have been initiated on: (a) effects of solute gradients on strengthening in materials; and (b) mechanical behavior of hydrogenated body centered cubic metals. In the completed programs, the new results obtained include: (a) the ubiquitous role of solute interactions in causing alloy softening in Nb and Ta base alloys has been verified experimentally and has been rationalized analytically; (b) c-v binding in austenitic alloys has been found to be large (~ 0.4 eV) from damping studies and analysis of diffusion data; (c) single crystal anisotropy of the Snoek phenomena in Ta-Re-N and Ta-Re-O alloys, coupled with thermodynamic studies of these alloys using damping techniques, has permitted detailed knowledge of substitutional-interstitial solute interactions; and (d) the relaxation strength of the C Snoek spectrum in textured commercial steels has been successfully rationalized from complementary studies of the inverse pole figures. Results from the newly initiated programs, which are only preliminary at present, are described briefly.

DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

A. Alloys Hardening and Softening in Body Centered Cubic Metals

In previous contract periods, we have reported that various alloys of Nb and Ta exhibit alloy hardening if a solute, either substitutional or interstitial, is added to the high purity metal. Yet, it was generally found that the same solute, if added to an impure metal or to a high purity metal to which another interstitial solute had also been added, can cause the phenomenon of alloy softening at temperatures $T \lesssim 0.15 T_m$, where T_m is the melting temperature. Alloy softening is generally observed as a decrease in the yield or flow stress of a material with respect to the addition of a particular solute. A typical result from work that we have done on Nb-W alloys intentionally contaminated with O is given in Fig. 1.

In the present contract period with the completion of the Ph. D. thesis of A.K. Vasudevan, we have completed our investigations on alloy hardening and softening in many Nb and Ta alloys. Results such as those given in Fig. 1 are completely general for all materials investigated. All of the following high purity binary alloys exhibited only alloy hardening at all test temperatures: Nb-O, Nb-N, Nb-C, Nb-H, Nb-Mo, Nb-W, Nb-Re. All of the following impure binary alloys exhibited alloy softening at $T \lesssim 0.15 T_m$: Nb-O, Nb-Mo, Ta-O, Ta-N. All of the following high purity ternary alloys also exhibited alloy softening at $T \lesssim 0.15 T_m$: Nb-O-N, Nb-O-H, Nb-O-C, Nb-Mo-O, Nb-Mo-C, Nb-W-O, Nb-Mo-Re, Ta-O-N. In the last two categories, alloy softening was not observed at temperatures approximately $T \gtrsim 0.15 T_m$, consistent with the re-

sults obtained in other less systematically studied bcc metals such as V, Mo, W, Cr, Fe and K.

From results such as these we have been able to conclude that interactions between various solute atoms, at least one of which is usually an interstitial solute, are responsible for alloy softening in bcc materials. The solute interactions appear to influence mainly the distribution of thermally activatable obstacles on a dislocation glide plane, although in several alloys the accompanying decrease in the total number of obstacles has an additional large importance. This conclusion comes from detailed analysis of the general equation (1).

$$\tau = \alpha\beta\gamma\mu/L$$

where: α is a function that describes the orientation, shape and distribution of obstacles; β is the obstacle distortion; γ is a function that describes the character of the moving dislocation; μ is the shear modulus; and L is the obstacle spacing. Our analysis, presented in preliminary form in Vasudevan's thesis, indicate that α often varies significantly between the binary and ternary alloys we have investigated.

Most of the detailed results, analyses and interpretations have been presented in other recent contract reports, theses and publications and are not reproduced here. (See the enclosed listing at the end of the report.) The remaining analytical and interpretive efforts in this area should be largely completed in the next year. Complete analyses of the large body of results obtained by K.V. Ravi, M.G. Ulitchny and A.K. Vasudevan in their theses research is anticipated.

B. Effects of Solute Gradients on Strengthening in BCC Materials.

A logical extension of the research described in section A on effects of microscopic or atomic level solute distribution on strengthening is to consider effects of larger, macroscopic solute gradients. Such effects are or can be of importance in many practical materials problems, e.g., coatings on materials; surface oxidation; decarburization or volatilization in service environments; composite materials etc. In this section we describe our initial progress in such a program initiated during the past year by Mr. V.K. Sethi.

The system selected for investigation is oxygen in niobium for the reasons that

(a) hardening caused by homogeneously distributed oxygen in niobium is well documented (2-4);

(b) the techniques (solid state outgassing) for purification of niobium have been perfected in our laboratories to give us interstitial impurity contents of less than a few atomic ppm (parts per million); and

(c) the degree of homogenization of oxygen can be controlled easily (as described elsewhere in this section).

Niobium single crystals, oriented near the center of the stereographic triangle, are grown in the form of ~ 25 cm long, and ~ 3 mm diameter rods. Tensile specimens are prepared from these rods by centerless grinding, and are then chemically polished to remove surface damage. The tensile specimens are then purified by heating at $\sim 2200^\circ\text{C}$ in vacuum ($\sim 10^{-9}$ mm Hg). Residual resistivities of ~ 2000 have been measured indicating that

the amount of interstitial impurities is less than a few at.ppm. Controlled amounts of oxygen are added using anodic oxidation⁽⁵⁾, followed by a diffusion anneal in vacuum at 1000°C. The gradients are obtained by varying the diffusion anneal time. Figure 2 shows a few gradients, the distribution is calculated from the series solution of the diffusion equation in cylindrical co-ordinates. Microhardness measurements across the cross-section of the specimen are being used to check the distribution profiles. Figure 3 shows the microhardness vs. oxygen concentration curve which we plan to use as our standard curve. The possibility of using the microhardness measurements made at lower temperatures^(6,7), where the effects of oxygen are expected to be larger is being looked into.

Tensile specimens, with characterized gradients are tested at a base shear strain rate of $\sim 7.5 \times 10^{-4}$ sec at a wide range of temperatures. A few of the shear stress vs. shear strain curves for some gradients tested at 77°K are shown in Figure 4 (different gradients are labeled with the diffusion anneal times). Figure 5 shows the variation of yield strength with diffusion anneal time for the specimens anodized at 40V (C_H - concentration on homogenization ~ 600 at.ppm), 20V ($C_H \sim 400$ ppm) and pure Nb. Pure Nb specimens were given the same treatment as anodized specimens. This was done to make sure that the effects we are seeing are truly caused by gradients, and not by anything which is picked up by the specimens during the diffusion anneal.

Figure 5 shows that with decreasing diffusion anneal time the yield strength is increased for the same total amount of oxy-

gen and a maximum exists in the vicinity of ~ 200 sec below which the yield strength drops very rapidly to values close to that of pure niobium. Such a behavior was expected, and can be explained on the basis of a model which considers the specimen with the gradient as a composite of a strong case and a weak core. Decreasing diffusion anneal times correspond to decreasing the ratio of the cross-sectional area of the case to the cross-sectional area of the core. Such a model was proposed for composite-materials by Hecker and Hamilton⁽⁸⁾, and we hope we can use it to explain our data on the macroscopic level.

The theoretical basis of strengthening under solute gradients are not well understood. The major source of knowledge comes from a few theoretical predictions:

a) computer calculations indicate a 20% strengthening effect if point obstacles on a glide plane are in an ordered array instead of being distributed randomly^(9,10);

b) Fleischer⁽⁹⁾ argues that shape of microgradients, and concentration dependence of the yield stress for the point obstacles involved determine the extent of softening or strengthening;

c) Fleischer⁽¹¹⁾ has also analyzed dislocation-level strengthening when macroscopic gradients are present. His analyses suggest that the strengthening observed will depend sensitively on the type of defect distortion (e.g. cubic vs tetragonal), the detail shape of the dislocation - solute atom interaction potential, and the orientation of the distortion relative to the moving dislocation. To what extent these predictions are compatible with macroscopic theories concerning rules of mixtures for composite type materi-

als⁽⁸⁾ has to be tested.

When all the data at different temperatures, diffusion anneal times, and various amounts of oxygen is obtained we hope to establish (1) the extent to which these various types of gradients cause hardening, softening or no effect on mechanical strength and (2) the extent to which the different theoretical predictions are (or should be) internally consistent.

C. Mechanical Behavior of Hydrogenated Body Centered Cubic Metals

Nb-H is one of several alloy systems in which we have noted alloy softening when a second solute atom (e.g. O) is added. We have also seen that the solute interactions have important effects on the hydrogen embrittlement characteristics of Nb, although our present results have been limited in the range of temperature, strain rate and composition over which they have been made^(12,13). Because recent investigations, e.g. by Birnbaum and by Arsenault and their co-workers⁽¹⁴⁾, confirm the importance of our previous work in understanding hydrogen strengthening and embrittlement phenomena, we have chosen to expand our efforts in these areas.

During the past year we have initiated an investigation to examine effects of various solutes systematically added to Nb-H alloys on hydrogen strengthening and embrittlement. Emphasis is being placed on effects of solutes on hydrogen solubility and hydride precipitation and on mechanical behavior during temperature and strain rate cycling. The program was initiated by Mr. T. Ramsey, who unfortunately had to discontinue his graduate studies recently. The program is being continued by Dr. A.A. Sagues as a post doctoral program. All base metal specimens for the inves-

tigation have been prepared and initial experiments are underway.

D. Carbon-Vacancy Interactions in Austenitic Alloys

Mr. John Slane has completed a M.S. Program in which he has been able to verify and extend the previous work of Ulitchny and Gibala⁽¹⁵⁾ in which it was argued that the C-V interaction energy in austenite is probably large, viz. ~ 9 kcal/mole.

He has demonstrated that the carbon "Snoek-type" interstitial relaxation peak in Fe-Ni-C austenites is enhanced by 2 mev electron irradiation near ambient temperature. (See Fig. 6.) This is consistent with efficient trapping of vacancies by carbon atoms into C-V clusters which contribute to the relaxation process.

He has also quantitatively analyzed diffusion data by Mead and Birchenall⁽¹⁶⁾ and Gruzin et.al.⁽¹⁷⁾ and has shown that the enhancement b of self-diffusion in fcc Fe by C additions, is given by

$$D(C) = D(O) [1 + bC]$$

where: $D(C)$ and $D(O)$ are the self diffusion coefficients in the carbon-containing and carbon-free γ -Fe respectively; C is the atomic fraction of carbon; and b is approximately given by

$$b \sim 2 \left[\exp \left(\frac{\Delta g}{kT} \right) - 1 \right]$$

for the limiting assumption that the jump frequency of a vacancy is not affected by the presence of carbon*. Slane has determined that Δg , the binding free energy of C-V pairs, is 9.5 kcal/mole.

* A much more detailed analysis is given in Slane's thesis.

This work lends strong support to the supposed importance of C-V interactions in irradiation swelling of stainless steels in reactor environments⁽¹⁵⁾. However, it will not be continued beyond normal publication of these results.

E. Snoek Anisotropy in Body Centered Cubic Metals

We have made extensive use of Snoek anelasticity in bcc metals as a means of characterizing the interstitial solid solutions for which we have investigated the mechanical behavior. Two related programs in this area have been completed in the past year.

1. Single crystal anisotropy of Snoek Anelasticity in Ta-Re alloys.

We have previously reported our results of a complete study of the concentration, temperature and orientation dependence of N and O Snoek anelasticity in Ta-Re alloys. These results, obtained by Dr. A.A. Sagues, have now been completely analyzed and show that (a) single N atoms, ReN pairs, Re₂N triplets, single O atoms and Re_XO clusters (where X = 1,2, and possibly 3) relax in these alloys, (b) the binding enthalpies are of the order 3-6 kcal/mole, and (c) the relaxation strength of N or O in the clusters is as large as or slightly larger than that of the single interstitial.

We have been able to combine such thermodynamic data with data on the single crystal anisotropy to determine the most likely cluster configurations in the alloys. Figs. 7-9 show this in one way for ReN or ReO clusters. Fig. 7a gives the orientation dependence of the ReN relaxation which is entirely consistent with

the defects DA or DC in Fig. 8a, but the detailed nature of the interstitial dipole distortion⁽¹⁸⁾ makes possible the occurrence of DB relaxators. However, our data on the concentration and temperature dependence of this relaxation (Fig. 9) is more consistent with DA occupancy. In a similar way, we can argue that the defect ECF is the most likely Re₂N relaxator.

These various results have now been published⁽⁹⁾, are in press, or are soon to be submitted for publication.

2. Effects of preferred orientation on Snoek phenomena in polycrystalline materials.

Because it is often necessary to use polycrystalline specimens to apply Snoek anelasticity to various problems, we have examined the role of preferred orientation on the relaxation strength. A first series of experiments on commercial steels has been completed by Mr. R.P. Krupitzer and demonstrates that the relaxation strength of a textured polycrystalline material can be correlated in detail with its inverse pole figure through a texture factor F defined as

$$F = 100 \sum_{i=1}^n \left(\frac{X}{X_{\max}} \right)_i f_i$$

Here $(X/X_{\max})_i$ is the fractional contribution of a given orientation of crystallite to the total relaxation strength relative to its maximum possible contribution if it were oriented for maximum damping and f_i is the volume fraction of that crystallite as determined by inverse pole figure analysis. ‡

Fig. 10 gives one example of the type of correlation ob-

‡Details of this analysis will appear in a later report.

tained. For a Si steel, for which the orientation to the rolling direction could be varied by large amounts, one sees that the relaxation strength and the texture factor F vary quantitatively in the same manner. Similar results have been obtained for three other steels.

We are now able to apply this analysis to any polycrystalline material for which we have obtained the inverse pole figure.

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EFFORT DISTRIBUTION OF THE PRINCIPAL INVESTIGATOR

During the contract period February 1, 1973 to January 31, 1974, the principal investigator, Professor Ronald Gibala, has allotted his time to Contract No. AT(11-1)-1676 as follows:

1. February 1, 1973 - May 31, 1973: 35% of these four months of the academic year. Of this amount, 30% has been financed by the Contract and 5% represents the University's cost sharing.
2. June 1, 1973 - July 31, 1973: 70% of these two months of the summer session.
3. August 1, 1973 - January 31, 1974 (estimated): 30% of these six months while Professor Gibala is on sabbatical leave. During this time he will be analyzing and writing-up several papers based on the Contract research. None of this time is charged to the Contract. 50% is supported by the University and 50% by the Centre d'Etudes Nucleaires de Grenoble, Grenoble, France, where Professor Gibala is located during the sabbatical.

AEC Contract No. AT(11-1)-1676

A SUMMARY OF REPORTS SUBMITTED TO
THE UNITED STATES ATOMIC ENERGY COMMISSION

<u>Report No.</u>	
C00-1676-1	R. Gibala, F. Povolo, M.G. Ulitchny, and K.V. Ravi, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1967.
C00-1676-2	R. Gibala, Comments on the Cold-Work Peak in Alpha Iron, October 31, 1967.
C00-1676-3	R. Gibala and C.A. Wert, Interstitial Alloys of BCC Metals, October 31, 1967.
C00-1676-4	R. Gibala, F. Povolo, M.G. Ulitchny, and K.V. Ravi Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1968.
C00-1676-5	F. Povolo, Comment on the Influence of Hydrogen Content on the Relaxation Spectrum of α Titanium, November 7, 1968.
C00-1676-6	F. Povolo and R. Gibala, A Marx Three Component Oscillator for Internal Friction Measurements at Low and High Temperatures in High Vacuum, January 8, 1969.
C00-1676-7	K.V. Ravi and R. Gibala, The Strength and Alloy Softening of BCC Metals, May 30, 1969.
C00-1676-8	R. Gibala, F. Povolo, M.G. Ulitchny, K.V. Ravi, and G.J. Klems, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1969.
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C00-1676-10	K.V. Ravi and R. Gibala, The Strength of Niobium-Oxygen Solid Solutions, December 20, 1969.
C00-1676-11	K.V. Ravi and R. Gibala, Low Temperature Strengthening in Niobium-Hydrogen Single Crystals, May 27, 1970.
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- C00-1676-16 A.A. Sagues and R. Gibala, Substitutional-Interstitial Solute Interactions in Ta-Re-N Solid Solutions, July 14, 1971.
- C00-1676-17 R. Gibala, A Report on the Discussion Meeting on Defects in Refractory Metals, at Mol, Belgium on September 20-22, 1971, October 31, 1971.
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AEC Contract No. AT(11-1)-1676

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20. R. Gibala and T.E. Mitchell, "Solid Solution Softening and Hardening", Scripta Met., in press.
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Fourteen other papers in preparation or submitted.

AEC Contract No. AT(11-1)-1676

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14. R. Gibala, "Alloy Softening in Body-Centered Cubic Materials", Cleveland Section, AIME Meeting, October 1971.

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16. A.K. Vasudevan and R. Gibala, "Alloy Softening in Nb-Mo-O Single Crystals", AIME Meeting, Detroit, October 1971.
17. M.G. Ulitchny and R. Gibala, "Mechanical Behavior of Niobium-base Interstitial Solid Solutions", AIME Meeting, Detroit, October 1971.
18. G.J. Klems, R.E. Miner, F.A. Hultgren and R. Gibala, "Internal Friction in Twinned Fe-Ni-C Martensites", AIME Meeting, Detroit, October 1971.
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20. M.G. Ulitchny and R. Gibala, "Internal Friction and Strain Aging Behavior of Ferrous Austenites", AIME Meeting, Boston, May 1972.
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24. R. Gibala, "The Strength Differential Effect in Niobium-Oxygen Solid Solutions", ONR Workshop on the Strength Differential Effect in Metals and Polymers, Boston, June 1972.
25. R. Gibala, "The Strength and Structure of Solid Solutions", General Electric Research and Development Laboratory, Schenectady, N.Y., November 1972.
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29. R. Gibala, "Hydrogen-Defect Interactions in Iron-Base Alloys", International Conference on Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Base Alloys, Unieux-Firminy, France, June, 1973.
30. M.G. Ulitchny, A.K. Vasudevan and R. Gibala, "Solution Hardening and Softening in Nb-Base Solid Solutions", Third International Conference on the Strength of Metals and Alloys, Cambridge, England, August, 1973.
31. A.A. Sagues and R. Gibala, "Crystallographic Orientation Dependence of Interstitial Anelasticity in Ta-Re-N and Ta-Re-O Alloys", Fifth International Conference on Internal Friction and Ultrasonic Attenuation in Crystalline Solids, Aachen, Germany, August, 1973.
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32. M.G. Ulitchny and R. Gibala, "Mechanical Properties of Nb Single Crystals Containing H and H+O", International Conference on "Hydrogen in Metals: Effects on Properties, Selection and Design", Seven Springs, Champion, Pa., September, 1973.

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1. M.G. Ulitchny (M.S., 1969), "Internal Friction and Strain Aging of Austenite".
2. K.V. Ravi (Ph.D., 1969), "The Strength of Niobium-Oxygen Solid Solutions".
3. G.J. Klems (Ph.D., 1971), "Internal Friction of Ferrous Martensites".
4. M.G. Ulitchny (Ph.D., 1972), "Mechanical Behavior of Interstitial Solid Solutions of Niobium".
5. A.A. Sagues (Ph.D., 1972), "Substitutional-Interstitial Solute Atom Interactions in Ta-Re-N and Ta-Re-O Solid Solutions".
6. J.A. Slane (M.S., 1973), "Carbon-Vacancy Interactions in Austenites".
7. R.P. Krupitzer (M.S., 1973), "Effects of Preferred Orientation on Snoek Phenomena in Commercial Steels".
8. A.K. Vasudevan (Ph.D., 1974), "Mechanical Behavior of Single Crystals of Niobium-Base Alloys".

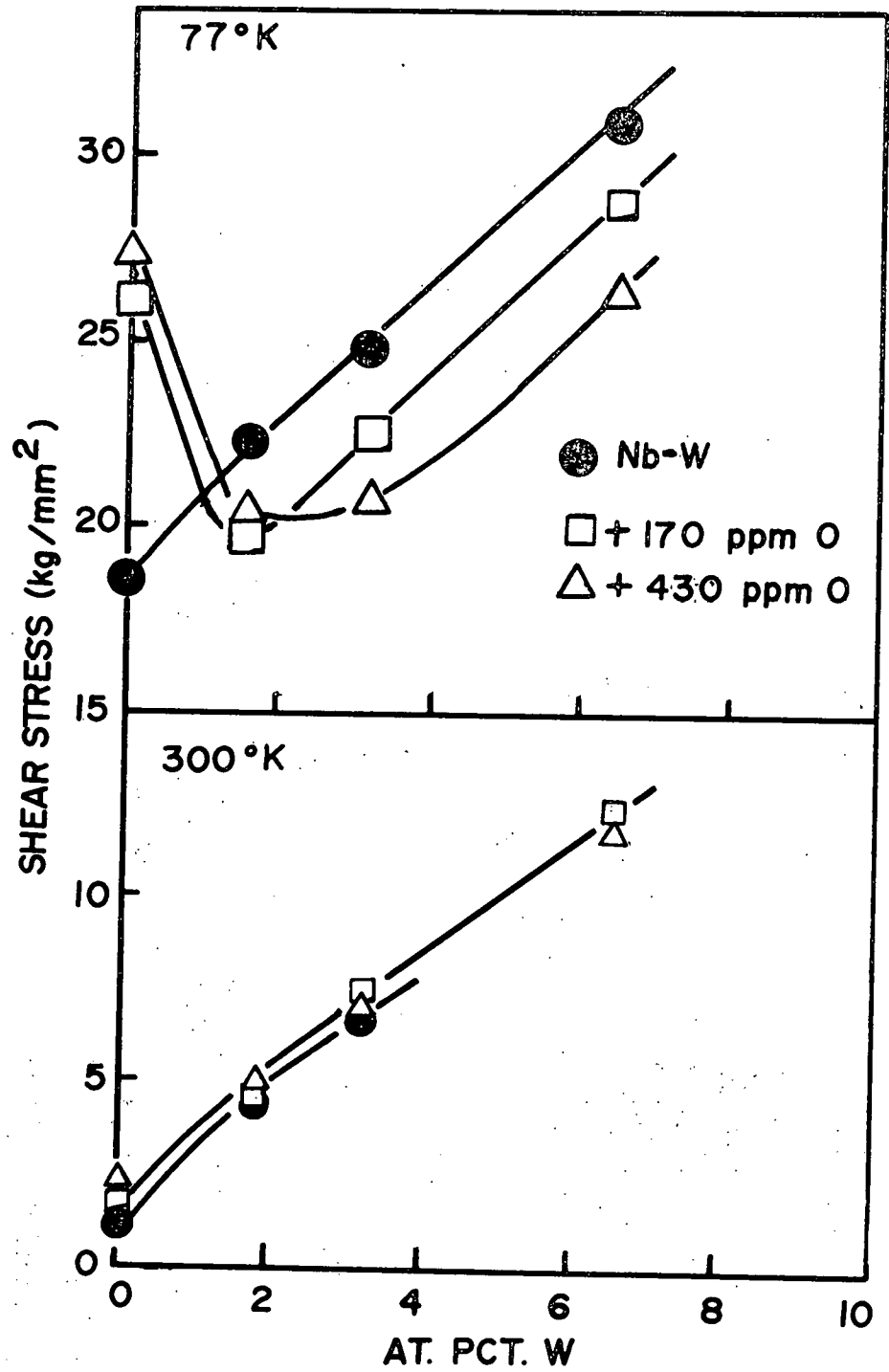


Fig. 1 Effect of tungsten concentration on shear stress at 77°K and 300°K for Nb-O-W alloys.

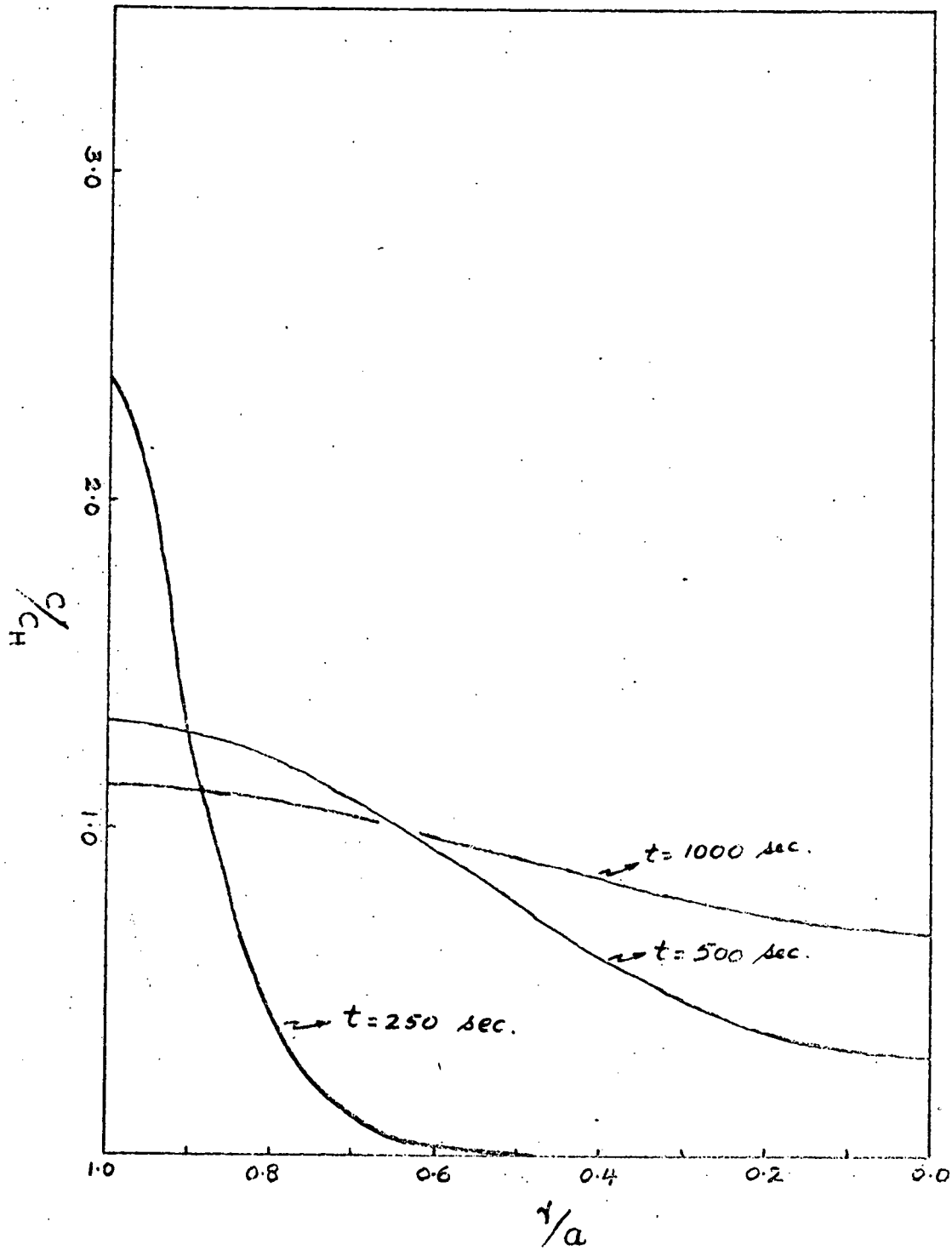


Fig. 2 Concentration profiles for various diffusion anneal times. C - concentration at a distance r from the center of the cylindrical specimen of radius a . C_H - concentration on homogenization.

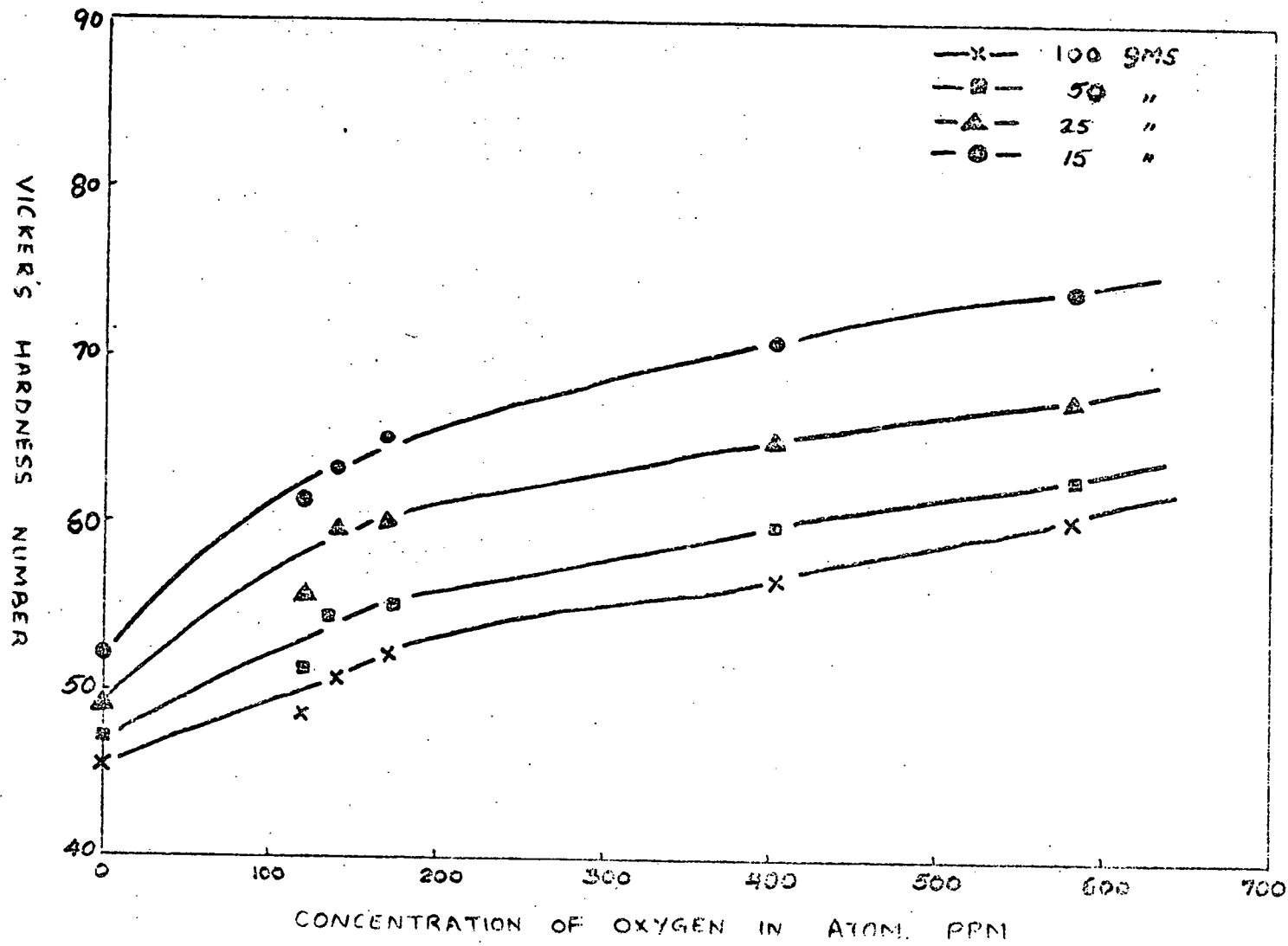


Fig. 3 Effect of oxygen on Vicker's Hardness of niobium for various loads.

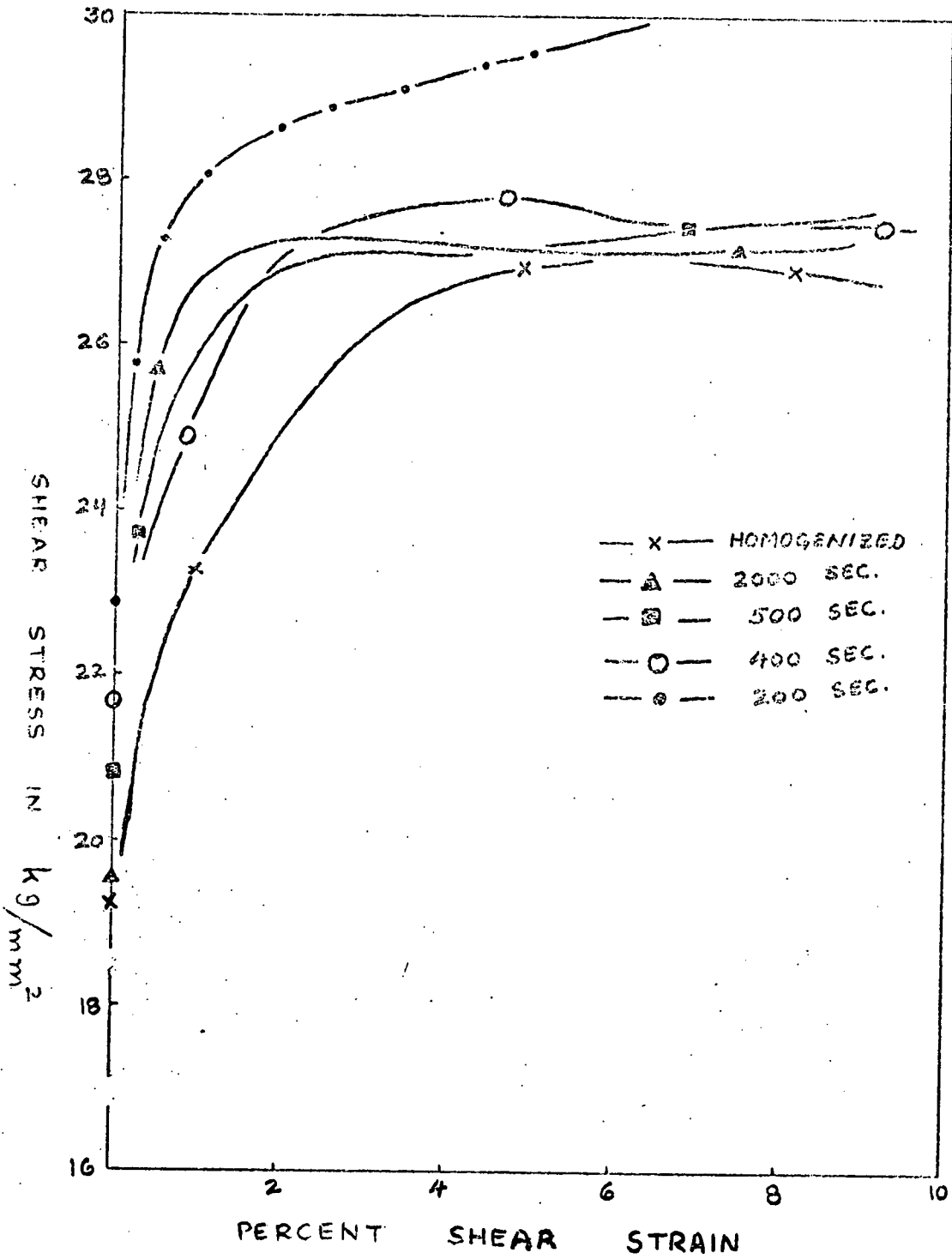


Fig. 4 Resolved shear stress vs. Resolved shear strain for niobium specimens tested in tension at 77°K for various diffusion anneal times. $C_H = 600$ atom ppm.

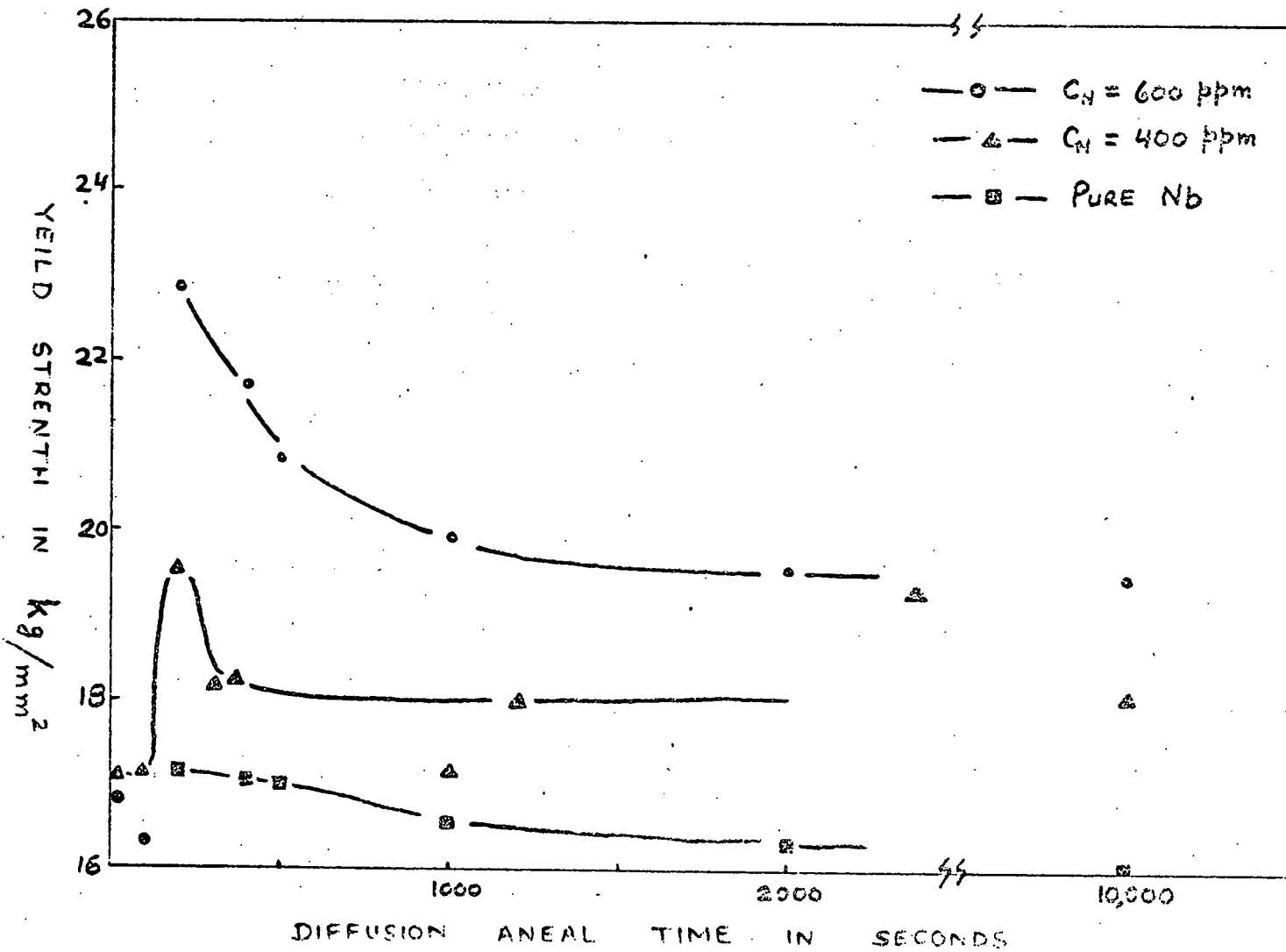


Fig. 5 Yield strength vs. diffusion anneal time for different initial concentrations of oxygen in niobium C_H - concentration on homogenization.

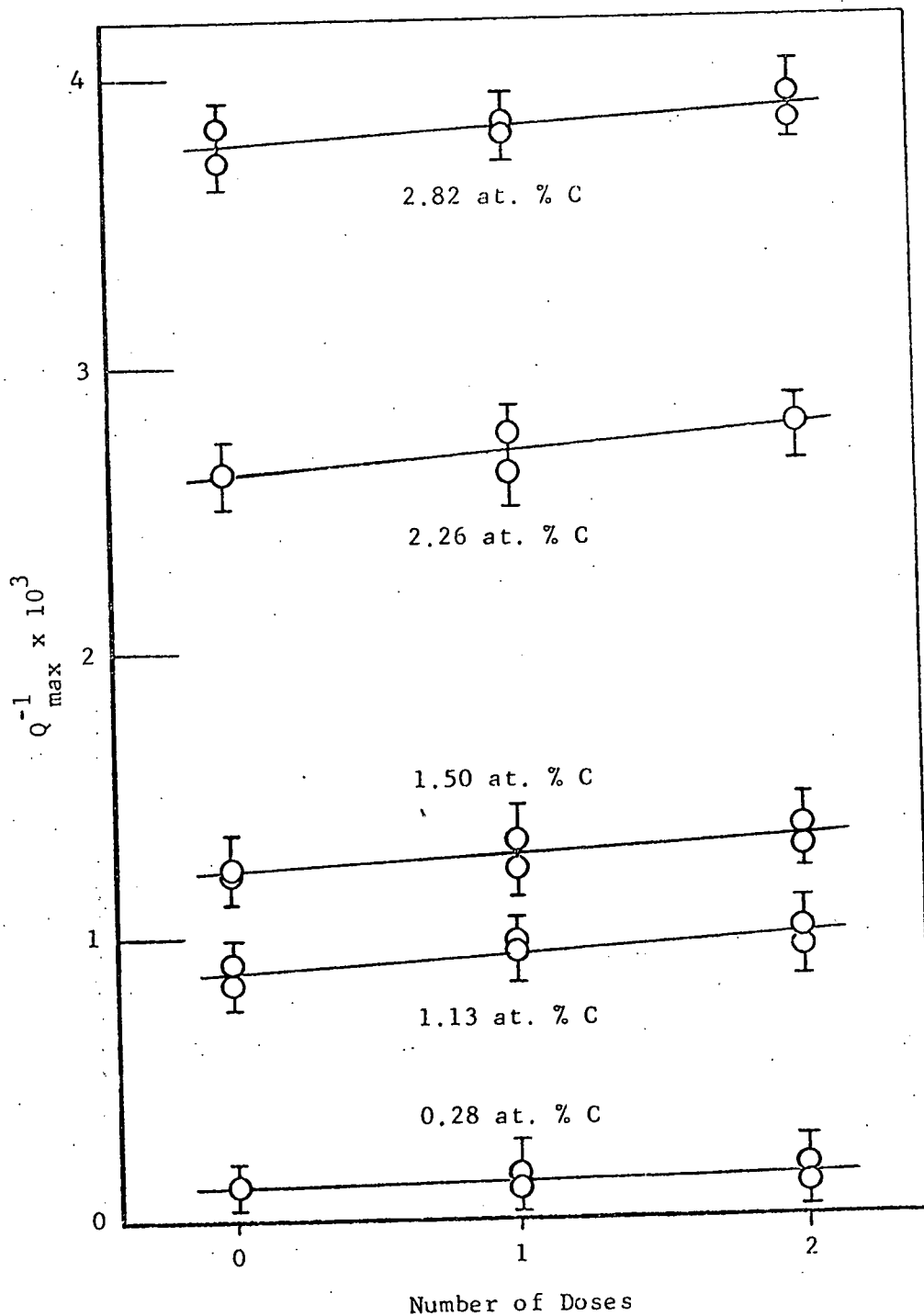


Fig. 6 Internal friction peak height versus 2 Mev electron irradiation flux for quenched Fe-36% Ni with various carbon contents.

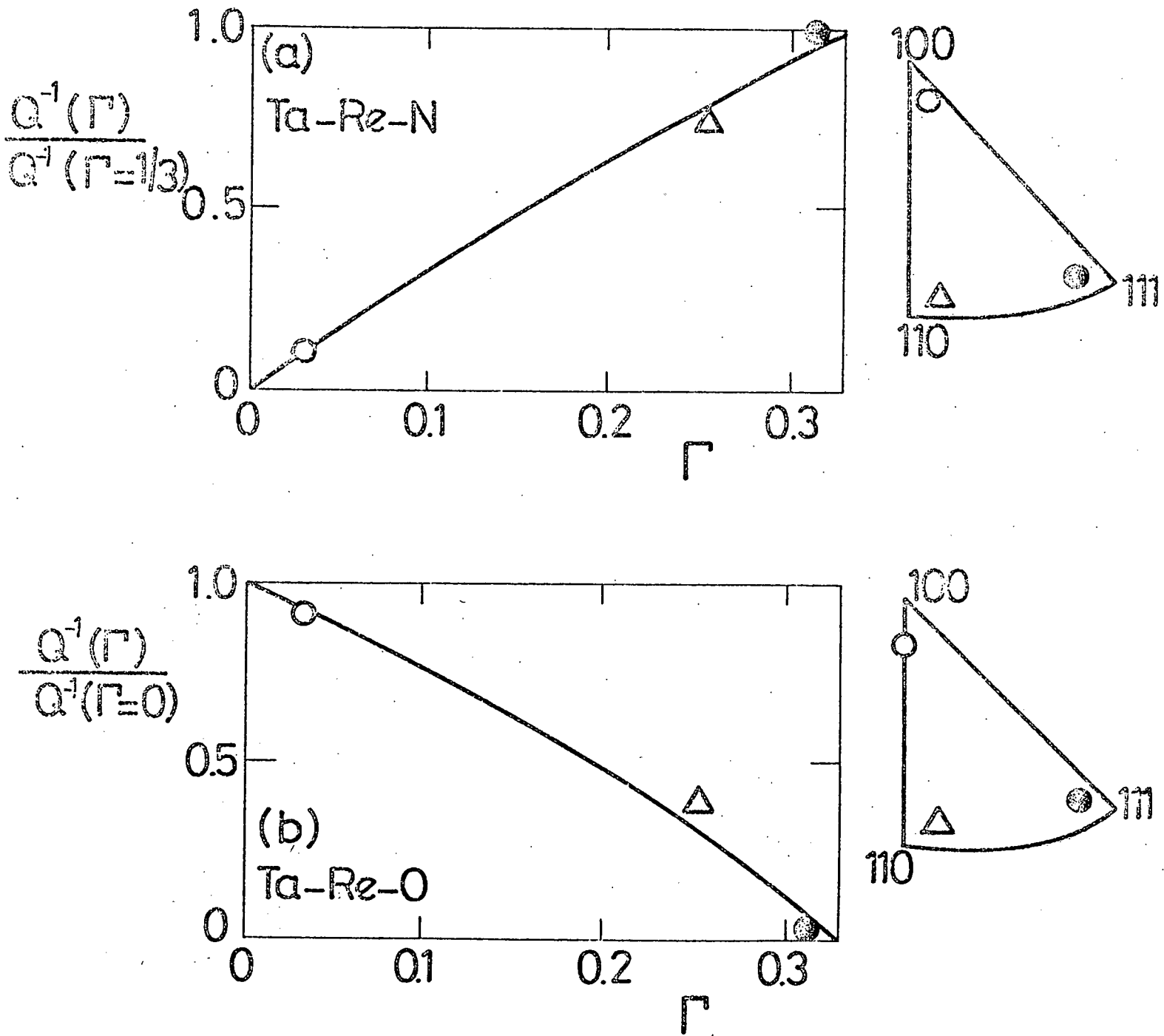


Fig. 7 The orientation dependence of the entire Snoek spectra in Ta-Re-N and Ta-Re-O alloys. The results (a) were torsional experiments; the results (b) were longitudinal experiments.

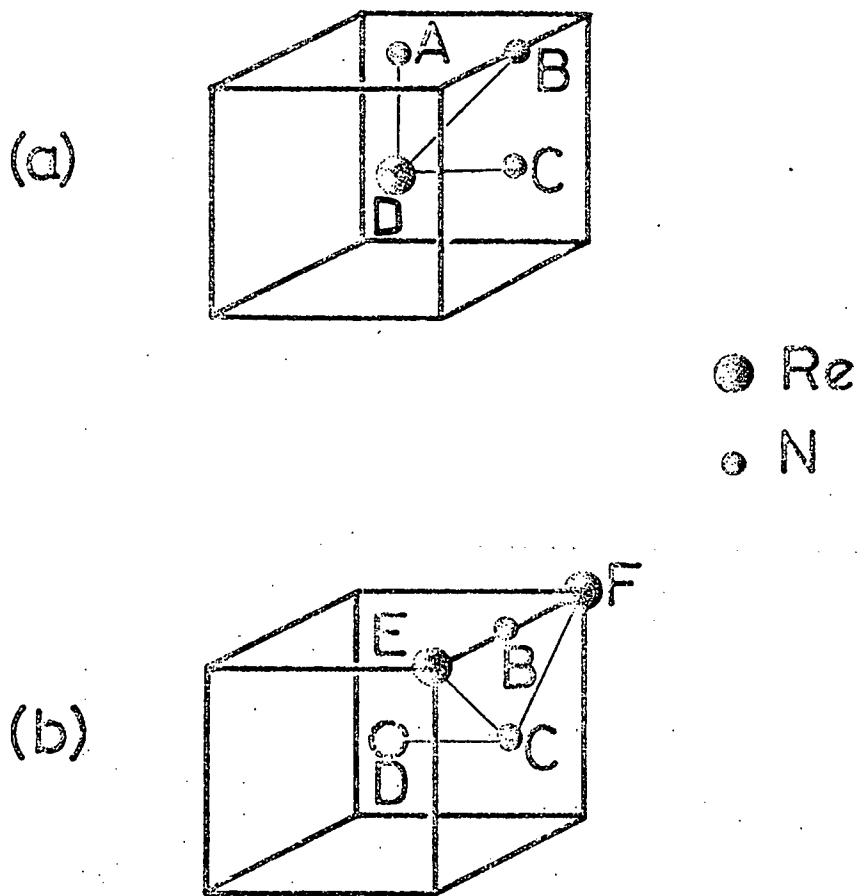


Fig. 8 Possible ReN and Re_2N defects which exhibit anelastic relaxations in Ta.

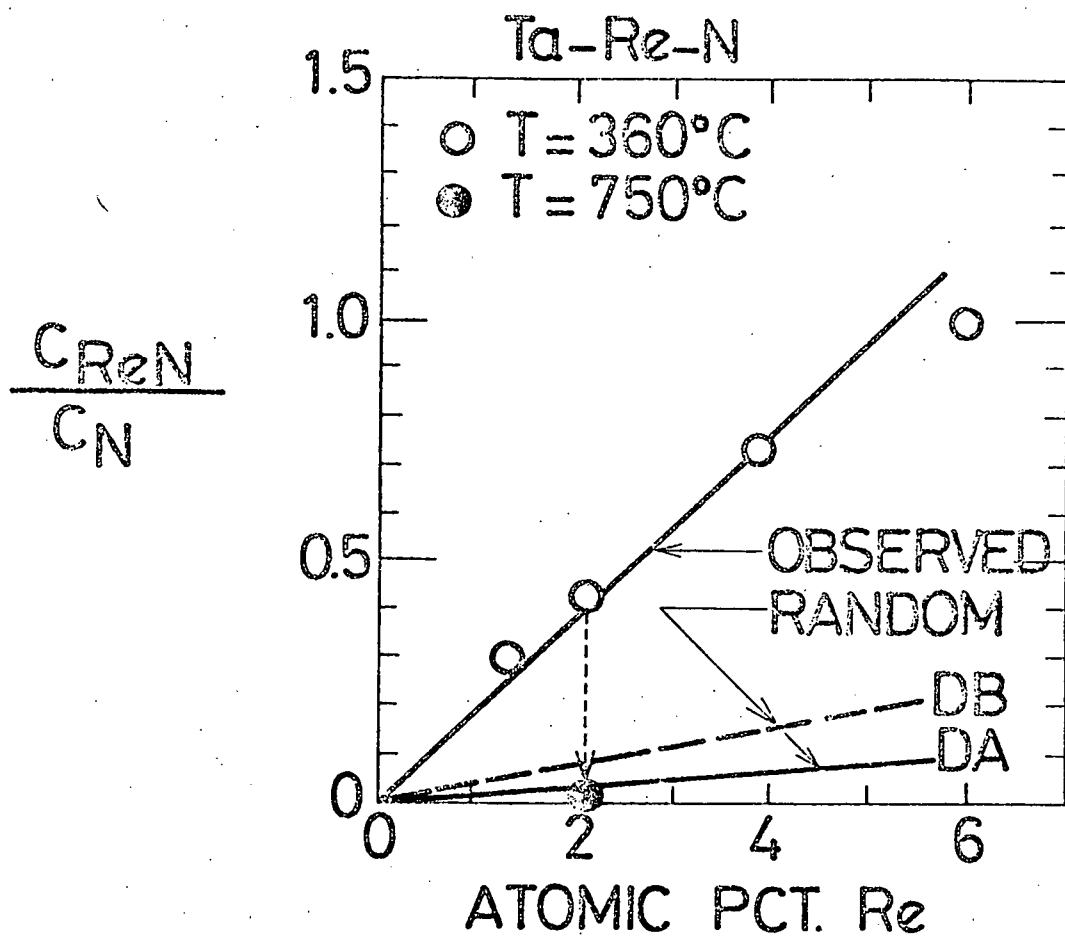


Fig. 9 The relative relaxation strength of the ReN relaxation in Ta-Re-N alloys at different temperatures of measurement.

