

AD-A108 654

ILLINOIS UNIV AT URBANA DEPT OF METALLURGY AND MINING--ETC F/G 11/6
THE EFFECT OF HYDROGEN ON THE SOLID SOLUTION STRENGTHENING AND --ETC(11)
NOV 81 J EASTMAN, F HEUBAUM, T MATSUMOTO N00014-75-C-1012

UNCLASSIFIED

NL

1-1

1-1

1-1

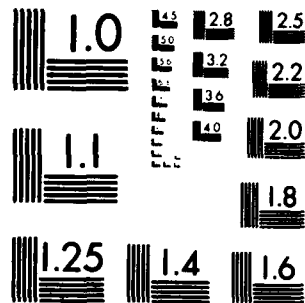
END

DATE

FORMED

1 1/2

DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

LEVEL

2

THE EFFECT OF HYDROGEN ON THE SOLID SOLUTION STRENGTHENING
AND SOFTENING OF NICKEL

AD A108654

J. Eastman, F. Heubaum, T. Matsumoto and H.K. Birnbaum

University of Illinois at Urbana- Champaign
Department of Metallurgy and Mining Engineering
Urbana, Illinois 61801

Contract N00014-75-C-1012

TECHNICAL REPORT
OFFICE OF NAVAL RESEARCH
November 1981

DTIC
ELECTE
DEC 17 1981
S D D

100% COPY

This document is unclassified. Reproduction and distribution is permitted for any purpose of the U. S. government.

DISTRIBUTION STATEMENT A

Approved for public release;
Distribution Unlimited

81 11 30 025

ABSTRACT

The effects of hydrogen on the plastic deformation of nickel and nickel-carbon alloys were studied using plastic deformation techniques over a wide range of strain rates at about 300 K. The emphasis of the study was on the behavior at very low strains and low strain rates. Hydrogen was introduced as a solute element by quenching from a gaseous H_2 atmosphere or by testing in a gaseous H_2 atmosphere. The behavior of a number of different purities of nickel with hydrogen additions was examined. The most significant impurity element seemed to be C and this element was varied over a wide composition range by annealing in different atmospheres. Both solution softening and solution strengthening was observed depending on the amount of H in solution relative to the amount of C in solution. The nature of this solution softening effect is discussed.

Accession For	
NTIS GRA&I	☒
DTIC TAB	☐
Unannounced	☐
Justification	
By <i>Per Ltr. on file</i>	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
<i>A</i>	

THE EFFECT OF HYDROGEN ON THE SOLID SOLUTION STRENGTHENING AND SOFTENING OF NICKEL

J. Eastman, F. Heubaum, T. Matsumoto and H. K. Birnbaum

University of Illinois

Department of Metallurgy and Mining Engineering

Urbana, Illinois 61801

INTRODUCTION

The effect of hydrogen on the mechanical properties of nickel has been extensively studied in the temperature range 77-400K (1-9). The most drastic effect observed is the decrease of ductility and change of fracture mode from ductile to "brittle" failure which is caused by solute hydrogen or by a hydrogen gas atmosphere. It has been recently suggested (8,10) that this embrittlement results from a ductile fracture mode in which the ductility is enhanced by hydrogen and results in a high degree of local deformation. Solute hydrogen has been shown to cause solid solution strengthening of single and polycrystalline nickel (1-5) and further evidence for the pinning of dislocations in nickel by solute H has been obtained from the observation of serrated yielding (Portevin-LeChatalier effect) (1-6). Prior to the present study there had been no evidence that hydrogen decreased the flow stress in Ni. In our first publication (12) we showed in-situ environmental cell deformation studies in the HVEM which showed increases in dislocation mobility for Ni stressed in gaseous H_2 atmospheres. The only other indication

that hydrogen might solution soften f.c.c. systems is a study of Pd-rare earth alloys (13) which showed pronounced decreases in indentation hardness on adding large amounts of solute H to the binary alloy.

The effect of hydrogen on mechanical properties has been more extensively studied in bcc systems in which strong evidence for dislocation pinning and solid solution strengthening by solute H has been obtained in the group Vb metals Nb, V, Ta in which H has appreciable solid solubility (14,15). In contrast to this behavior, it has recently been shown that H in high purity iron causes solid solution softening in the temperature range 170 - 300 K (16-21) and solid solution strengthening at lower or higher temperatures. In lower purity iron, solid solution strengthening is always observed (22-24). Solid solution softening has also been observed for dilute solutions of C in iron and it has been shown that the effect of the two solutes, C and H, are additive if a critical concentration of solute is not exceeded (25).

The mechanisms for these solid solution effects have not been established. Solution strengthening effects in Ni and the group Vb metals appear to be consistent with the elastic interaction between solute H, acting as a relatively immobile pinning point, and the dislocation line which results in an increase in the thermally activated portion of the flow stress. This relatively straightforward mechanism is complicated by the fact that H is very mobile at the temperature at which solution strengthening occurs and by the precipitation of H as a hydride

in the Group Vb metals in the same temperature range. Stress effects on this precipitation process and possible reorientation of the hydride phase in the stress field of the dislocation can cause some unusual temperature dependences of the flow stress (15).

The mechanism of solution softening in iron appears to be much more complex. It has been suggested (16,26-28) that the solution softening results from a) a decrease of the Peierls stress due to the presence of solute H or C, b) a decrease in the energy required for double kink nucleation on screw dislocations due to H or C, or c) a decrease in the effectiveness of other solute pinning points due to the presence of solute H or C. The first two of these proposed mechanisms are "intrinsic" in nature and depend on the Peierls stress being the rate limiting barrier to dislocation motion. Hence they may have applicability in b.c.c. metals but should not apply in f.c.c. metals where the Peierls stress is considered to be small. Mechanism c) is "extrinsic" in character and should have applicability in both b.c.c. and f.c.c. systems which contain solute pinning points in addition to the added interstitial. The case for this extrinsic mechanism has been presented based on data obtained primarily on the Group Vb metals while the measurements on Fe have been interpreted on the using mechanisms a) or b).

Thus the theoretical understanding of these solid solution effects is rather poor. In the present paper we examine the possibility of obtaining solid solution softening effects due to solute H in nickel, a metal in which the Peierls stress is small

and the thermal portion of the flow stress appears due to short range elastic interactions with solutes and with intersecting dislocations. We will show that solution can be demonstrated for specimens tensile tested in H_2 gas or containing solute H.

EXPERIMENTAL PROCEDURE

Specimen Preparation

A number of different specimen configurations, purities, and heat treatments were used depending on the nature of the tests carried out and these are shown in Table I. To examine the effect of gaseous H_2 on the mechanical properties, 25 micrometer thick sheet specimens having a nominal purity of 99.9% were chosen so that H could diffuse across the thickness during the time of the test (designated 25-). Both the 70% CO - 30% CO_2 and the 95% N_2 - 5% H_2 atmospheres were reducing relative to NiO but the CO - CO_2 mixture added C to the nickel solid solution while the N_2 - H_2 mixture did not change the specimen composition as indicated by analyses performed after the anneals. Metallographic examination revealed a grain size of about 50 micrometers.

Specimens in the form of rods 0.64 cm in diameter (designated R-) were used to study the effect of solute H on the flow curves. These were heat treated as shown in Table I and hydrogen was introduced by quenching from 100 kPa of H_2 gas into silicone oil. The amount of H in solid solution was determined by controlling the quenching temperature. Specimens were stored under liquid N_2 to minimize loss of H solute. High purity Ni specimens, (designated HP-) which contained only 1 appm Fe as a

significant substitutional impurity, were tested in sheet form (0.05 cm thickness) after the heat treatments shown in Table I. These specimens were tested in gaseous atmospheres or after the introduction of solute H by quenching.

Chemical analysis of the specimens was carried out using spark source mass spectrometer and vacuum fusion techniques with the results given in Table II. In some cases duplicate analyses were carried out and these indicated a relatively high degree of reproducibility for the analytical results. For the results discussed in this paper the most significant impurity appears to be the interstitial C, the level of which varied with the source of the Ni and could be controlled by the annealing atmosphere. The H solute concentrations were estimated from the quenching temperature and were checked by vacuum extraction analysis.

Mechanical Property Testing

Tension testing of the specimens was carried out in an Instron machine modified to allow very low strain rate testing (less than 10^{-7} sec^{-1}). These low rates were necessary, particularly in the cases where the testing was done in a H_2 atmosphere, to allow equilibration between the H and the structure developed during the test. All testing was done at about 300 K where the H diffusivity was about $10^{-9} \text{ cm}^2/\text{sec}$ and the diffusion time across the 25 micrometer specimens was of the order of 1.5×10^3 seconds. Since the oxide surface film was ruptured by the plastic deformation it is expected that equilibrium was obtained between the gas phase and the solute H concentration during the deformation. For the tests carried out

at 100 kPa of H_2 gas atmosphere this concentration corresponds to $H/Ni=10^{-5}$. Much higher H concentrations, of the order of $H/Ni = 10^{-3}$, were achieved by quenching the specimens in H_2 gas atmospheres from elevated temperatures.

Since various size specimens were tested, the load cells and load ranges were selected to be suitable for each case. Good reproducibility was obtained for each test with the greatest variability being obtained during testing the thin 25 micrometer specimens. In this case the load cell stability over the period of the test was about $\pm 1\%$ at the very small test loads required. All data reported for these thin specimens are average values for a number of specimens and the error bars are the standard deviations of the results.

Specimens which contained solute H introduced by quenching were tested in air. The 25 micrometer specimens were tested in either a He or a H_2 gaseous atmosphere at a pressure of 100 kPa provided by slowly flowing the appropriate gas through the chamber. Due to the very slow strain rates and light loads required to deform the specimen, provisions had to be made to control and/or compensate for temperature changes in the room, atmospheric pressure changes and for drift in the electronics used to measure the load. The specimens were epoxied to prealigned grips and the load on the specimen was monitored and controlled to prevent loading during the setting of the epoxy. Grip alignment was maintained during the testing by low friction fixtures. A frictionless oil seal was used to separate the test chamber atmosphere from the air.

The fragile nature of the specimens generally precluded use of strain gage fixturing. Consequently, the stress was defined as the load divided by the initial specimen crosssection and the strain was defined as the elongation (determined from the crosshead displacement) divided by the initial length. Crosshead displacement could be monitored directly as well as determined by the speed of a synchronous motor control. Since the total strain used in these tests is very small, errors associated with the above definitions of stress and strain are negligible.

EXPERIMENTAL RESULTS

Gaseous Hydrogen Environment

The general result of slow strain rate tests in gaseous H_2 environments is that the flow stress in the microstrain and initial plastic strain region of the flow curve is reduced relative to the values obtained on testing in inert gaseous environments such as He. The detailed behavior appeared to depend on the strain rate and on the level of other solute elements with the largest effects observed in specimens containing significant C concentrations such as the 25-70/30 series. At strain rates of the order of 10^{-7} sec^{-1} , testing in H_2 resulted in a significantly decreased flow stress at small strains as shown in Fig. 1. The work hardening rate was however higher in the tests in H_2 gas leading to a convergence of the two flow curves. At higher strain rates ($7 \times 10^{-5} \text{ sec}^{-1}$) there was no significant difference between the behavior of specimens tested in the two atmospheres (Fig. 2). Tests at intermediate strain rates are consistent with the solution softening being

predominant at low strains and low strain rates. Since the hydrogen must enter the specimen by diffusion or dislocation transport from the gaseous atmosphere, the decrease in the softening effect at high strain rates may simply reflect the limitation imposed by the kinetics of this transport process. The decrease in the softening effect of H_2 at high strains is consistent with the higher work hardening rate observed for tests in H_2 atmospheres. Much smaller softening effects were observed for tests on specimens 25-95/5 which had significantly lower C interstitial concentration. At strain rates of 10^{-7}sec^{-1} the specimens tested in H_2 gas had lower flow stresses in the low strain region than those tested in He but the effect was less than for the 25-70/30 series and did not appear to be statistically significant. It was not possible to systematically vary the C concentration in these thin specimens to study the effect of the C solutes.

Tests on high purity Ni (HP-70/30) in which the major impurity was C (3200 appm) were used to establish the significance of this particular solute in the softening mechanism. Tension tests at strain rates as high as $2 \times 10^{-5} \text{sec}^{-1}$ showed H softening in these specimens when the C was retained in solid solution by rapid cooling from 1573K as shown in Fig. 3. In these specimens the significant softening relative to tests in air persisted to relatively high plastic strains. In contrast to this behavior, HP-70/30 specimens which were vacuum annealed at 1173K followed by furnace cooling (designated HP-70/30-V), had reduced levels of C in solid solution due to

precipitation of carbides and exhibited the behavior shown in Fig. 3. The lower C concentration in solid solution resulted in an increase in the flow stress relative to tests in air when the tests were carried out in H_2 gas.

In order to determine the source of this softening, the strain rate sensitivity or "activation volume", $v^* = kT (\partial \ln \sigma_f / \partial \dot{\epsilon})$, (where σ_f is the flow stress), was determined at various strains in the H_2 and He atmospheres. The values obtained for the two atmospheres were identical to within the experimental error of the determination and were independent of the strain for $\epsilon \lesssim 4 \times 10^{-3}$. The value of v^* did depend on the heattreatment however, presumably due to variation in the impurity level. For example, the 25-70/30 series (annealed at 1240 K for 24 hrs.) had $v^* = 1.3 \pm 0.2 \times 10^{-26} m^3$ while those annealed at slightly lower temperatures for longer times (1225 K for 78 hrs.) which leads to a somewhat higher C concentration gave $v^* = 0.5 \pm 0.2 \times 10^{-26} m^3$. Within each heattreatment group there was no effect of H_2 on the v^* values.

Stress dip tests were used to determine whether the H_2 softening results from the athermal or temperature dependent portion of the flow stress. In these tests the plastic deformation was interrupted, the specimen partially unloaded and the stress relaxation was observed for a short period of time to determine whether relaxation occurred in the forward direction (applied stress greater than the internal stress, σ_{int}) or in the reverse direction (applied stress less than the internal stress). The applied stress at which no stress relaxation occurred was

taken as the internal stress. The internal stress determined in this way for 25-70/30 specimens was $\sigma_{int}/\sigma_f = 0.85 \pm 0.04$ in He atmospheres and was independent of strain between 9×10^{-4} and 5×10^{-3} m/m. In H_2 atmospheres the values were $\sigma_{int}/\sigma_f = 0.88 \pm 0.05$ for $\epsilon > 2.4 \times 10^{-3}$ m/m. At lower strains the σ_{int}/σ_f values were significantly below this value.

Solute Hydrogen Effects

Results obtained with solute hydrogen introduced by quenching from elevated temperatures are generally consistent with those obtained on testing in the gaseous atmospheres. Solute H resulted in a decreased flow stress at low strains in Ni specimens which contained C interstitials and had little effect (or perhaps even a solution strengthening effect) in the absence of C interstitials. These effects were observed in several specimen types and can be illustrated by the data shown in Fig. 4. Specimens with heat treatment R-V contained about 32 appm of C and had the same stress-strain curve with and without 700 appm solute of H (Fig. 4, Curves A and B). The solute C concentration could be further reduced by a low temperature anneal at 775K for 24 hours (designated R-V-V) which tended to cluster and precipitate some of the solute C. This had little effect on the stress-strain curves in the absence of H (Fig. 4, Curve C) but the addition of 300 appm solute H resulted in significant solution strengthening (Fig. 4, Curve D). Increasing the solute C concentration to 180 appm (R-70/30) resulted in a significant solution strengthening (Fig. 4, Curve E) and very significant solution softening when 600 appm of H was added to the solid

solution.

These results were repeated using a variety of "pure" Ni specimens with the same general result. In very low C concentration specimens (or where the C was allowed to cluster or precipitate at low temperatures) solute H resulted in solution hardening. However, at moderate solute C concentrations the addition of solute H resulted in solution softening during slow strain rate tests and at small plastic strains.

DISCUSSION

While the present results seem to be at variance with the previously reported observations of solution strengthening in Ni this in fact is not the case since the previous studies were carried out at higher strain rates and at higher strains than the present experiments. In the case of tests in H_2 gas the amount of solute H is very small (of the order of 10 ppm) and little solution strengthening would be expected. In fact, however the higher work hardening rates observed on testing in H_2 atmospheres would be expected to lead to higher flow stresses at the larger strains used in previous studies. Additionally, it was observed that in the absence of interstitial C solutes, solute H does lead to solution strengthening even at relatively low strains and strain rates (Fig. 4). The observation of solution softening due to H has been made only in those specimens in which C was present as a solid solution strengthening element.

The observations of the present study are consistent with the extrinsic mechanisms of solid solution softening initially proposed for b.c.c. alloy systems (26,27). When H is present as

a solute in the absence of significant concentrations of other solid solution strengthening elements or when it is present at concentrations greatly in excess of these other elements, it acts as a solid solution strengthener and increases the flow stress. On the other hand, when it is added at low or about equal concentrations relative to the other solid solution strengthening elements such as C it acts to decrease the flow stress, ie as a solid solution softening element. This behavior is shown schematically in Fig. 5.

The observations are not consistent with the intrinsic softening mechanisms discussed (16-21) for the recent observations on iron alloys. These have as a requisite that the primary obstacle to dislocation mobility is the lattice interaction and focus attention on the effects of H on the lattice interaction, on the dislocation core structure, or on the energy to form kinks. None of these factors are expected to control the mobility of dislocations in the f.c.c. structure and yet the solution softening and strengthening effects of H are qualitatively similar to those observed in the b.c.c. systems.

Direct observation (12) of the solution softening has been made using in situ HVEM straining studies in an environmental cell. It was shown that the introduction of H_2 gas resulted in a large increase in the mobility of dislocations which were held under stress after straining in vacuum. The resulting dislocation structure, typical examples of which are shown in Fig. 6, highly exhibited tangled and curved dislocations with a much higher dislocation density than would be retained in the

structure by straining an equivalently small amount in vacuum. This high degree of dislocation entanglement and retention in the presence of H_2 atmospheres, while it has not been demonstrated for the case of solute H, is consistent with the increased work hardening rate in the early strain portion of the stress strain curve and with the rapid increase in the σ_{int}/σ_f observed in the early stage of deformation.

The initially low flow stress caused by the addition of H to a solid solution of C in Ni remains to be explained. In the case of the b.c.c. alloys it was suggested (29) that the second alloy element (H) forms small clusters with the first alloy addition (C) thereby decreasing the effectiveness of each element in interacting with dislocations. There is a large body of information which demonstrates that H does in fact interact with other solute elements to form small solute clusters in both b.c.c. and f.c.c. metals (30,31). It has also been recently shown (29) in the case of the Fe-N-H system that the formation of such a cluster decreases the Snoek relaxation associated with the N interstitials and that this is probably due to a decrease in the tetragonality of the strain field around the N solute. A direct demonstration of such an effect is not available for the f.c.c. systems.

Introduction of C interstitial solutes to Ni causes solution strengthening as a result of the elastic interaction between the solutes and the dislocations. While the C interstitial occupies a site of cubic point group symmetry (the octahedral or tetrahedral interstitial site) and therefore has a cubic

distortion field, C-C pairs can form in Ni (32,33) and these have distortion fields whose symmetry is less than cubic. These pairs may interact and pin both edge and screw dislocations thereby leading to significant solution strengthening. Hydrogen solutes can decrease this interaction in several ways. The thermodynamics of the C-C interaction may be affected by the formation of C-H pairs in a way which decreases the number of dislocation pinning points. Alternatively, the distortion field around the C-C pair may be affected by the interaction of the pair with H solutes in a manner which decreases the tetragonality of the pair and thereby its effectiveness as a dislocation barrier. Either a decrease in the number of solute pinning points or a decrease in their effective interaction with dislocations is consistent with the observations of enhanced dislocation mobility. Qualitative observations of the type reported here however are not sufficient to distinguish between these possibilities.

CONCLUSIONS

On the basis of the present experimental information the mechanism for the observed solid solution softening and solid solution strengthening can not be definitively established. We may however conclude that the effects are due to an extrinsic mechanism of solution softening or strengthening. That is the addition of H to solid solution affects the strength of the extrinsic barriers to dislocation motion rather than affecting the interactions with the lattice. A tentative suggestion that H decreases the effectiveness of other impurity barriers, such as C interstitials, is consistent with the experimental observations.

The main experimental observations may be summarized as follows:

- a) Solid solution softening is observed in nickel during slow strain rate tensile tests at low strains when H is added as a solute element or when the test is carried out in a gaseous atmosphere of H_2 . In this latter gas, the strain rate must be sufficiently low to allow equilibration of H throughout the lattice.
- b) The solution softening requires the presence of other solution strengthening elements in the lattice. The most effective element in this regard appears to be C.
- c) In pure nickel the addition of H appears to cause solid solution strengthening.
- d) Hydrogen does not have a significant effect on the "activation volume" which characterizes the deformation mechanism. It does appear to decrease the ratio of the internal stress to the total flow stress at low strains.
- e) The macroscopic stress-strain results are completely consistent with observations of dislocation behavior during deformation in the HVEM in an environmental cell containing a H_2 gaseous atmosphere.

ACKNOWLEDGEMENTS

This work was supported by the Office of Naval Research Contract USN 00014-75-C-1012. We would like to acknowledge the use of the facilities of the Center for Microanalysis of Materials at the Materials Research Laboratory of the University of Illinois.

Table I. SPECIMEN HEATTREATMENTS

Specimen Designation	Heattreatments
25-70/30	1225K anneal for 24 hours in 70% CO - 30% CO ₂ , rapid cool to 873K, slow cool to room temperature
25-95/5	1225K anneal for 24 hours in 95% N ₂ - 5% H ₂ , rapid cool to 873K, slow cool to room temperature
R-V	1573K anneal for 30 minutes in a vacuum of 10 ⁻³ Pa, oil quenched
R-V-V	Same heattreatment as R-V followed by vacuum anneal at 775K for 24 hours, furnace cooled to room temperature
R-70/30	1225K anneal for 20 hours in 70% CO - 30% CO ₂ , oil quenched
HP-70/30	1225K anneal for 24 hours in 70% CO - 30% CO ₂ , rapid cool to 873K, slow cool to room temperature
HP-V	1573K anneal for 30 minutes in a vacuum of 10 ⁻³ Pa, oil quenched
HP-70/30-V	Same heattreatment as HP-70/30 followed by vacuum anneal at 1173K for 1 hour, furnace cooled at 100K per hour

Table II. CHEMICAL ANALYSIS OF SPECIMENS

16.

Element*

Specimen Type

	25-70/30	25-95/5	R-V	R-70/30	HP-V	HP-70/30
--	----------	---------	-----	---------	------	----------

Spark Source Mass Spectrometer

Pb	—	10	—	—	—	—
Ba	≤1	2	—	—	—	—
Sn	—	—	—	—	—	—
As	7	5	—	—	—	—
Zn	2	10	—	—	—	—
Cu	20	8	—	—	—	—
Fe	30	30	10	10	1	1
Mn	7	2	—	—	—	—
Ti	—	2	≤1	≤1	—	—
Ca	30	5	30	30	—	—
K	8	20	—	—	—	—
Cl	7	20	1	—	—	—
S	8	50	—	—	—	—
P	—	—	—	—	—	—
Si	10	5	—	—	—	—
Al	2	7	1	1	—	—
Mg	10	10	≤70	≤70	—	—
Na	8	3	—	—	—	—
F	4	5	1	1	—	—
Ta	—	—	—	—	—	—
Sb	—	—	—	—	—	—
Se	—	—	—	—	—	—
Co	—	—	1	1	—	—
Cr	—	—	≤3	≤3	—	—
V	—	—	—	—	—	—
B	—	—	—	—	—	—

Vacuum Fusion

C	1200	230	32	184	140	3200
S	9	28	—	—	2	44
N		61				
O			3	40		

Notes: All concentrations are in appm.

* All other elements were "not detected" i.e. their concentrations were less than the sensitivity of the mass spectrometer (~0.1 appm).

A blank entry indicates the presence of the element was not detected.

A (-) entry indicates the element was present but at concentrations too low to be estimated.

FIGURE CAPTIONS

1. Average stress-strain curves for 25-70/30 specimens tested in H_2 (Δ) and He (o) atmospheres at strain rates of 10^{-7}sec^{-1} and at 300K. The curves are the average of at least seven tests and the error bars indicate the standard deviation of each set of results.
2. Average stress-strain curves for 25-70/30 specimens tested in H_2 (Δ) and He (o) atmospheres at strain rates of $7 \times 10^{-5} \text{sec}^{-1}$ and at 300K. The curves are the average of at least seven tests and the error bars indicate the standard deviation of each set of results.
3. Stress-strain curves for HP-70/30 specimens tested in air (—) and in H_2 (- - - -) atmospheres and for HP-70/30-V specimens tested in air (—) and in H_2 (— - - -) atmospheres.
4. Stress-strain curves for R specimens tested at a strain rate of $7 \times 10^{-8} \text{sec}^{-1}$ at 300K.
 Curve A: R-V specimen
 Curve B: R-V specimen containing 700 appm H
 Curve C: R-V-V specimen
 Curve D: R-V-V specimen containing 300 appm H
 Curve E: R-70/30 specimen
 Curve F: R-70/30 specimen containing 600 appm H
5. Schematic representation of solution strengthening due to C and H solutes in Ni (—). The broken curve (- - -) represents the solution softening which occurs when H is added to a binary solution of C in Ni.

6. HVEM microstructure results from in-situ environmental cell deformation studies (Ref. 12)
- (a) Structure after deformation in vacuum
 - (b) Same area as (a) after 10^4 Pa of H_2 gas was admitted to the environmental cell while the displacement applied to the deformation stage was held constant.
 - (c) Typical dislocation structure observed after deformation in vacuum and admission of 10^4 Pa of H_2 gas to the environmental cell. This area had a very low dislocation density as a result of deformation prior to admission of the H_2 gas.

REFERENCES

1. B. A. Wilcox and G. C. Smith, *Acta Met.*, 13, 331 (1965).
2. A. H. Windle and G. C. Smith, *Metal Science Journal*, 2, 187 (1968).
3. A. H. Windle and G. C. Smith, *Metal Science Journal*, 4, 136 (1970).
4. G. C. Smith, Hydrogen in Metals, p. 485, Ed. I. M. Bernstein and A. W. Thompson, (ASM, Metals Park) (1974).
5. R. M. Latanision and H. Oppenheimer, Jr., *Met. Trans.*, 5, 483 (1974).
6. R. M. Latanision and H. Oppenheimer, Jr., Hydrogen in Metals, p. 539, Ed. I. M. Bernstein and A. W. Thompson, (ASM, Metals Park) (1974).
7. M. H. Kamdar, Paper 3D 10, *Proc. of Second Int. Congress on Hydrogen in Metals*, (Pergamon Press, Oxford) (1977).
8. S. P. Lynch, *Scripta Met.*, 13, 1051 (1979).
9. T. Matsumoto and H. K. Birnbaum, Hydrogen in Metals, *Proc. of the 2nd J.I.M. International Symposium, Transactions of the Japan Institute of Metals Supplement*, 21, 493 (1980).
10. J. Eastman, T. Matsumoto, N. Narita, F. Heubaum and H. K. Birnbaum, Hydrogen Effects In Metals, p. 397, Ed. I. M. Bernstein and A. W. Thompson, (AIME, New York) (1980).
11. R. M. Latanision and R. W. Staehle, *Scripta Met.*, 2, 667 (1968).
12. T. Matsumoto, J. Eastman and H. K. Birnbaum, *Scripta Met.*, 15, 1033 (1981).
13. J. R. Hirst, M. L. H. Wise, V. Fort, J. P. G. Fan and I. R.

- Harris, Jnl. of Less Common Metals, 49, 193 (1976).
14. A. A. Sagues, M. G. Ulitchny and R. Gibala, Effect of Hydrogen on the Behavior of Materials, Ed. A. W. Thompson and I. M. Bernstein, p. 390 (AIME, New York) (1976).
 15. C. C. Chen and R. J. Arsenault, Acta Met., 23, 255 (1975).
 16. H. Matsui, H. Kimura and S. Moriya, Mat. Sci. and Eng., 40, 207 (1979).
 17. S. Moriya, H. Matsui and H. Kimura, Mat. Sci. and Eng., 40, 217 (1979).
 18. H. Matsui, H. Kimura and A. Kimura, Mat. Sci. and Eng., 40, 227 (1979).
 19. C. G. Park, K. S. Shin, J. Nagakawa and M. Meshii, Scripta Met., 14, 279 (1980).
 20. I. M. Bernstein, Scripta Met., 8, 343 (1974).
 21. C. G. Park, K. S. Shin, J. Nagakawa and M. Meshii, Scripta Met., 14, 279 (1980).
 22. S. Asano and R. Otsuka, Scripta Met., 10, 1015 (1976).
 23. S. Asano and R. Otsuka, Scripta Met., 12 (1978).
 24. Y. Tobe and W. R. Tyson, Scripta Met., 11, (1977).
 25. H. Kimura, H. Matsui, A. Kimura, T. Kimura and K. Oguri, Hydrogen Effects in Metals, p. 191, Ed. I. M. Bernstein and A. W. Thompson, (AIME, New York) (1980).
 26. A. Sato and M. Meshii, Acta Met., 21, 753 (1973).
 27. E. Pink and R. J. Arsenault, Prog. in Met. Sci., 24, 1 (1979).
 28. R. Gibala and T. Mitchell, Scripta Met., 7, 1143 (1973).
 29. J. Au and H. K. Birnbaum, Scripta Met., 15, 941 (1981).

30. C. A. Wert, Hydrogen in Metals II, p. 305, Ed. G. Alefeld and J. Volkl, (Springer-Verlag, Berlin) (1978).
31. J. P. Hirth, Met. Trans. A., 11A, 861 (1980).
32. C. Tsien, Scientia Sinica, 10, 930 (1961).
33. M. Wuttig and H. K. Birnbaum, Appl. Phys. Let., 7, 320 (1965).

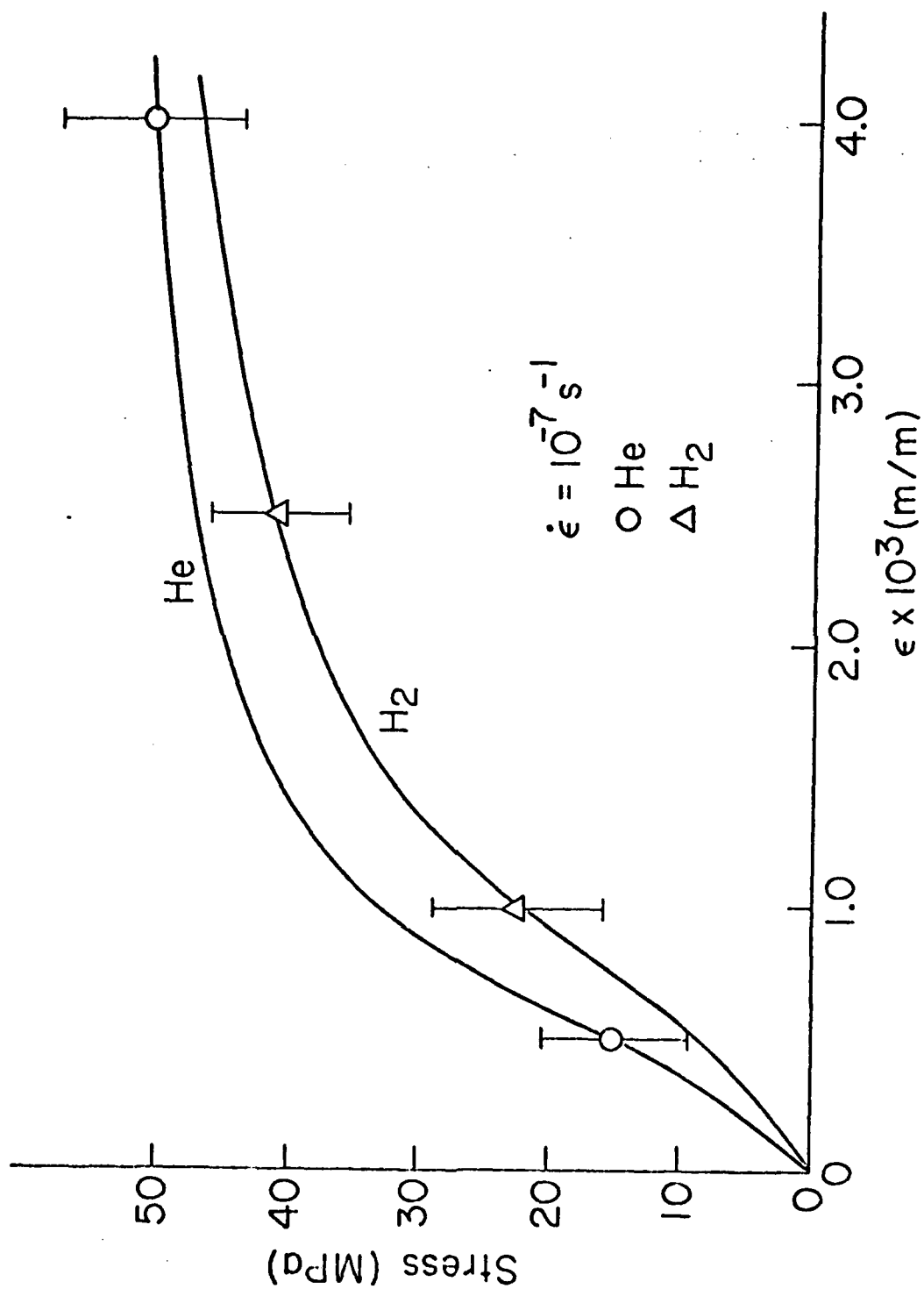


Fig. 1 Eastman et al

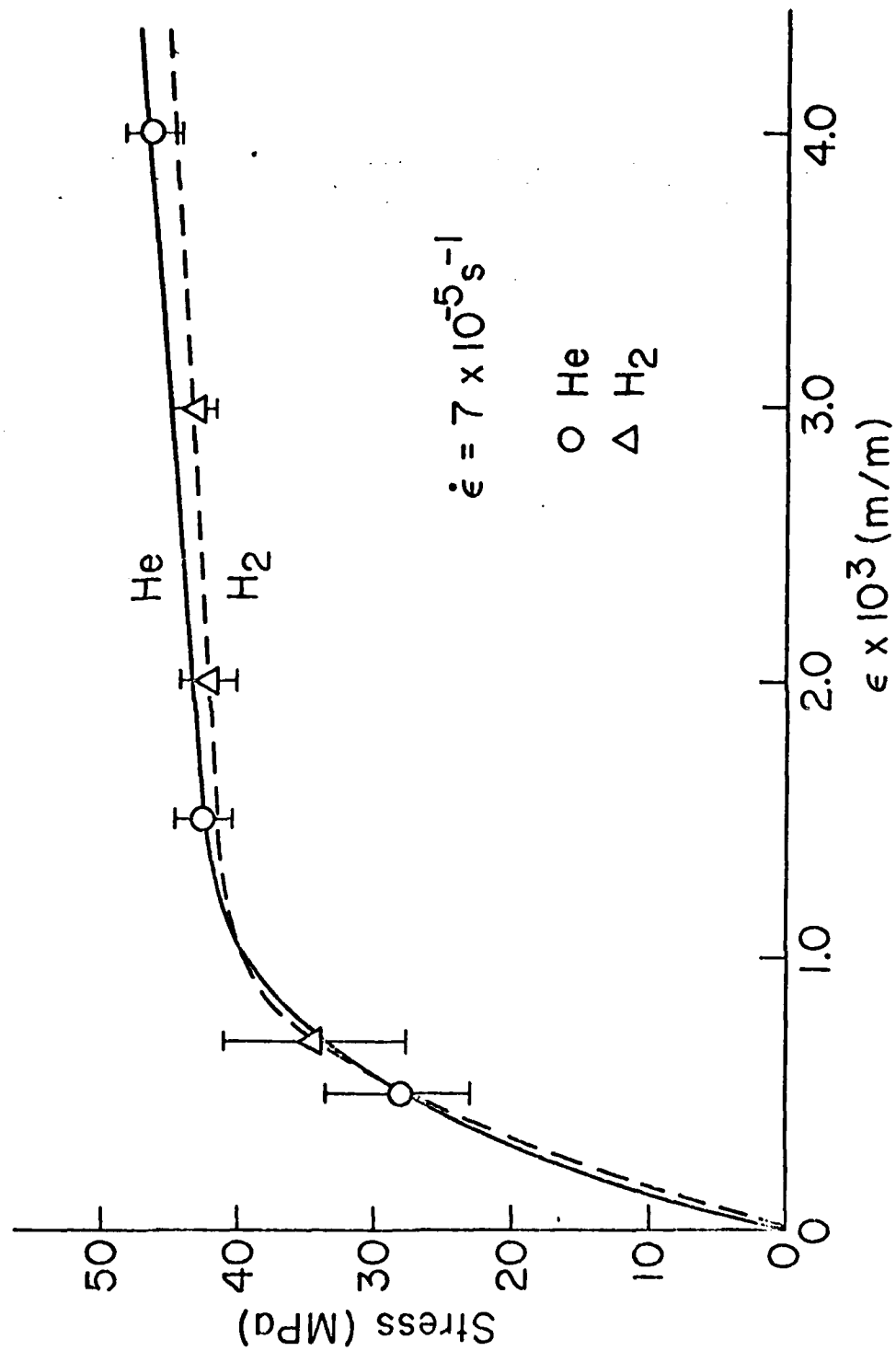


Fig. 2 Eastman et al

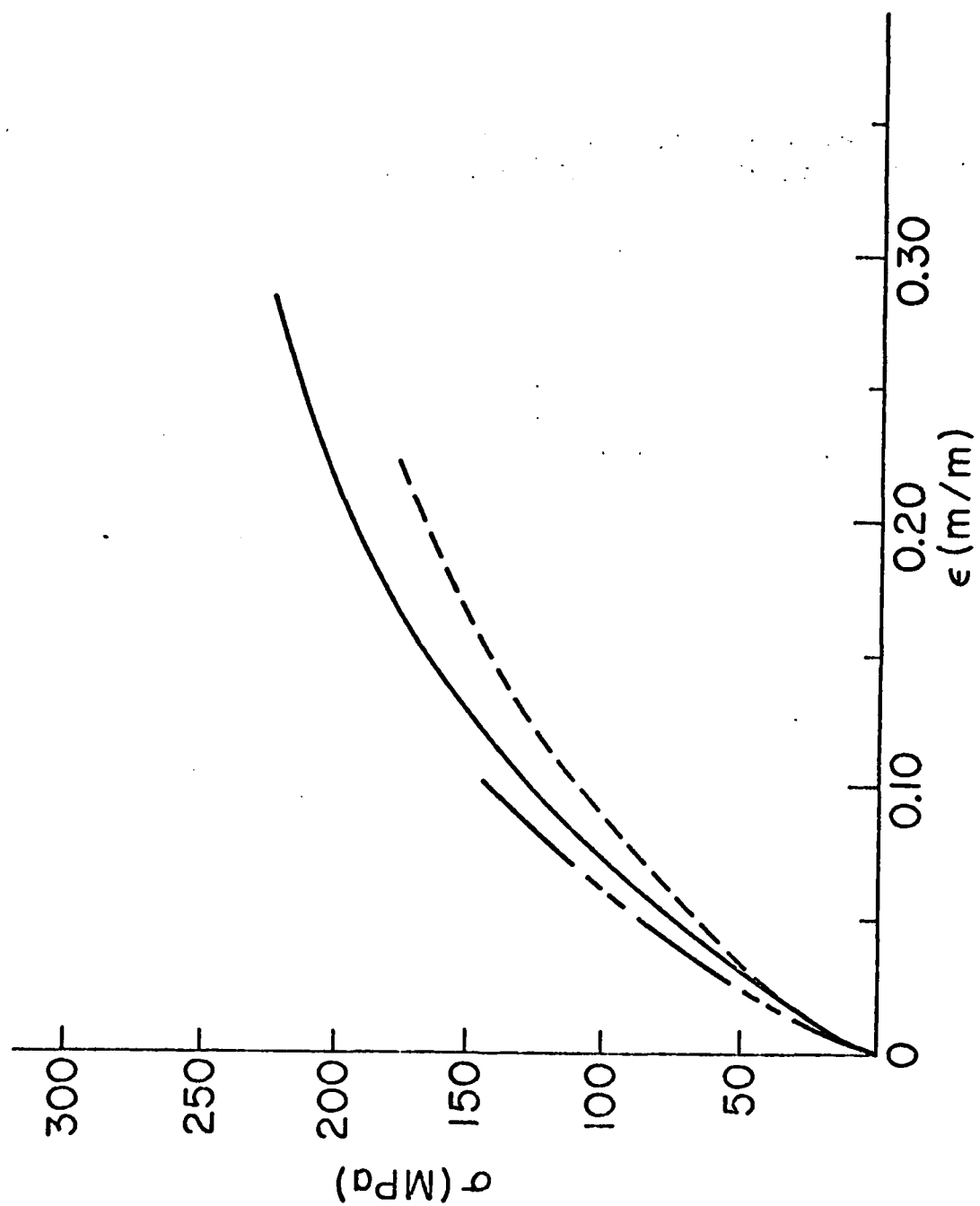


Fig. 3 Eastman et al

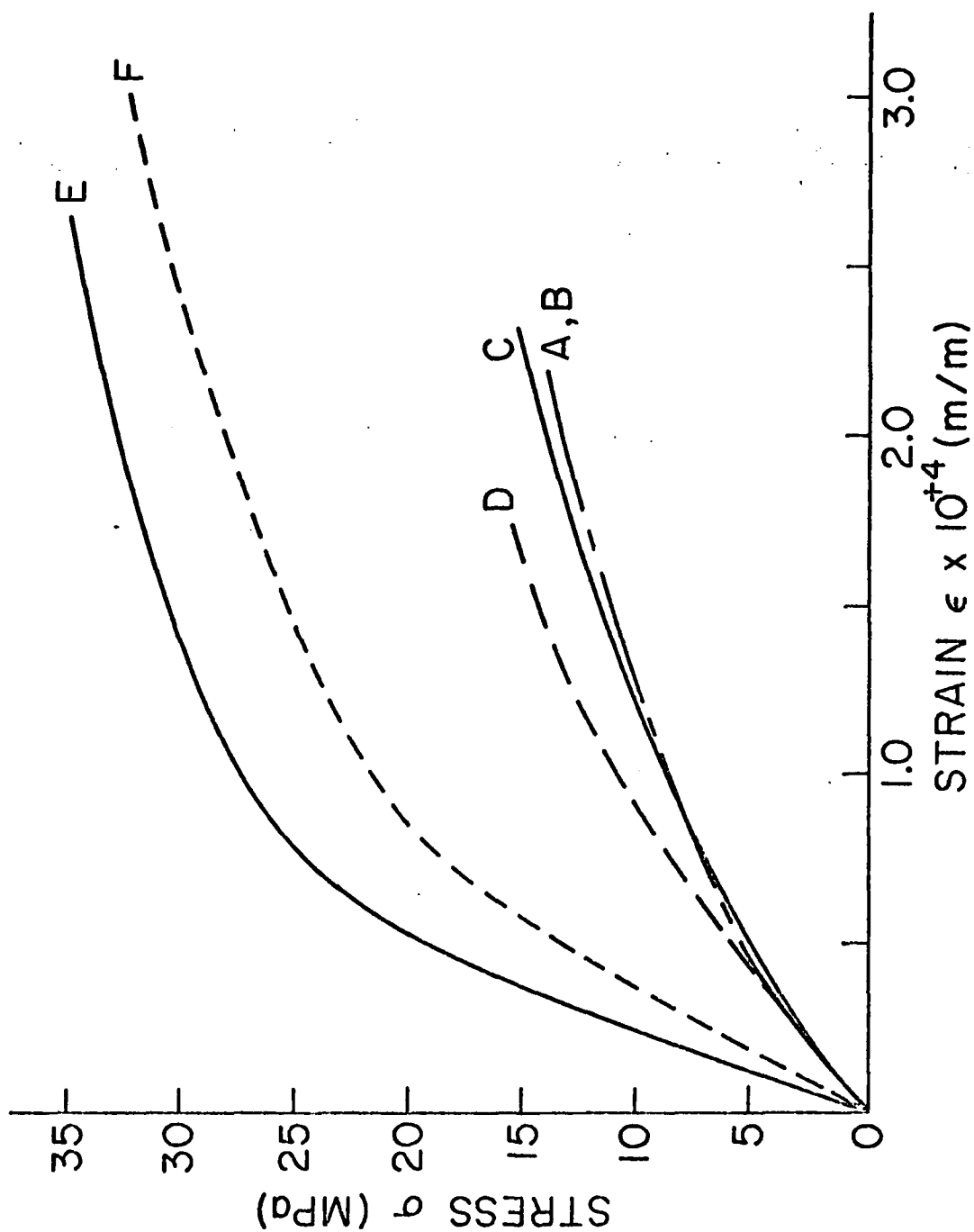


Fig. 4 Eastman et al

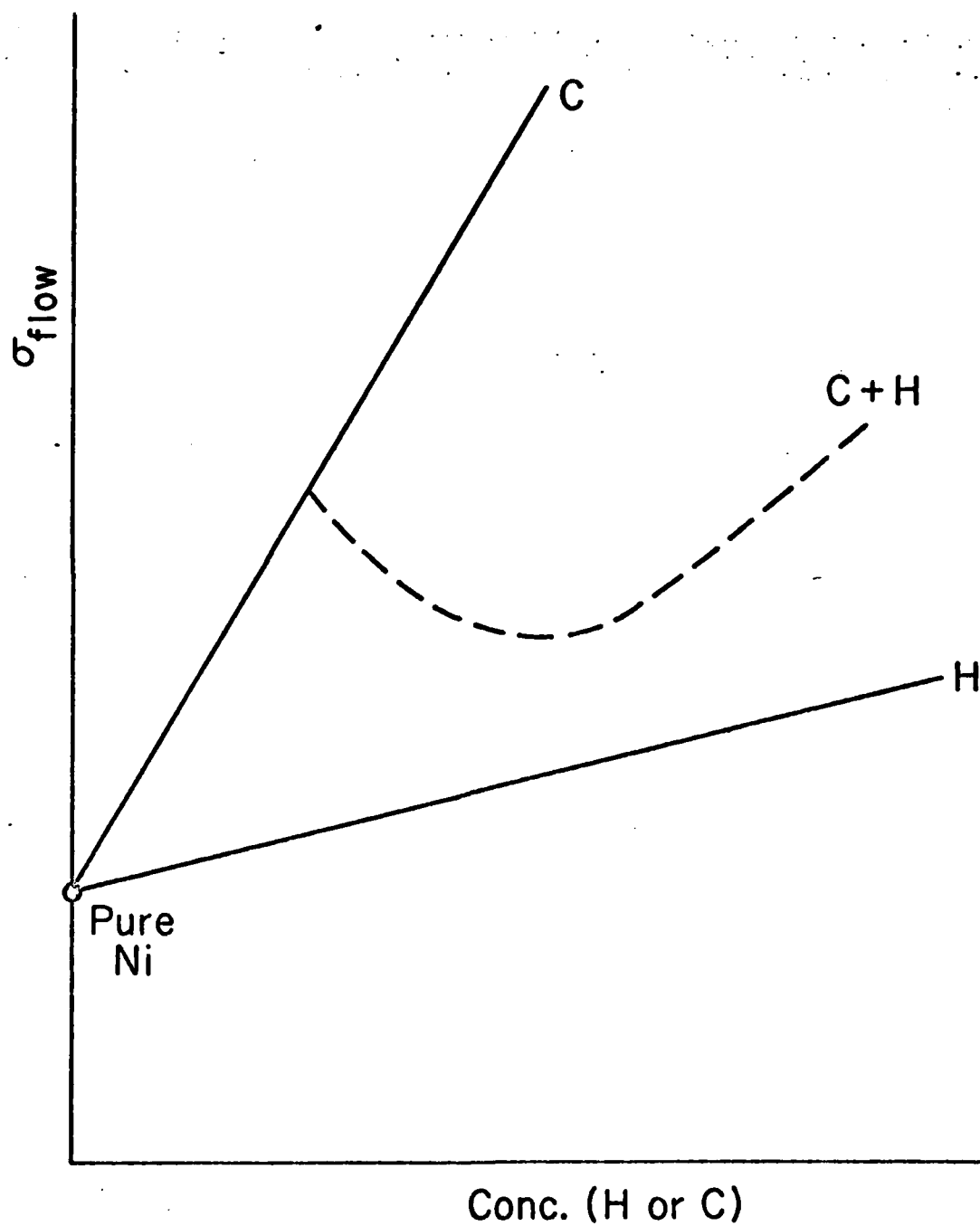
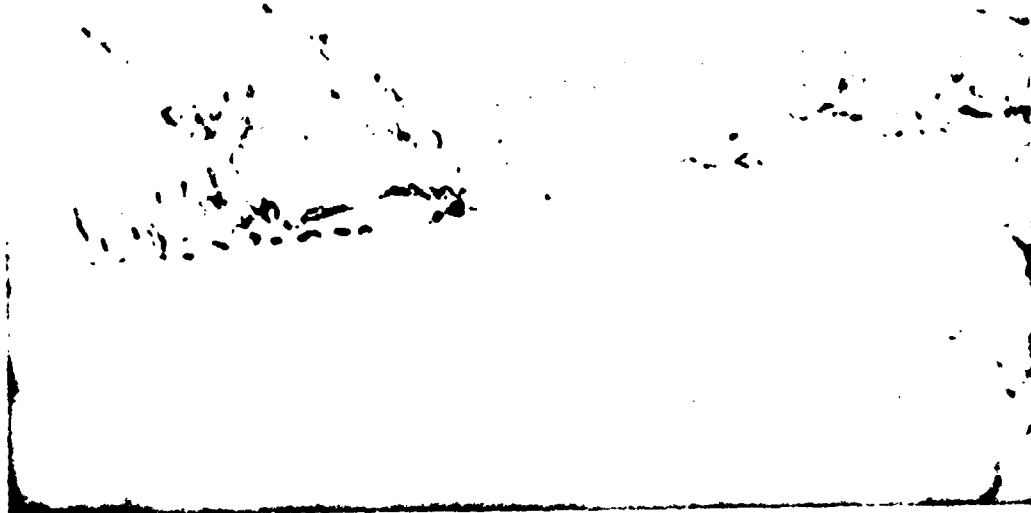
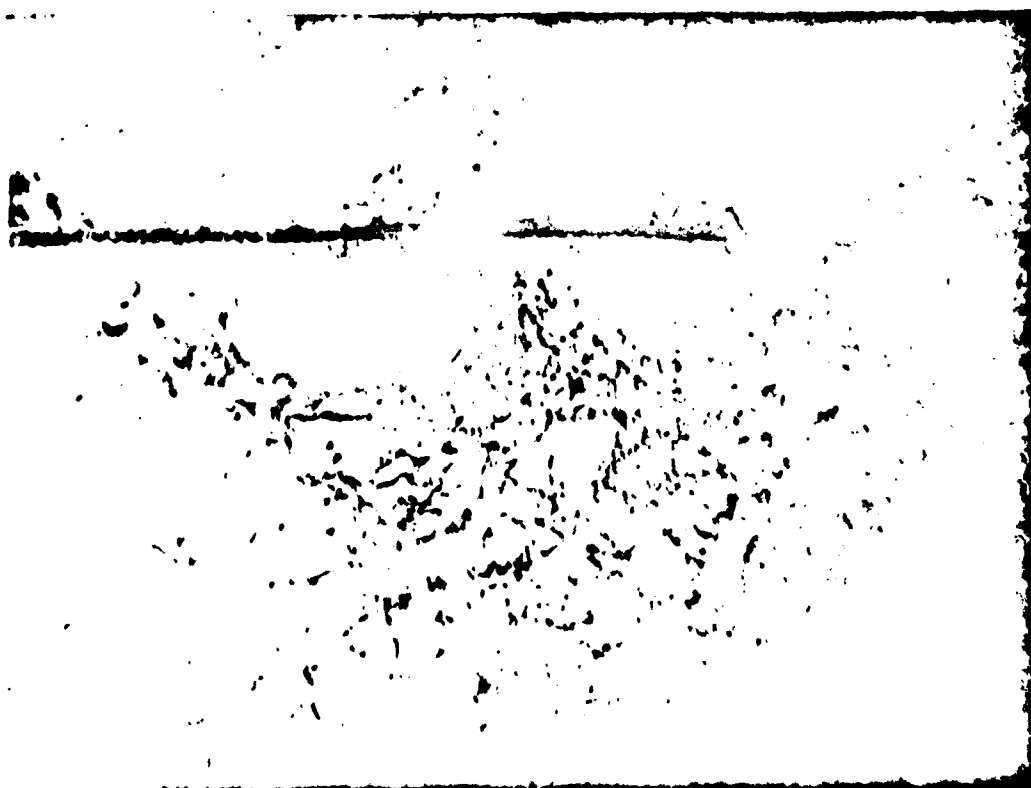


Fig. 5 Eastman et al



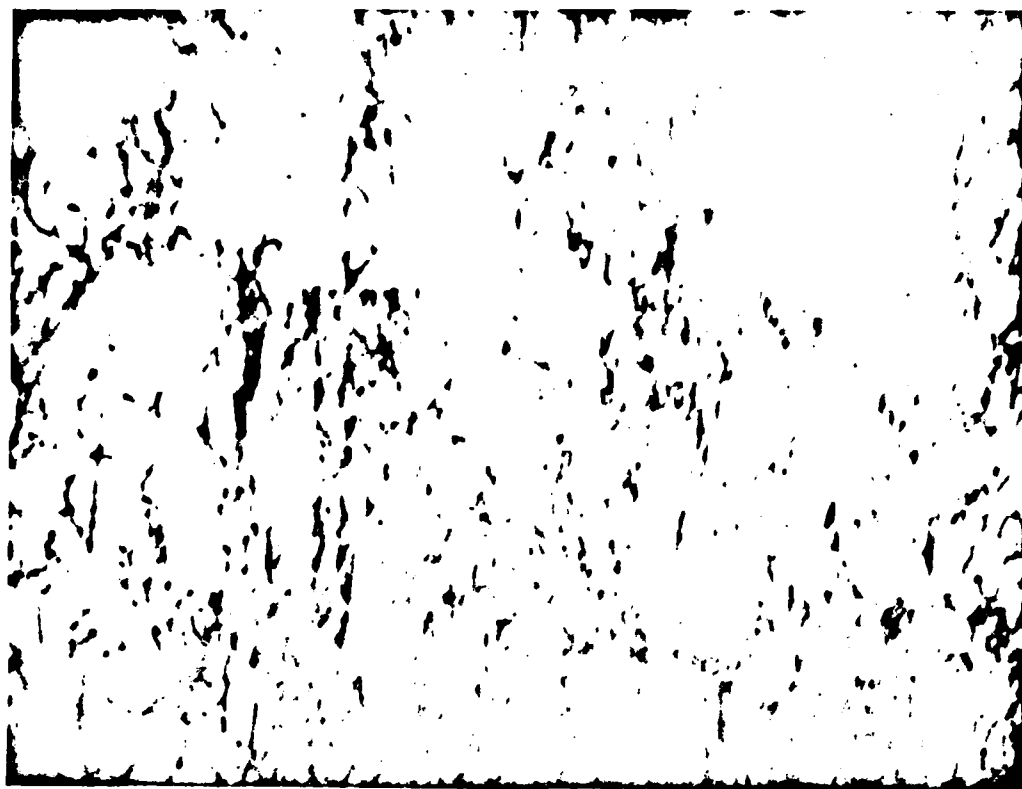
(A)

0.5 μ



(B)

Fig. 6 Eastmaneta



(C)

—
0.5 μ

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified

1. ORIGINATING ACTIVITY (Corporate author)		2a. REPORT SECURITY CLASSIFICATION	
University of Illinois, Dept. of Metallurgy and Mining Eng.		Unclassified	
		2b. GROUP	
		11-4108652	
3. REPORT TITLE			
The Effect of Hydrogen on the Solid Solution Softening and Hardening of Nickel			
4. DESCRIPTIVE NOTES (Type of report and, inclusive dates)			
Technical Report November 1981			
5. AUTHOR(S) (First name, middle initial, last name)			
J. Eastman, F. Heubaum, T. Matsumoto, and H.K. Birnbaum			
6. REPORT DATE		7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
Nov. 1981		28	33
8a. CONTRACT OR GRANT NO.		9a. ORIGINATOR'S REPORT NUMBER(S)	
USN 00014-75-C-1012			
b. PROJECT NO.		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10. DISTRIBUTION STATEMENT		DISTRIBUTION STATEMENT A	
		Approved for public release Distribution Unlimited	
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY	
		Office of Naval Research	
13. ABSTRACT			
The effects of hydrogen on the plastic deformation of nickel and nickel-carbon alloys were studied using plastic deformation techniques over a wide range of strain rates at about 300 K. The emphasis of the study was on the behavior at very low strains and low strain rates. Hydrogen was introduced as a solute element by quenching from a gaseous H₂ atmosphere or by testing in a gaseous H₂ atmosphere. The behavior of a number of different purities of nickel with hydrogen additions was examined. The most significant impurity element seemed to be C and this element was varied over a wide composition range by annealing in different atmospheres. Both solution softening and solution strengthening was observed depending on the amount of H in solution relative to the amount of C in solution. The nature of this solution softening is discussed.			

FORM 1473 (PAGE 1)

107-6811

Unclassified

Security Classification

A-21404

~~SECRET~~
Security Classification

Hydrogen, Nickel, Solid solution strengthening,
Solid solution softening, Deformation

END

DATE
FILMED

1-82

DTIC