# 14-MeV Neutron Radiation-Induced Microhardness Changes in Copper Alloys

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# 14-MeV NEUTRON RADIATION-INDUCED MICROHARDNESS CHANGES IN COPPER ALLOYS

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## Abstract

Transmission electron microscopy (TEM) disks of pure copper and copper alloyed with five atomic percent of either aluminum, manganese, or nickel have been irradiated at 25°C with 14-MeV neutrons. Vickers microhardness measurements were obtained as a function of fluence up to a maximum level of $2.2 \times 10^{21}$ n/m$^2$. Measurements were made at two different values of indenter loading (5 g and 10 g) in order to facilitate correlations with high-load microhardness data. A simple anti-vibration test stand was designed which allowed reproducible microhardness results to be obtained independent of background vibrations down to indenter loads of 2 grams. The radiation-induced microhardness change at both indenter loads scales linearly with the fourth root of neutron fluence following an incubation fluence. At a fluence of $2 \times 10^{21}$ n/m$^2$, the microhardness is increased by at least 50% over the unirradiated microhardness value for all four metals. The alloys, in particular Cu-5% Mn, exhibited a shorter incubation fluence and a larger radiation hardening than pure copper. Estimates of the defect cluster density obtained from 10 g microhardness data are in good agreement with the density observed by TEM methods. The irradiation-induced microhardness change is about 40% less at an indenter load of 5 g than at an indenter load of 10 g for a fluence of $2 \times 10^{21}$ n/m$^2$.

## Introduction

The materials properties of irradiated copper and copper alloys have been extensively studied over the past two decades (see e.g. Ref. 1). The irradiated properties of copper and its alloys are of current interest since high-strength copper alloys have recently been considered for use as high-magnetic field insert coils in tandem mirror and tokamak fusion reactors. For these reasons, copper is an ideal material on which to investigate the effect of irradiation on low-load microhardness. Simple binary copper alloys were also studied in order to determine the effect of solutes on low-load microhardness.

This paper describes the effect of a room temperature 14-MeV neutron irradiation on the Vickers microhardness of pure copper and three copper alloys. Microhardness data was obtained at indenter loadings of 5 g and 10 g in order to determine the effect of low indenter loads on microhardness results. This allows a determination to be made whether low-load microhardness results are representative of bulk material behavior. Since 14-MeV neutron irradiation results in relatively uniform, fine-scale damage compared to the sampling volume of a typical microhardness test, it may be argued that the irradiation-induced change in microhardness should be independent of indenter load (sampling volume). This investigation is designed to determine whether or not such an assumption is valid.

Resistivity and transmission electron microscope (TEM) data have also been obtained from these materials as a function of fluence in order to facilitate correlations.[2] These results may be compared to the microhardness predictions of defect survivability in order to determine the applicability of low-load microhardness data for modelling bulk properties.

## Experimental Procedure

Foils of pure ($99.99^+$ atom %) copper and copper alloyed with five atom percent of either aluminum, nickel or manganese obtained from Hanford Engineering Development Laboratory[3] were cold-rolled from 250 μm down to a thickness of 25 μm. TEM disks were cut from these foils, annealed in high-purity argon, and allowed to air cool. The pure copper samples were annealed at 400°C (0.5 $T_M$) for 15 minutes and the copper alloys were annealed at 750°C (0.75 $T_M$) for 30 minutes. The metals were irradiated at room temperature using 14-MeV neutrons from the Rotating Target Neutron Source-II (RTNS-II) at Lawrence Livermore National Laboratory. The irradiation consisted of four incremental fluences up to a maximum level of about $2 \times 10^{21}$ n/m$^2$. This was achieved by spacing the TEM disks away from the neutron source so that the disks experienced different flux levels during the irradiation. A previous investigation[3] determined that 14-MeV neutron PKA-damage events in copper were essentially independent of flux for fluences up to $10^{22}$ n/m$^2$ and flux levels of $\leq 2 \times 10^{16}$ n/m$^2$-s. Six TEM disks of each metal were irradiated to the various fluence levels. Pre-irradiation materials properties and irradiation conditions of the metals investigated are given in Table 1.

Vickers microhardness measurements were performed using a Buehler Micromet® microhardness tester. This instrument is designed for operation at indenter loads between 5 and 500 grams, with loads down to 1 gram available from the manufacturer. The indenter loading was restricted to be less than or equal to 10 grams in order for the diamond pyramid indentation depth to be less than one-tenth of the nominal TEM foil thickness of 25 μm. At this low indenter load, special care must be taken to ensure that background vibrations do not introduce errors into the measurement.[4] A simple anti-vibration test

Table 1 Irradiation Data for TEM Samples

| Alloy | Initial Resistivity ($\Omega$-m) | Grain Size ($\mu$m) | Maximum Fluence of TEM Disks (n/m$^2$) |
|---|---|---|---|
| Cu | $4.48 \times 10^{-11}$ | 13 | $1.9 \times 10^{21}$ |
| Cu-5% Al | $3.96 \times 10^{-8}$ | 23 | $2.1 \times 10^{21}$ |
| Cu-5% Mn | $1.08 \times 10^{-7}$ | 22 | $2.0 \times 10^{21}$ |
| Cu-5% Ni | $5.16 \times 10^{-8}$ | 12 | $2.2 \times 10^{21}$ |

stand (shown schematically in Fig. 1) was developed which effectively isolated the microhardness tester from background vibrations down to indenter loadings of 2 grams. The 1000 kg lead brick base provides the test stand with a large inertia so that all except very low frequency vibrations are damped out. The air pad serves as a dashpot to effectively suppress most of the remaining vibrations. The other cushion materials were chosen on the basis of their different stiffness values. The use of varied materials was found to be effective in eliminating external vibrations. Also, the value of a multiple-interface design, which serves to reflect/dampen vibration waves, was found to be important. The anti-vibration stand was found to be very effective in suppressing background vibrations down to indenter loads of 2 grams as determined by various testing methods (hardness vs. load curves and reproducibility of low-load hardness results in the presence of various external vibrations).

A standard sample preparation procedure was developed for all of the TEM disks prior to indentation. Each disk was given a light mechanical polish in a 0.3 μm alumina slurry on a rotating cloth wheel in order to remove the oxide layer and give a relatively smooth surface. The samples were then electropolished in a 33% $HNO_3$/67% $CH_3OH$ solution cooled to -20°C at an applied

## MICROHARDNESS ANTI-VIBRATION TEST STAND

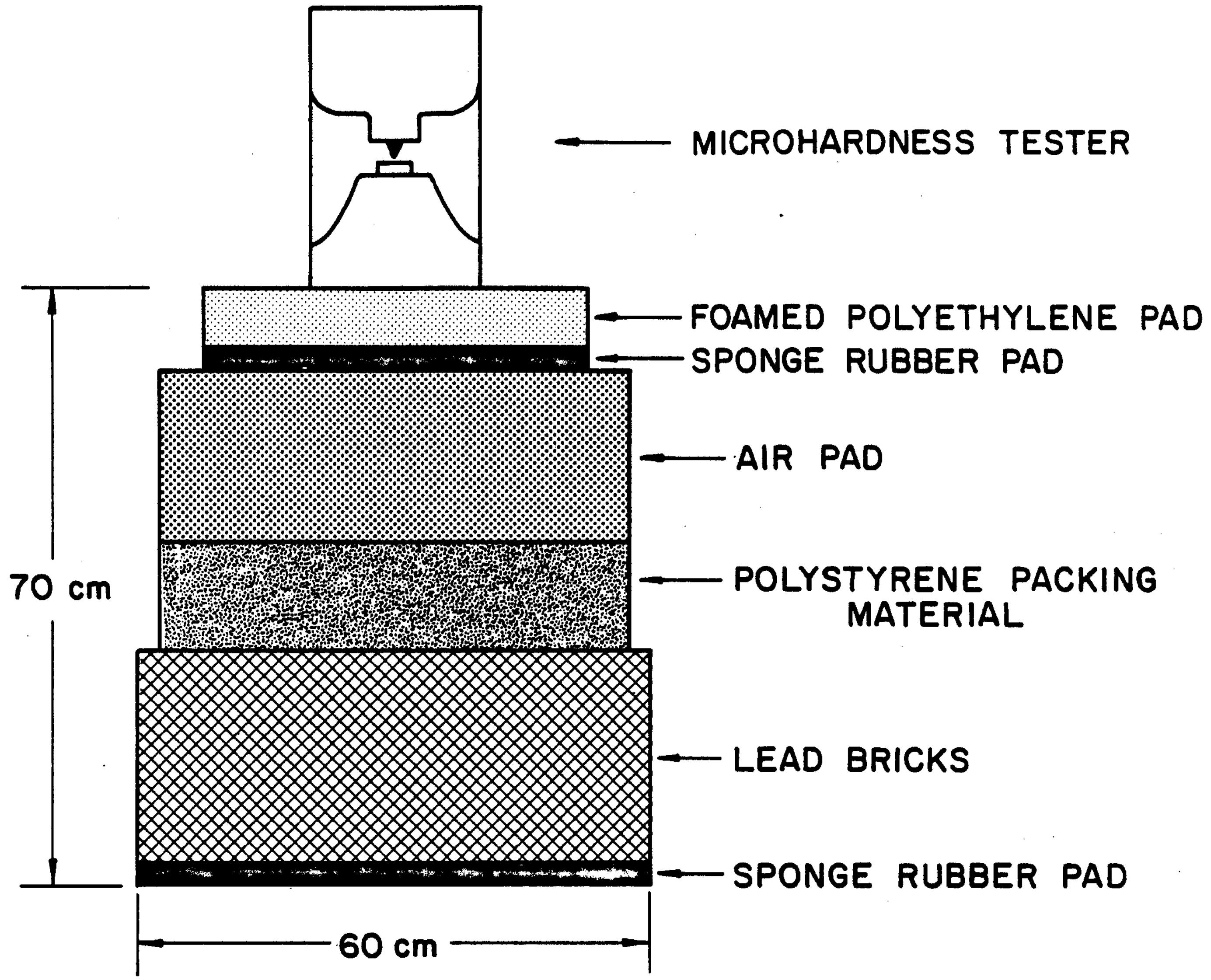



Figure 1

potential of 5 V for 5 seconds. The purpose of the electropolish was to remove the work-hardened layer introduced by mechanical polishing, and also to give an optically smooth surface. The electropolish treatment removed 1-2 μm, which is expected to exceed the work-hardened surface layer in copper for the type of mechanical polishing which was utilized.[5]

A minimum of 60 indentations were made for each value of indenter load on four different TEM disks for each metal at every fluence level. All measurements were made by same operator in order to minimize the effect of different observer's biases. The lengths of the diagonals of the indentation were measured at a magnification of 600X using a calibrated eyepiece accurate to ± 0.2 μm. Both diagonals of the indentation were measured twice and the results averaged in order to minimize measurement errors. The length of the chisel tip of the Vickers diamond[4] used in this study was found to be less than 0.2 μm by SEM methods. Indentations were made around the periphery of the TEM disk so that the disk could still be used for electron microscope observations.

Several TEM disks were re-examined at different time periods and on different days of the week in order to determine the reproducibility of the results. Agreement on the Vickers microhardness ($H_V$) for a single disk was typically found to be within about 1 $kg/mm^2$ ($\lesssim$ 2% deviation). A larger variation was noticed between different TEM disks of the same metal irradiated to identical fluences -- typical standard deviations about the mean for a set of 4 disks (60 indentations) were 2-3 $kg/mm^2$.

## Experimental Results

The Vickers microhardness numbers of the unirradiated samples at indenter loads of 5 g and 10 g are summarized in Table 2, along with the experimental

Table 2 Vickers Microhardness of Control Samples

| | Sample $H_v$ (kg/mm$^2$) | | | |
|---|---|---|---|---|
| Indenter Load (g) | Copper | Cu-5% Al | Cu-5% Mn | Cu-5% Ni |
| 5 | 71 ± 4 | 71 ± 3 | 70 ± 2 | 65 ± 2 |
| 10 | 57 ± 4 | 54 ± 1 | 53 ± 3 | 53 ± 3 |

standard deviation for each set of 4 disks which were examined. The 10 g microhardness values of the metals are all about 54 kg/mm$^2$. The result for copper is slightly higher than the alloy values. The 5 g microhardness numbers are all about 70 kg/mm$^2$ with the exception of a somewhat lower value for Cu-5% Ni. Therefore, it appears that addition of Al, Mn and Ni solutes does not have a significant effect on the low-load microhardness of copper, i.e. solute hardening is negligible.

Figure 2 shows the microhardness change at an indenter loading of 10 g as a function of fluence. Following a short incubation fluence, the data for all four metals scales linearly with the fourth root of 14-MeV neutron fluence. The duration of the incubation period is shorter for the alloys (in particular Cu-5% Mn) than for pure copper. As can be seen from Fig. 2, room temperature 14-MeV neutron irradiation to a fluence of 2 x $10^{21}$ n/m$^2$ induces significant hardening in these metals -- microhardness changes are on the order of 50% or more of the control value. The Cu + 5% Mn alloy exhibited significantly larger radiation hardening as compared to the other metals at the fluence levels investigated. All four metals in Fig. 2 have roughly equal slopes in their curve of microhardness vs. fluence following the incubation period.

CHANGE IN VICKERS MICROHARDNESS vs. FOURTH ROOT OF 14-MeV NEUTRON FLUENCE

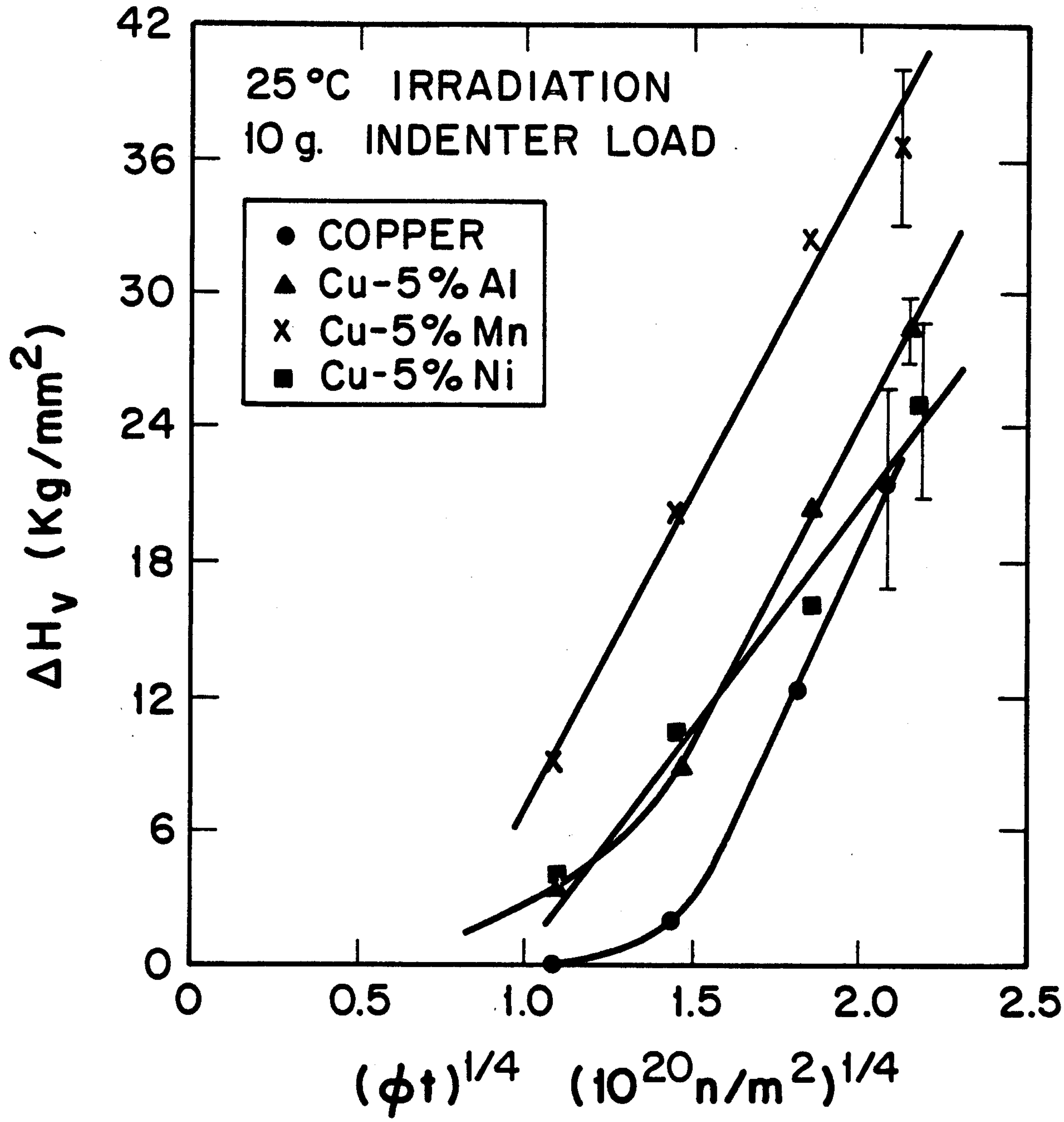



Figure 2

The pronounced load dependence of the microhardness induced by irradiation is illustrated for the four metals in Figs. 3 and 4. Figure 3 shows the Vickers microhardness as a function of the fourth root of neutron fluence at indenter loads of 5 g and 10 g, while Fig. 4 shows the <u>change</u> in Vickers microhardness vs. the fourth root of neutron fluence for the same indenter loads. The results at both loads are linear with $(\phi t)^{1/4}$ following an incubation fluence. The duration of the incubation fluence is slightly longer for the 5 g indenter load, and the slopes of the curves for the 5 g and 10 g loads are similar. The general differences between the alloys observed at a 10 g indenter loading are also present at a 5 g indenter loading -- Cu-5% Mn exhibits the largest radiation hardening and shortest incubation fluence of the metals investigated.

## Discussion

### a. 10 g Data

The Vickers microhardness value of the control pure copper samples at a load of 10 g is in good agreement with the value obtained by Brager et al.[3] at an indenter load of 50 g. However, the hardness values for the alloy control samples (in particular Cu-5% Al and Cu-5% Mn) are significantly lower than their reported values. The cause of this discrepancy is uncertain, as the materials were obtained from the same lot and were given identical heat treatments. The difference in the results may be caused by the different indenter loads. Another possible source of the variance may be due to the heavily cold-worked condition of our alloys prior to annealing caused by cold-rolling the foils down from 250 μm to 25 μm. The pure copper grain size in the present study (Table 1) is about 13 μm, which may be compared to a grain size of 55 μm found by Brager et al.[6]

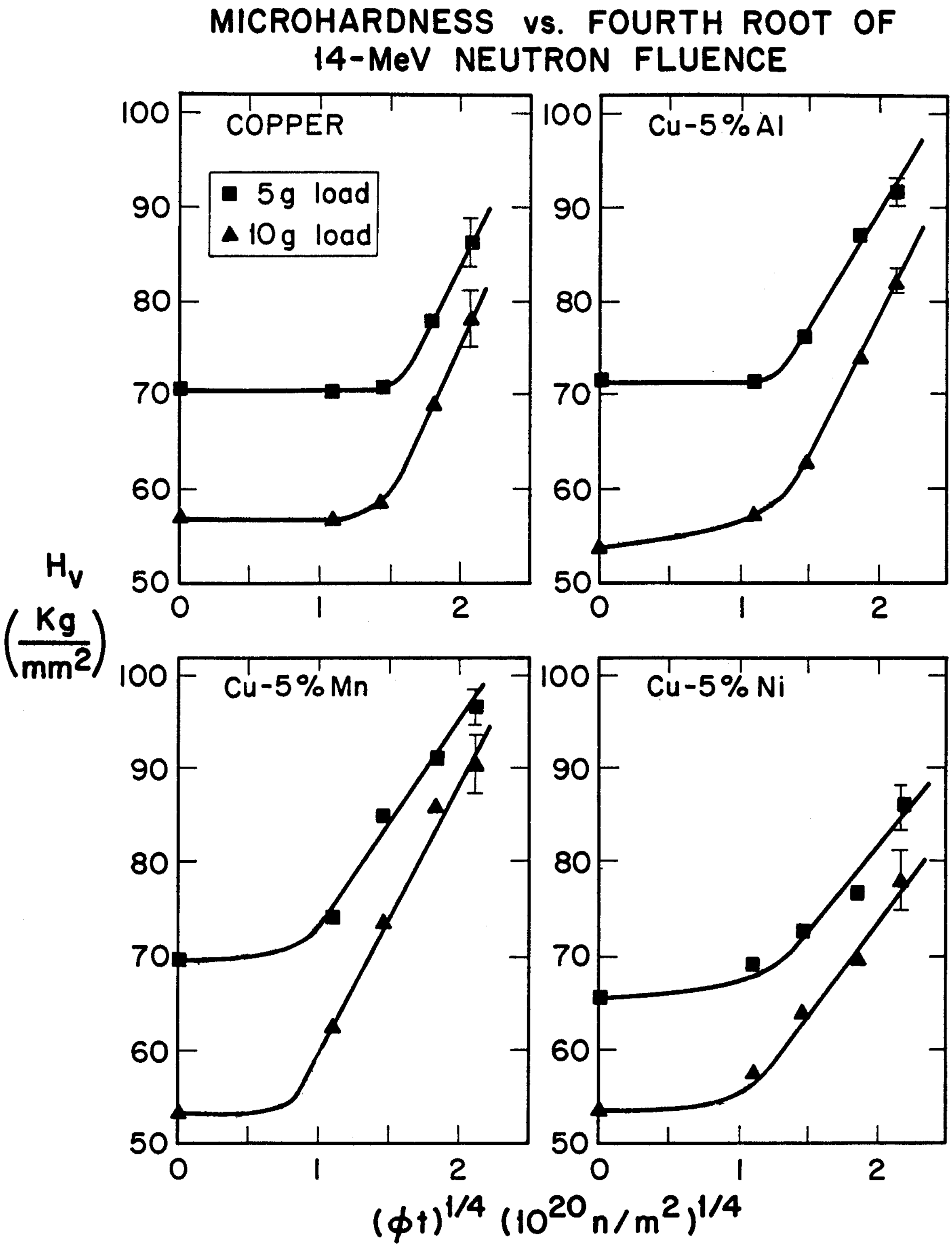
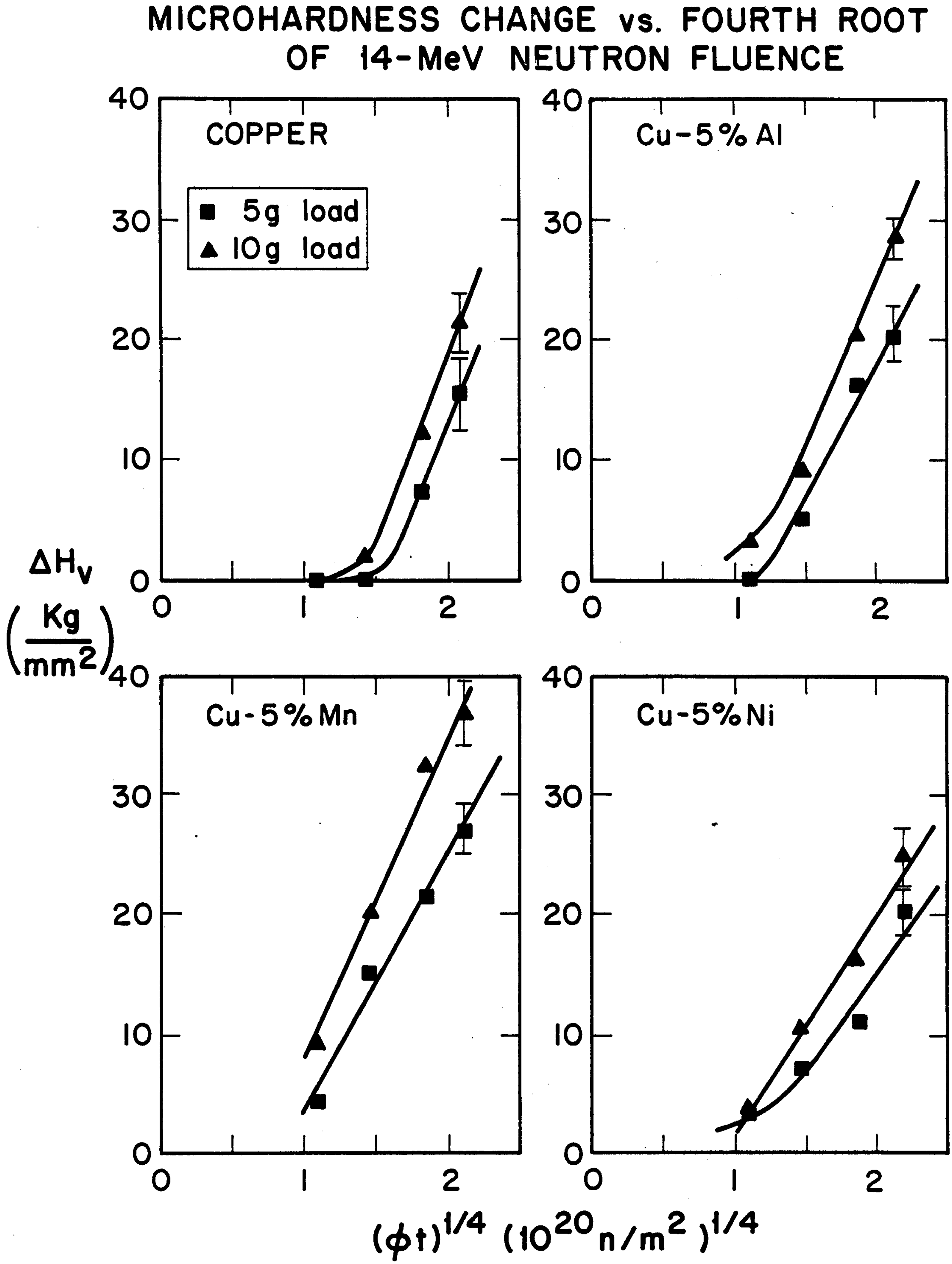

MICROHARDNESS vs. FOURTH ROOT OF
14-MeV NEUTRON FLUENCE
COPPER
5g load
10g load
Cu-5% Al
Cu-5% Mn
Cu-5% Ni
$H_V$
$\left(\frac{Kg}{mm^2}\right)$
$(\phi t)^{1/4}\ (10^{20} n/m^2)^{1/4}$

Figure 3



MICROHARDNESS CHANGE vs. FOURTH ROOT
OF 14-MeV NEUTRON FLUENCE
COPPER
5g load
10g load
Cu-5% Al
Cu-5% Mn
Cu-5% Ni
ΔHv
(Kg/mm2)
(φt)1/4 (1020 n/m2)1/4


Figure 4

The large effect of microstructure on low-load Vickers microhardness is illustrated in Fig. 5. In this figure, the low-load microhardness of as-received copper is compared to results from the same foil after it had been annealed in air at 600°C for 1 hour. The annealed copper microhardness increases at low-loads (indicating a "hard surface")[7] while the as-received copper microhardness remains constant, then decreases rapidly with decreasing load ("soft surface" layer).[7] This hard/soft surface layer effect has been commonly observed in the past,[4,8] but the exact mechanism which causes this effect is the subject of much controversy.[7]

Figure 2 indicates that the change in microhardness appears to be linear with the fourth root of neutron fluence. The roughly equal slopes for all four metals in the curve of microhardness change vs. the fourth root of neutron fluence may be taken as an indication that there are equivalent damage production rates in these materials following the initial transition period. The shorter incubation periods of the alloys, compared to pure copper, may be caused by the solute atoms acting as trapping sites. This leads to a shorter nucleation period for dislocation loops.

The theoretical hardening due to dislocation loops can be represented by[9]

$$\Delta\tau = \frac{\mu b}{\beta \bar{\ell}} = \frac{\mu b}{\beta} \sqrt{\sum_k n_k d_k} \tag{1}$$

where $\tau$ = shear stress and n is the loop density. Using the Von Mises criterion to relate yield strength to shear strength, $\Delta\sigma_Y = \sqrt{3}\,\Delta\tau$, and assuming a correlation may be obtained between microhardness data and yield strength[6] (i.e., $\Delta\sigma_Y = K\,\Delta H_v$), indicates that the microhardness increase is proportional to $\sqrt{n}$ . Since $\Delta H_v \sim (\phi t)^{1/4}$, this indicates that the loop density $n \sim \sqrt{\phi t}$ .

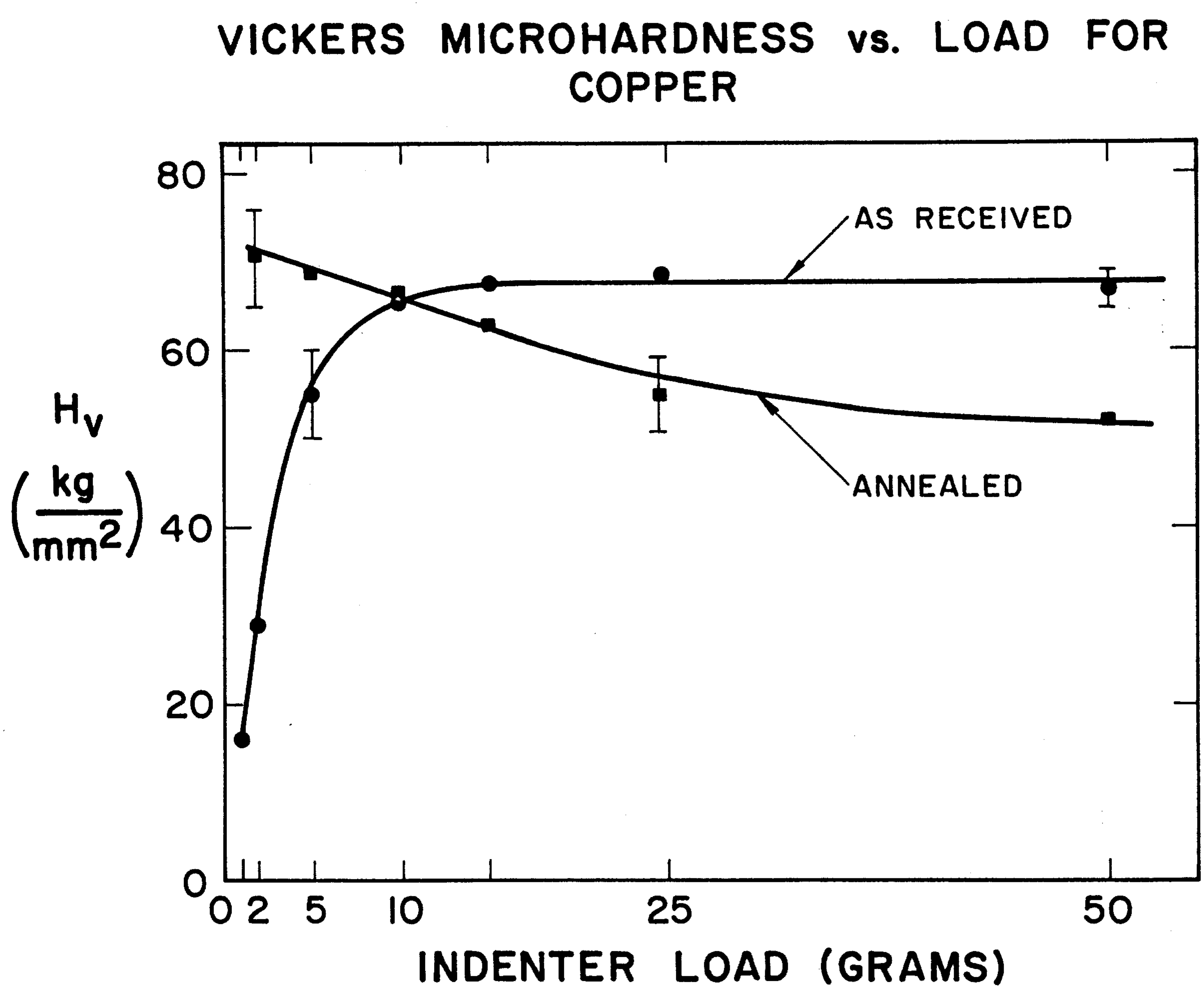

VICKERS MICROHARDNESS vs. LOAD FOR COPPER
AS RECEIVED
ANNEALED
80
60
40
20
0
$H_v$ $\left(\frac{kg}{mm^2}\right)$
0 2 5 10 25 50
INDENTER LOAD (GRAMS)

Figure 5

Table 3 Comparison of Calculated and Observed Defect Cluster Densities in Copper at a Fluence of 3 x $10^{21}$ n/$m^2$ Assuming Perfect Dislocation Loops. (From Ref. 2)

| Type of Analysis | Method | Copper Type | Cluster Density ($10^{22}/m^3$) |
|---|---|---|---|
| Calculation | Resistivity | DAFS | 11 |
| Calculation | Microhardness | DAFS | 12 |
| Calculation | Tensile [1] | LLNL/Cominco | 12 |
| Observed | TEM [3] | DAFS | 13 |

Analysis of the resistivity data obtained during the irradiation also predicts that the defect cluster density is proportional to the square root of 14-MeV neutron fluence.[2] In addition, good agreement between resistivity and 10 g microhardness estimates of the magnitude of the defect cluster density have been obtained.[2] The results of these calculations and the observed cluster density are compared in Table 3.

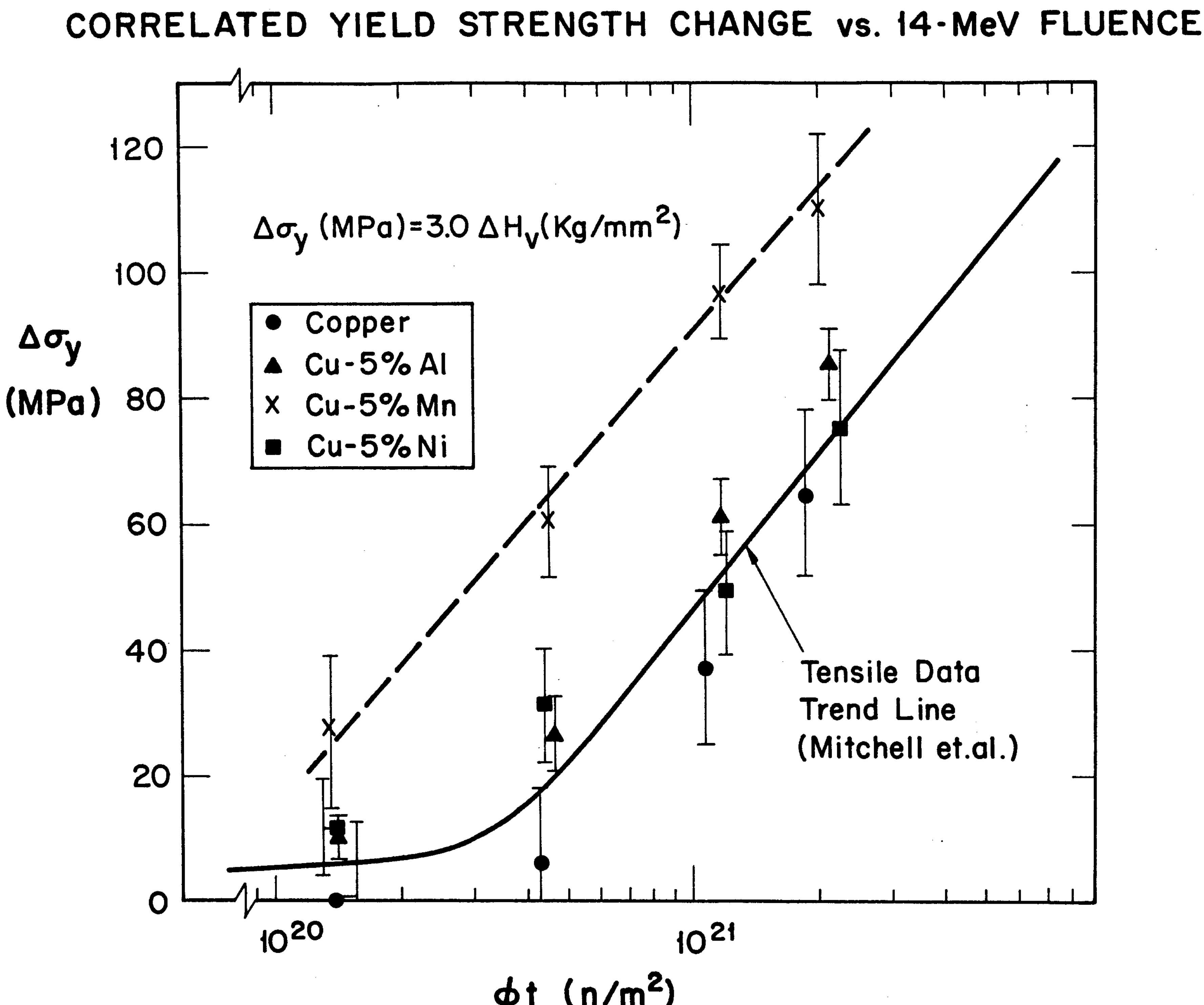
Previous experiments have found that the yield stress is proportional to the one-third power of the neutron fluence.[10] Unfortunately, there is insufficient high-fluence data in the present investigation to conclusively determine whether the present microhardness data scales as $(\phi t)^{1/4}$ or $(\phi t)^{1/3}$. The fact that the microhardness data was obtained at low-loads may have an effect on the observed fluence dependence. However, the good agreement be-tween the 5 g and 10 g results concerning the fluence dependence indicates that this is not the case. The agreement of the microhardness and resistivity data concerning the fluence dependence of the cluster density also lends credence to $\Delta H_v \sim (\phi t)^{1/4}$ .

Tensile tests performed on samples irradiated with neutrons and ions show a similar dependence on fluence as the microhardness results reported here --

an incubation period which lasts until a fluence of about $2 \times 10^{20}$ particles/$cm^2$ is followed by a monotonically increasing sample hardness. This behavior has been observed in tensile tests of a room temperature 14-MeV neutron irradiation of copper,[11] and for a 20°C irradiation of nickel by 16-MeV protons and 14-MeV neutrons.[12] A similar effect was also observed during microhardness measurements of molybdenum irradiated at 300°C with 10-MeV $^4He^+$ ions.[13]

Vickers microhardness data can be correlated to tensile data results found in the literature, $\Delta\sigma_Y(\mathrm{MPa}) = K\ \Delta H_v(\mathrm{kg/mm^2})$ . A recent microhardness correlation[6] found the result K = 3.27 to be valid for 14-MeV neutron irradiated copper tested at an indenter loading of 50 g. As seen in Fig. 6, we have obtained a reasonable correlation between the pure copper tensile data of Mitchell et al.[11] and the 10 g microhardness data for three metals (Cu, Cu-5% Al, Cu-5% Ni) with K = 3.0. The Cu-5% Mn microhardness data does not correlate well with the pure copper tensile data using this value of K. The change in $H_v$ at a load of 5 g during neutron irradiation is smaller than the observed change in $H_v$ at a 10 g load for all 4 metals (Fig. 4). Therefore, the bulk microhardness change during irradiation should be greater than or equal to the 10 g hardness change, $\Delta H_v)_{bulk} \gtrsim \Delta H_v)_{10\ g}$ . It then follows that the result K = 3.0 should be taken as a maximum value for a correlation of bulk microhardness with tensile data. The small discrepancy (10%) in the value of the correlation constant K between the present investigation and previous work[6] may be due to a difference in initial microstructure, as discussed earlier.

In summary, it appears that the two main effects of low-loads on the microhardness results of irradiated metals are: (1) a longer incubation fluence, which leads to (2) a smaller amount of radiation-induced microhardness change as compared to high-load results.



CORRELATED YIELD STRENGTH CHANGE vs. 14-MeV FLUENCE
$\Delta\sigma_y$ (MPa) = 3.0 $\Delta H_v$ (Kg/mm$^2$)
Copper
Cu-5% Al
Cu-5% Mn
Cu-5% Ni
$\Delta\sigma_y$ (MPa)
120
100
80
60
40
20
0
$10^{20}$
$10^{21}$
$\phi t$ (n/m$^2$)
Tensile Data Trend Line (Mitchell et.al.)

Figure 6

b. Implications of the Load Dependence of $H_v$

As is evident in Figs. 3 and 4, the choice of indenter loading at low loads can have a significant effect on the interpretation of microhardness results. At low-loads, it becomes increasingly difficult to correlate observed behavior to bulk properties -- the microhardness values become very sensitive to the sample's microstructural condition. In this study the 5 g microhardness number was greater than the 10 g value at all fluences. Conversely, the change in microhardness at a 5 g load was less than the 10 g result for all fluences. The dependence of low-load microhardness on the microstructure of the irradiated samples is illustrated in Fig. 7. In this figure, it is seen that the difference between the microhardness numbers obtained at indenter loads of 5 g and 10 g decreases with increasing neutron fluence. As the neutron fluence increases, there is a higher density of small dislocation loops present which can affect the low-load $H_v$. Extrapolation of the observed results to higher fluence indicates that the values of $H_v$ obtained at indenter loads of 5 g and 10 g may become equivalent for $\phi t \sim 1 \times 10^{22}$ n/m$^2$. For still higher fluences, it is possible that the value of $H_v$ obtained at a load of 5 g could become smaller than the 10 g microhardness value. Therefore, it appears that low-load results may not be representative of bulk behavior since the bulk hardness (measured at high indenter loads) is known to be independent of indenter load.

Samuels and Mulhearn experimentally determined that the strain falls below 1% for a Vickers diamond indenter at a distance from the point of indentation of about 7 times the indenter depth. The indenter depths for the control samples in the present study were about 2.5 μm for an indenter loading of 10 g and about 1.6 μm for an indenter loading of 5 g. The corresponding

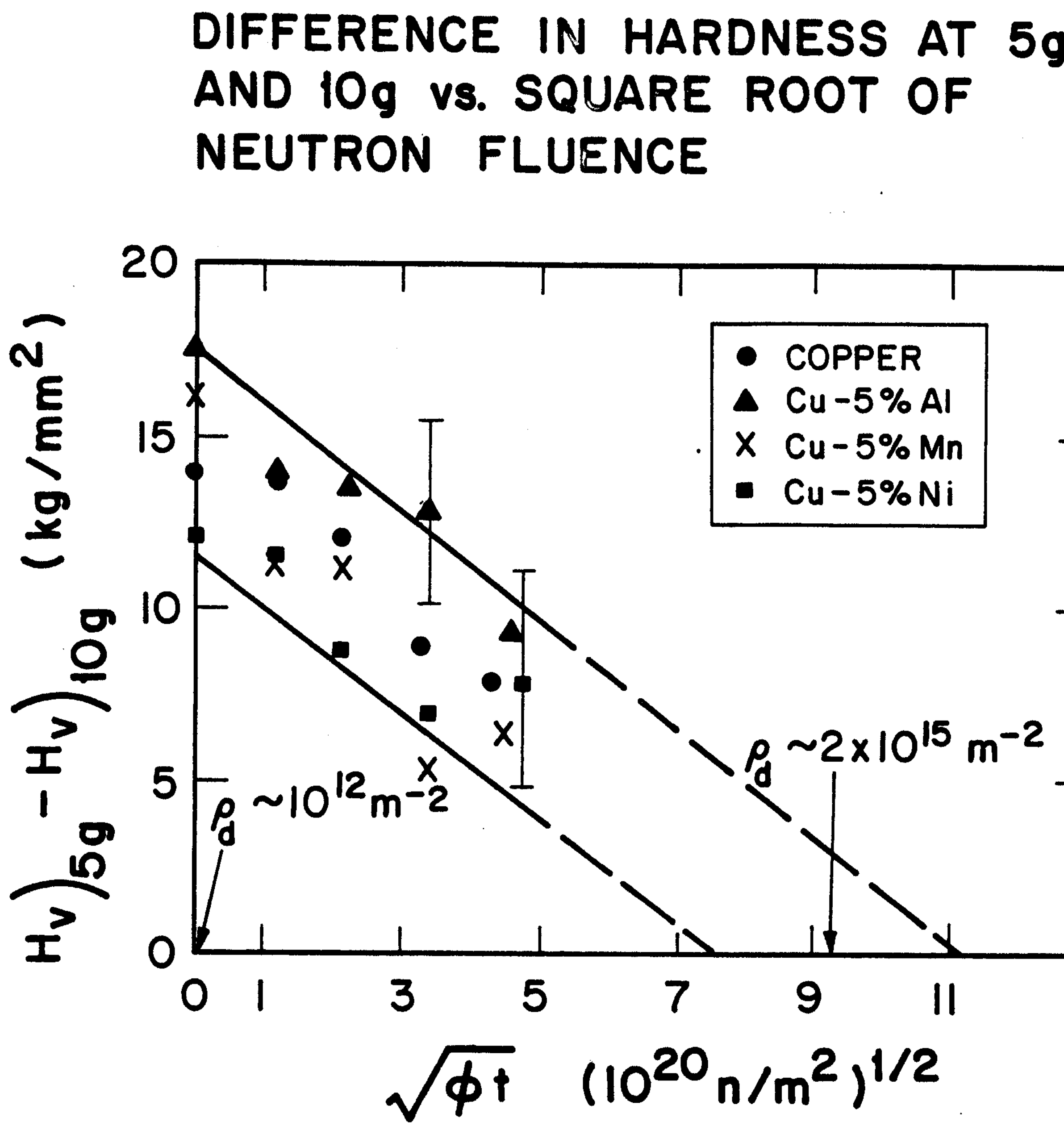

DIFFERENCE IN HARDNESS AT 5g
AND 10g vs. SQUARE ROOT OF
NEUTRON FLUENCE
COPPER
Cu-5% Al
Cu-5% Mn
Cu-5% Ni
$\rho_d \sim 10^{12} m^{-2}$
$\rho_d \sim 2 \times 10^{15} m^{-2}$
$H_v)_{5g} - H_v)_{10g}$ (kg/mm$^2$)
$\sqrt{\phi t}$ $(10^{20} n/m^2)^{1/2}$
0
5
10
15
20
0
1
3
5
7
9
11

Figure 7

"influence volume" ($\gtrsim$ 1% strain) of the indenter in this study therefore extends into the foil to a distance of about 18 μm for a load of 10 g and about 11 μm for a load of 5 g. Hence, the 5 g load is more indicative of the near-surface hardness of the foil and the 10 g load is relatively more representative of the bulk hardness. The approach of the 5 g and 10 g microhardness numbers to a common value with increasing neutron fluence may be taken as an indication that the defect clusters created during irradiation tend to decrease the value of the near-surface hardness. There is no known theory which adequately explains the near-surface microhardness phenomenon. It appears reasonable to assume that the observed microhardness should be given by

$$\left.H_v\right)_{observed} = \left.H_v\right)_{bulk} + \left.\Delta H_v\right)_{surface} . \tag{2}$$

The bulk term increases monotonically with irradiation and at large fluences dominates the observed microhardness value. The near surface term, $\left.\Delta H_v\right)_{surface}$, should depend strongly on the sample's microstructure (dislocation density) and also on the depth (x) from the surface. The surface term should become important only for indentation depths which are near the foil surface. It may be postulated that this term should be of the form

$$\left.\Delta H_v\right)_{surface} \sim \frac{1}{x^m} * f\ (\text{microstructure}) . \tag{3}$$

At large loads (large impression depths) the surface term becomes negligible and the microhardness approaches a constant value. At small loads, the surface term should become dominant and the measured hardness in single crystals may increase up to values comparable to the metal's theoretical shear strength. Gane[15] measured the hardness of gold under a load of about 0.1 mg and found a significant surface hardening ($\tau \sim \mu/100$, where $\mu$ = shear modulus). This value is a factor of twenty larger than the measured bulk hardness and corresponds to a shear strengh[15] of $\tau \sim \mu/20$ .

The microstructure factor (f) in the surface term may be positive or negative depending on the microstructure and condition of the foil surface. A negative value of f would indicate a "soft surface", whereas a positive value would indicate a "hard surface" layer. The exact cause of the hard vs. soft surface layer is unknown and is the subject of much controversy. Both work-hardened and work-softened surface layers have been experimentally observed.[7,16] It appears that in general, annealed materials exhibit readily work-hardened surface layers, while heavily cold-worked materials have soft surface layers. One known exception to this behavior was reported by Gane and Cox.[17] They found that the microhardness of gold increased at low-loads for both annealed and cold-worked samples.

Application of Eqs. 2 and 3 to microhardness vs. load curves of various materials in annealed and cold-worked states gave values of the exponent m ranging from 1/2 to 7. The larger values of m were found for the cold-worked materials (with a negative microstructure factor, f). This indicates a very short-range influence of the surface term for cold-worked metals. Upit and Varchenya[8] have reported that the total observed hardness near the surface is well approximated by $H)_{observed} = c_1 X^n$ . A cursory analysis of their results indicates that, by subtracting away the bulk hardness value, the surface hardness is well represented by Eq. 3. The dependence on depth of their reported results is then given by $m \sim 1\text{-}2$.

One possible explanation of the hard vs. soft surface phenomenon would be to consider the effect of surfaces and cold-work level on dislocation sources and dislocation multiplication. Near-surface sources began operating at lower stresses than bulk dislocation sources in a material with an initially low dislocation density.[7] If the dislocations that are produced do not readily glide out to the surface, then the near surface region will become preferentially work-hardened (Kramer's "debris layer" model)[7]. On the other hand, cold-worked materials already have an ample amount of Frank-Read dislocation sources. The high dislocation density (with resulting back stresses) may tend to minimize the preferential activation of near-surface dislocation sources compared to interior sources. Near-surface dislocations would tend to move to the free surface due to repulsive interactions with interior dislocations, where they are annihilated. This results in a lower dislocation density in the near-surface region upon deformation for cold-worked metals. Figure 7 may be seen as an indication that the surface becomes softer relative to the bulk with increasing dislocation density (in this case, dislocation loops). The 5 g (surface) hardness increases less rapidly than the 10 g (bulk) hardness with increasing neutron fluence. The abscissa is plotted as the square root of neutron fluence, which is proportional to the dislocation loop density (see earlier discussion). Extrapolating to higher fluences, it is seen that the 5 g and 10 g hardness values become equivalent for a fluence of $\sim 1 \times 10^{22}$ $n/m^2$. This fluence corresponds to a total dislocation line density of about $2 \times 10^{15}/m^2$. This density was calculated from TEM data[3] using an average loop diameter of 2.5 nm and assuming $n \sim \sqrt{\phi t}$ . It is seen that the calculated dislocation density extrapolated to where there is no surface hardening is typical of values for a cold-worked metal.

From the above discussion it appears that it is impossible to correlate low-load microhardness results to bulk behavior unless measurements are obtained at several different values of indenter load. With several properly chosen load values, it may be possible to ascertain the magnitude of the near-surface microhardness term and thereby determine the bulk contribution to the observed microhardness.

## Conclusions

The fluence incubation period and the magnitude of the radiation-induced microhardness change at low indenter loads are in modest disagreement with bulk microhardness results. The incubation period of the radiation-induced microhardness change at 5 g loads is longer than it is at 10 g loads. This leads to a smaller irradiation-induced change in $H_v$ at low loads compared to the change in the $H_v$ at higher loads. Low-load microhardness results are strongly influenced by the material's microstructure.

The general fluence dependence of low-load microhardness $\left(\Delta H_v \sim (\phi t)^{1/4}\right)$ appears to be in fair agreement with bulk results. This conclusion is based on the observation that the 5 g and 10 g load results show a similar fluence dependence following the incubation period.

Unirradiated copper alloys behave similar to pure copper with regard to low-load microhardness (i.e., no significant solute hardening). Conversely, alloying has a significant effect on the low-load microhardness of irradiated copper samples. The alloys have a shorter incubation fluence than pure copper. This is probably due to solute atoms affecting the nucleation of irradiation-produced dislocation loops. The Cu-5% Mn alloy exhibited a significantly shorter incubation fluence, and hence shows a larger radiation hardening at a given fluence compared to the other metals.

A reasonable correlation has been obtained between the 10 g microhardness data and tensile results. We have found $\Delta\sigma_Y(\mathrm{MPa}) = K\ \Delta H_v(\mathrm{kg/mm^2})$ gives a good correlation with K = 3.0. The slight discrepancy with another correlation found in the literature[6] (K = 3.27) is believed to be primarily due to different initial microstructures.

The general phenomenon of an increase in microhardness at low loads appears to be related to an intrinsic materials property which depends on the sample's microstructure. It is believed that the commonly observed increase in hardness near the surface is not an experimental artifact, but rather is an indication that annealed metals are more susceptible to work-hardening in the near-surface region as compared to the bulk.

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