Correlation of Nanoindentation and Conventional Mechanical Property Measurements

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ABSTRACT

A series of model ferritic alloys and two commercial steels were used to develop a correlation between tensile yield strength and nano-indentation hardness measurements. The NanoIndenter-II® was used with loads as low as 0.05 g_f (0.490 mN) and the results were compared with conventional Vickers microhardness measurements using 200 and 500 g_f (1.96 and 4.90 N) loads. Two methods were used to obtain the nanohardness data: (1) constant displacement depth and (2) constant load. When the nanohardness data were corrected to account for the difference between projected and actual indenter contact area, good correlation between the Vickers and nanohardness measurements was obtained for hardness values between 0.7 and 3 GPa. The correlation based on constant nanoindentation load was slightly better than that based on constant nanoindentation displacement. Tensile property measurements were made on these same alloys, and the expected linear relationship between Vickers hardness and yield strength was found, leading to a correlation between measured changes in nanohardness and yield strength changes.

INTRODUCTION

Ion irradiation provides samples well suited to investigation of microstructural evolution by transmission electron microscopy (TEM). However, special techniques are required to obtain mechanical property data from such specimens because the thickness of the irradiated area is only a few micro-meters. The high-precision NanoIndenter-II® [1] was used in this work to measure the change in hardness caused by radiation damage as a function of distance from the irradiated surface. Since the corresponding radiation-induced microstructure can be characterized by TEM, the relationship between microstructural and mechanical property changes can be investigated. This study was undertaken to determine the effects of minor solutes on radiation-induced property changes in model ferritic alloys, and the results of the microstructural investigations have already been published [2,3]. This paper focuses on the work done to develop a correlation between the nanoindentation data and conventional mechanical property measurements.

The model alloys used in this study are described in Table 1. They have been used by the University of California, Santa Barbara (UCSB) in neutron irradiation experiments [4] and by AEA Technologies (AEAT) in thermal aging studies [5]. The AEAT heat treatment was similar to that listed in Table 1, 16 hours at 770˚C, but their material was water quenched [5,6]. The as-received microstructure of the alloys was characterized by TEM, and an extensive description of the observations was published in Ref. [7].

COMPARISON OF VICKERS HARDNESS AND YIELD STRENGTH

Experiments were conducted using the model ferritic alloys and two commercial alloys to provide a basis for correlating the low load hardness values determined by the NanoIndenter-II® with macroscopic bulk property measurements. The yield strengths (0.2% offset) of the nine model alloys had been measured at UCSB and were in the range of 150 to 220 MPa. Two methods

Table I. Model alloy designations and compositions

Alloy Number	N (appm)	Composition (wt-%)			
		Cu	Mn	C	Ti
VM348	5	---	---	---	---
VM349	80	---	---	---	---
VM350	120	---	---	---	---
VM390	20	0.51	0.06	<0.005	0.002
VM397	20	0.91	<0.01	<0.01	<0.01
VM399	120	0.51	0.01	<0.01	<0.01
VM387	10	0.51	0.05	0.17	0.003
VM360	10	0.89	1.03	<0.003	---
VM373	100	<0.01	0.01	<0.003	0.3
Heat Treatment at UCSB: Solution treated at 775^{o}C for 17 hours, quenched in salt bath to 450^{o}C and held for 3 minutes, air cooled.					

were used to extend the range of yield strengths in the present study. First, the high-copper alloy VM397 was thermally aged to induce additional hardening by the formation of copper precipitates. Aging at 550˚C for 1, 2, 5, 10, and 15 hours lead to yield strengths in the range of 250 to 300 MPa. Second, two commercial alloys were included. These were a fine-grained A533B reactor pressure vessel steel with a yield strength of 465 MPa and a ferritic/martensitic alloy HT-9 (12Cr - 1MoVW) with a yield strength of 750 MPa.

Vickers hardness measurements using loads of 200 or 500 g_f (1.96 or 4.90 N) were used for comparison with the yield strength measurements. Prior to hardness testing, the specimens were mechanically polished down to 0.05 μm and then electrochemically polished with a perchloric acid solution so that the surface region was flat and damage free. The hardness values were determined by averaging six indents from a commercial Vickers hardness tester. Vickers hardness data from the VM397 aging study are shown in Figure 1, where the error bars are the standard deviation of the measurements at each aging time. The peak hardness change was observed after the 10 hour anneal. This is similar to the results reported by AEAT on the same alloy [5], which employed 20 kg_f (196 N) Vickers hardness measurements following aging of thick samples at 500˚C. They found a time to peak hardness of about 15 hours, with a maximum hardness change of 637 MPa. As mentioned above, the UCSB heat treatment included a short temper at 450˚C followed by air cooling, whereas the AEAT treatment involved quenching directly from the solution treatment. The AEAT heat treatment would retain more of the copper in solution. This difference could be responsible for the somewhat lower peak Vickers hardness change and shorter time to peak hardening observed here.

A complete comparison of the Vickers hardness and yield strength measurements obtained in this study is shown in Figure 2, where a linear relationship between the two property measurements is demonstrated over the complete range of the data. A linear least-squares fit to the yield strength versus Vickers hardness data yielded a slope of 0.2836 MPa/MPa. If the Vickers hardness data is expressed in traditional units, this value corresponds to 2.78 MPa/(kg_f/mm^2). This compares favorably with other recently published data [8], but is slightly below the traditional value of 3.0 [9].

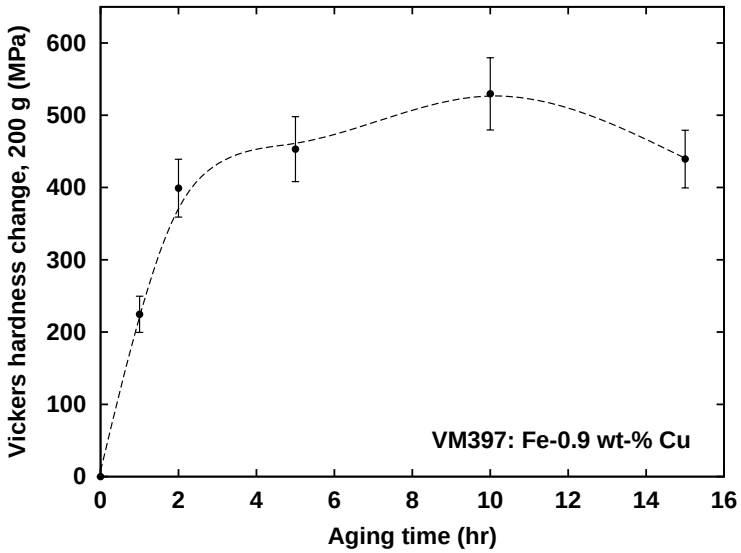

Figure 1. Hardness change observed in alloy VM397 under thermal aging at 550˚C.

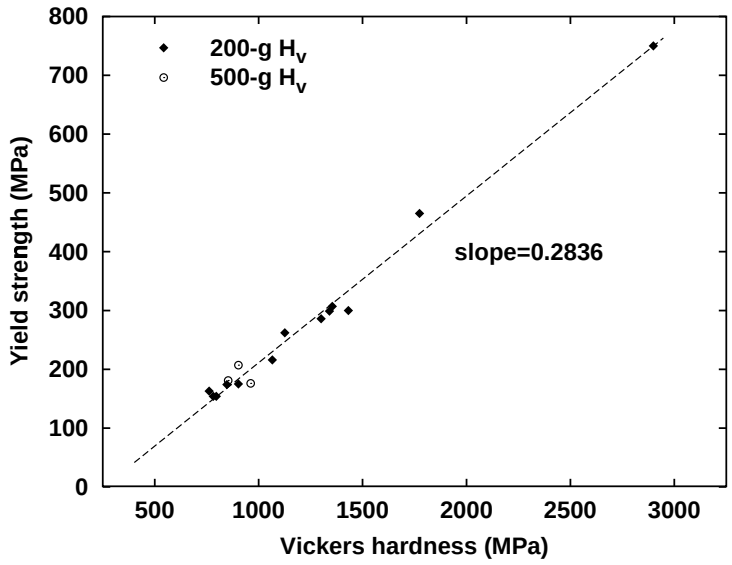

Figure 2. Comparison of yield strength and Vickers hardness for all materials in study.

COMPARISON OF VICKERS AND NANOHARDNESS

Nanoindentation measurements were made on the same specimens as used for the Vickers hardness measurements. Specimens were indented to various depths (50, 100, 400 nm) using the nanoindenter with the hardness determined by averaging ten indents at each depth. The comparison of the nanohardness values and the Vickers hardness values is plotted in Figure 3a. Hardness values determined with the NanoIndenter-II® are based on the ratio of load to projected contact area, which is determined by carefully characterizing the geometry of the indenter tip. Since the indenter contact area is used in the definition of conventional Vickers hardness, the values for nanohardness used in the correlation were multiplied by the ratio of projected to contact area (0.927 for a perfect Berkovich diamond) so that both types of hardness share the same definition. The nanohardness values in Figure 3 are marked with an asterisk to indicate this modification. The nanohardness values show the well-known apparent increase in hardness with

decreasing indent depth [10], which is responsible for the different zero-intercepts in Figure 3a. The physical origin of the zero-offset in Figure 3 is not known, but the hardness changes are consistent for all the alloys. Thus, only the intercept, and not the slope of the ΔH_N vs. ΔH_V line changes and changes in hardness are well correlated. The A533B datum with a Vickers hardness of ~1.75 GPa shows the greatest deviation from the best-fit line obtained for all the data at a given displacement. Overall, the slope of each line is within a few percent of the bottom dashed line which was drawn with a slope of 1.0.

The load required for a 50 nm indenter displacement differs for each alloy. For the alloys tested here, the range was 0.022 g_f (0.216 mN) to 0.043 g_f (0.422 mN). Previously reported comparisons between ultra-low load hardness and Vickers hardness [11,12] yielded a poorer correlation than that reported here. Since those researchers conducted their nanohardness measurements using a constant load instead of a fixed displacement, a further experiment was carried out for purposes of comparison. Using only a subset of five of the alloys, a series of

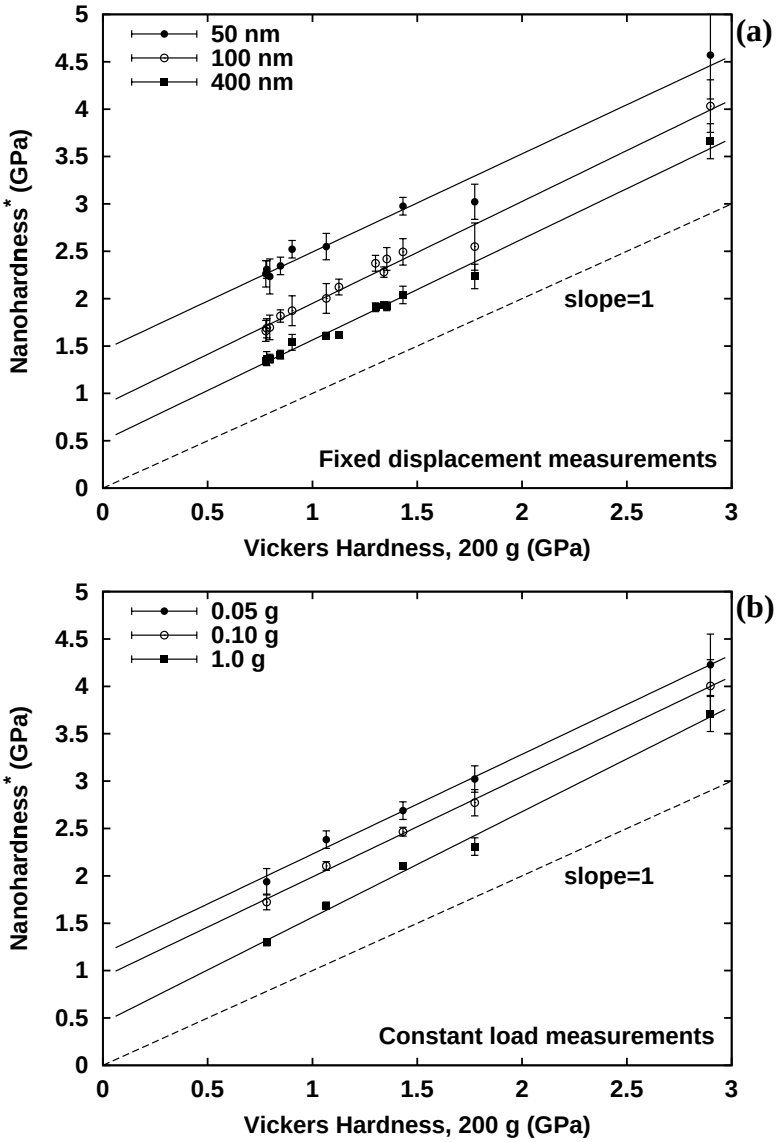

Figure 3. Comparison of nanohardness and 200-g_f (1.96 N) Vickers hardness measured with: (a) fixed displacement, and (b) constant load. *(See text for definition of nanohardness)

measurements were made with constant maximum loads of 0.05, 0.1 and 1.0 g_f (0.490, 0.981, and 9.81 mN). A comparison of the modified nanohardness values and Vickers hardness values is displayed in Figure 3b. The one-to-one agreement between Vickers and nanohardness change is similar to that shown in Figure 3a. The lines for the two lowest loads are both within 5 percent of 1.0, while the line with the 1.0 g_f load has a slope of about 0.90. In this case, the A533B data appears more consistent with the other four alloys. The average value from all six lines in Figures 3a and 3b leads to the following correlation:

$$\Delta H_v\,[GPa] = 0.937\,\Delta H_N\,[GPa] \qquad (1)$$

For these alloys, a load of 0.05 g_f results in an indent depth on the order of 50 nm. Since the size plastic zone is about 7 to 10 times the indent depth, this corresponds to a spatial resolution of less than 0.5 microns [13]. Indents with a 50 nm contact depth proved to provide both sufficient spatial resolution and the ability to produce hardness data with acceptable scatter [2]. Thus, at least for these ferritic and ferritic/martensitic alloys, the data shown in Figure 3 indicate that any change in hardness measured with sub-micron spatial resolution using the nanoindenter is nearly identical to the bulk property change measured in a Vickers hardness test.

CORRELATION OF NANOHARDNESS AND YIELD STRENGTH CHANGES

Using the available data, a correlation between nanohardness and tensile yield strength can be obtained in two ways. The first is derived from the linear relationships shown between Vickers hardness and yield strength in Figure 2 (slope=0.2836 MPa/MPa), and between nanohardness and Vickers hardness in Figure 3 (Eqn. (1)). The product of these slopes yields the following relationship (in the indicated units) between yield strength change and nanohardness change:[1]

$$\Delta\sigma_y\,[MPa] = 266\,\Delta H_N\,[GPa] \qquad (2)$$

Alternately, the tensile data can be plotted against the nanohardness data and the slope obtained directly. Using the tensile data from Figure 2 and the corresponding six sets of nanohardness data from Figure 3, the average relationship obtained is (as it must be) nearly same as Eqn (2):

$$\Delta\sigma_y\,[MPa] = 274\,\Delta H_N\,[GPa] \qquad (3)$$

SUMMARY

Linear correlations have been established between the change in nanohardness and changes in both Vickers hardness and tensile yield strength in ferritic and ferritic/martensitic steels with yield strengths in the range of 150 to 750 MPa. The change in Vickers hardness was found to be about 0.94 times the nanohardness change when both are expressed in the same units. The change in tensile yield strength (in MPa) can be obtained as about 270 times the change in nanohardness (in GPa). The successful correlation of nanohardness measurements with bulk mechanical properties is significant not only because of the validation of the nanoindentation technique, but also because of its implications for ion irradiation studies. This validation supports the application of ion irradiations to simulate the effects of neutron irradiation, allowing studies to be carried out without the complications associated with testing radioactive specimens. For example, reliable estimates of mechanical property changes can be obtained for the high dose conditions reached at the end of fission reactor lifetimes. Similarly, the technique can be used to more rapidly screen

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1. Somewhat lower than the value published previously [3] as a result of further data analysis.

alloys for irradiation performance and to investigate variables such as alloy composition or thermal-mechanical treatment. With the cross-section technique [2,3], is possible to use the nanoindenter to obtain data for a range of doses on a single specimen. In conjunction with TEM observation, changes in mechanical properties can be correlated with microstructural changes, and parameters such as the strength of microstructural obstacles preventing dislocation motion can be measured.

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