Evaluation Technique of the Hardness and Elastic Modulus of Materials with Fine Microstructures*¹

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Quantitative data of mechanical properties such as hardness, H , and elastic modulus, E , are required for the constituent phases of an alloy in the process of alloy design. To meet the needs, the evaluation technique of H and E of phases in composites through nanoindentation tests is proposed. Moreover, H and E of Nb solid solution (Nb_{SS}) and niobium silicide (Nb₅Si₃) phases in Nb-base *in-situ* composites were characterized by the proposed method. To clarify the quantitative relationship between the nanohardness, H_n , and the micro-Vickers hardness, H_v , nanoindentation tests were carried out on the H_v Standard blocks with H_v 100, 500, 700, 900 and 1600, under a wide range of applied loads from 0.1 to 40 mN. As a result, it was clarified that H_v and H_n are linearly related under each applied load. Therefore, H_v could be estimated from H_n by applying the linear relation. It was also confirmed that the elastic modulus is almost independent of the applied loads. Therefore, the elastic modulus, E , could also be directly estimated by nanoindentation tests with Poisson's ratios of tested materials. H_v and E of Nb_{SS} and Nb₅Si₃ in the Nb-base composites determined by the method show good agreement with the reported values for both phases. Accordingly, it is possible to conclude that the proposed method is useful to quantitatively evaluate the hardness and elastic modulus of constituent phases in a composite.

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1. Introduction

Indentation hardness testing has been used to evaluate the mechanical properties of materials. More recently, the advent of nano- and micro-scale science, engineering and technology, coupled with substantial progress in instrumentation, has resulted in depth-sensing indentation. When conducted in a sub-micrometer regime, this is broadly referred to as nanoindentation. A typical measurement via a nanoindenter can record displacement h from the surface of the material and load P with resolutions in sub-nanometer and sub-mN, respectively. From the recorded P - h relations, various characteristics of the individual phases in a composite material, such as the elastic modulus, hardness, the strain-hardening exponent, yield strength, fracture toughness, and residual stress^{1,2)} can be directly estimated. Among these properties, the elastic modulus, E , and hardness, H , are obtainable without complicated testing apparatus and spending. Therefore, a lot of studies concerned with the evaluation of E and H of bulk materials and thin films by nanoindenter have been reported in recent years.³⁻⁵⁾

The micro-Vickers hardness, H_v , which represents the reliable hardness of metals and ceramic materials determined in the micro-scale regime, is one of the most widely used hardness standards. Therefore, comparison with the hardness data obtained by micro-Vickers tests and nano-indentation tests has significant importance, because when a clear relationship between both hardness values is developed, the nanoindentation tests can provide a hardness evaluation that is compatible to micro-Vickers hardness, on nanoscale-size phases. However, there were only few reports that dealt with the relationship quantitatively.

The Nb_{SS}/Nb₅Si₃ *in-situ* composites, which originate from

Nb-Si alloy, exhibit excellent high temperature strength. In this decade, significant progress has been achieved in understanding the mechanical behavior of Nb-base *in-situ* composites, such as their high temperature compression properties,^{6,7)} high temperature tensile properties^{8,9)} and room temperature fracture toughness.¹⁰⁾ However, there are only a few reports available on the hardness, H , and elastic modulus, E , of the constituent phases of Nb_{SS}/Nb₅Si₃ *in-situ* composites, despite of the importance of the property for material design, especially for fracture toughness improvement.

This present study aims to clarify the quantitative relationship between the nanohardness that is determined by nanoindentation tests, H_n , and the micro-Vickers hardness, H_v , on H_v Standard blocks with a wide range of applied loads, and to evaluate the mechanical properties (H_n , H_v and E) of Nb_{SS} and Nb₅Si₃ phases in Nb-base *in-situ* composites of fine microstructure.

2. Experimental Procedure

The nanoindentation experiments were performed at 300 K using the Elionix ENT1100a Nano-Indentation Hardness Tester (Indenter: Berkovich type, $\alpha = 65$ degrees). To clarify the quantitative relationship between the nanohardness, H_n , and the micro-Vickers hardness, H_v , nanoindentation tests were carried out on H_v Standard blocks obtained from Yamamoto Scientific Tool Laboratory Co. Ltd. with H_v 100, 500, 700, 900 and 1600, under a wide range of applied loads from 0.1 to 40 mN. The indentations were arranged in a 5×5 array with 20 μ m spacing on each of the H_v Standard blocks for each of the applied loads.

Nanoindentation tests were also performed on prepared samples of 6 kinds of Nb-base *in-situ* composites of fine microstructures. The materials were prepared by arc casting, and then some of the ingots were subjected to heat treatment at 1870 K for 100 h or at 2070 K for 20 h. The procedure for

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Table 1 Designated names of materials, nominal compositions and heat treatment conditions.

Designated name of materials	Elements and composition (in mol%)	Heat treatment conditions
SMH16	Nb–18Si–5Mo–5Hf	1870 K 100 h
SMH18	Nb–18Si–5Mo–5Hf	2070 K 20 h
SMHC16	Nb–18Si–5Mo–5Hf–2C	1870 K 100 h
SMHC18	Nb–18Si–5Mo–5Hf–2C	2070 K 20 h
SMHCW	Nb–18Si–5Mo–1Hf–1C–2W	As cast
SMTW	Nb–18Si–10Mo–10Ti–15W	As cast

Fig. 1 Back-scattered SEM micrograph of SMHCW. The bright phase is the Nb_{SS} and the dark phase is the Nb₅Si₃.

fabricating Nb-base *in-situ* composites is reported in more detail elsewhere.^{5,6} Table 1 shows the designated names, nominal compositions and conditions of the heat treatments of the materials. Figure 1 shows a typical microstructure of an Nb-base *in-situ* composite. The bright and dark phases are the Nb solid solution (Nb_{SS}) and the niobium silicide (Nb₅Si₃), respectively. The energy dispersive X-ray spectroscopic (EDX) analyses indicated that the matrix phase is the Nb_{SS} and the secondary phase is the Nb₅Si₃ for all Nb_{SS}/Nb₅Si₃ *in-situ* composites. The samples for the nanoindentation tests were electro-discharge machined from a heat-treated/as-cast ingot. After machining, the testing side of the samples was polished to a mirror surface with a vibratory polisher to minimize the negative effect of surface roughness, which is an intrinsic factor that influences the result, and originates during the polishing process due to the difference of properties between the two phases. The indentations were arranged in a 10 × 10 array with 10 μm spacing, so that 100 points in a testing area of 10⁴ μm² were examined on each specimen. The indentation test procedure was as follows: a maximum constant load of 2 mN was applied on the sample for 1 s after initial loading at a rate of 0.4 mNs^{−1}, and then the load was released at the same rate. The applied load, *P*, and the displacement of the indenter, *h*, were precisely measured at a time step of 10 ms with resolutions of 0.78 μN and 0.3 nm, respectively.

According to the method of Oliver and Pharr,¹¹ the data of the indentation load, *P*, and displacement, *h*, were analyzed to determine the reduced elastic modulus, *E_r*, and the nanohardness, *H_n*, by the following relations:

$$E_r = \frac{\sqrt{\pi}}{2\sqrt{A_S}} \frac{dP}{dh} \quad (1)$$

$$H_n = \frac{P_{\max}}{A_S}, \quad A_S = f(h) \quad (2)$$

As seen from the formulas above, in addition to the maximum load on the indenter, *P_{max}*, the contact stiffness, *dP/dh*, between the indenter and the material being tested and the projected contact area, *A_S*, are required to determine *E_r* and *H_n* values. The *dP/dh* was obtained by determining the slope of the initial portion of the unloading curve and the *A_S* was determined by the indenter tip geometry.

The elastic modulus, *E*, of the tested material can be calculated from the following equation:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}, \quad (3)$$

where *E_i* and *ν_i* are the elastic modulus and Poisson's ratio of the diamond indenter (*E_i* = 1050 GPa and *ν_i* = 0.1), and *E* and *ν* are those of the materials being tested, respectively. In the present study, 0.2 and 0.35 were taken as Poisson's ratios of the niobium silicide (Nb₅Si₃) and Nb solid solution (Nb_{SS}), respectively.¹²

3. Results and Discussion

3.1 Nanoindentation tests on *Hv* Standard blocks

Figure 2 shows the obtained relationship between the two hardness values *H_n* determined by nanoindentation tests and

Fig. 2 Linear relationship between nanohardness and micro-Vickers hardness estimated by nanoindentation tests on the *Hv* Standards with a load range of 0.1–40 N.

Table 2 Constants in linear relations of H_n and H_v estimated by nanoindentation tests performed on H_v Standard blocks with a load range 0.1–40 mN and the representative displacement obtained at H_v700 Standard block.

Load (mN)	Displacement in H_v700 (μm)	A	B
0.1	0.0158	46.838	18.740
1	0.0739	102.354	30.890
2	0.1078	117.925	65.169
10	0.2687	154.560	78.354
40	0.5715	176.056	64.820

H_v . The relationship between H_n and H_v was found to be linear for all applied loads in the ranges of 0.1–40 mN as expressed by the following relation:

$$H_v = A \times H_n - B \quad (4)$$

where A and B are constants that depend on the applied load. The constants determined for each of the applied loads are summarized in Table 2, along with the representative displacement obtained with an H_v700 Standard block. It should be noted that decreased applied loads give larger H_n due to the effect of the contacted area size of the indenter, that is, a large enough contacted area size necessary for correct evaluation of the genuine bulk properties could not be attained under the small load of 0.1 mN, so that, the overestimated datum were obtained for all H_v Standard specimens. A load of 0.1 mN was presumably too small to thoroughly penetrate the hard surface layer of H_v Standards. However, as seen from the figure, the standard deviation of nanohardness values obtained with all the applied loads were in the vicinity of 4%, except for those under 0.1 mN that shows a large deviation of about 10–13% for all H_v Standards. Therefore, it was concluded that with an applied load larger than 1 mN, the bulk mechanical properties could be well estimated by the nanoindentation method.

In earlier experiments, it was also confirmed that the elastic modulus, E , remained constant over the entire range of applied loads, which is consistent with the previous report.¹¹⁾ Therefore, the elastic modulus, E , could be directly evaluated from the data from nanoindentation tests with Poisson's ratios of tested materials by the above relation (3).

3.2 Evaluation of the mechanical properties of $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$

The nanohardness, H_n , and elastic modulus, E , of $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$, which are constituent phases of Nb-base *in-situ* composites, were evaluated using a nanoindenter with an applied load of 2 mN, and then, the Vickers hardness, H_v , was calculated by the relation discussed in the previous section. As $\text{Nb}_{50}\text{Si}_{50}$ is hard, brittle and finely dispersed in the composites, an applied load as high as possible was necessary to obtain genuine bulk properties unless fracture of $\text{Nb}_{50}\text{Si}_{50}$ occurs during the indentation. Thus, a load of 2 mN was adapted in nanoindentation tests.

Figure 3 shows the displacement histogram of the indenter obtained for SMHC18. The histogram reveals two distinct peaks around 0.0730 μm and 0.1485 μm . Since the constituent phases $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$ have distinctly different proper-

Fig. 3 Displacement histogram of SMHC18 of Nb-base *in-situ* composite obtained with an applied load of 2 mN.

ties from each other, it is conceivable that each of the peaks in the displacement histogram corresponds to both $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$, respectively. The length of the perpendicular bisector of the projected indentation was calculated to be 0.5018 μm for $\text{Nb}_{50}\text{Si}_{50}$ and 0.9682 μm for $\text{Nb}_{50}\text{Si}_{50}$, which are significantly smaller than both the average particle size of the $\text{Nb}_{50}\text{Si}_{50}$ (1–2 μm) and average width of the $\text{Nb}_{50}\text{Si}_{50}$ channel (2–6 μm), as can be seen in Fig. 1. Therefore, the indentations would mostly be developed on each of $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$, and rarely at the interface of $\text{Nb}_{50}\text{Si}_{50}/\text{Nb}_{50}\text{Si}_{50}$.

The displacement data in the range of 40 nm around each of the peaks were ascribed for each of the phases, and the average values were used in the calculation of nanohardness, H_n , and elastic modulus, E , of $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$. The adopted Poisson's ratios in the elastic modulus calculation were 0.35 for $\text{Nb}_{50}\text{Si}_{50}$ and 0.2 for $\text{Nb}_{50}\text{Si}_{50}$, respectively, as noted previously.

The nanohardness, H_n , of $\text{Nb}_{50}\text{Si}_{50}$ determined by nanoindentation tests was $\approx H_n 4$, except for SMTW that exhibits the highest value of $H_n 6.5$, and that of $\text{Nb}_{50}\text{Si}_{50}$ was $\approx H_n 13$ –14. Table 3 shows the nanohardness, H_n , and the micro-Vickers hardness, H_v , converted by the linear relation with the constants for an applied load of 2 mN in Table 2. The micro-Vickers hardness of $\text{Nb}_{50}\text{Si}_{50}$ is calculated to be about 340–480 for all tested materials, except for SMTW, which contains a large amount of solution strengthening elements

Table 3 Hardness values ($H_{n2\text{mN}}$) of $\text{Nb}_{50}\text{Si}_{50}$ and $\text{Nb}_{50}\text{Si}_{50}$ measured with an applied load of 2 mN and its calculated values in Vickers hardness (H_v).

Materials	$\text{Nb}_{50}\text{Si}_{50}$		$\text{Nb}_{50}\text{Si}_{50}$	
	$H_{n2\text{mN}}$	H_v	$H_{n2\text{mN}}$	H_v
SMH16	3.97	400	14.70	1670
SMH18	3.98	400	14.04	1600
SMHC16	4.46	460	13.28	1500
SMHC18	3.44	340	14.23	1610
SMHCW	4.56	480	13.06	1470
SMTW	6.50	700	13.07	1480

Table 4 Elastic moduli of Nb_{SS} and Nb₅Si₃ measured with an applied load of 2 mN.

Materials	Nb _{SS} (GPa)	Nb ₅ Si ₃ (GPa)
SMH16	140	420
SMH18	150	380
SMHC16	140	360
SMHC18	130	400
SMHCW	150	370
SMTW	200	360

such as W, Ti and Mo. The Vickers hardness of Nb₅Si₃ was almost constant in the range of 1470–1670. These values are in reasonable agreement with the reported micro-Vickers hardness values of H_v500 –700 and H_v1200 –1300 for the Nb_{SS} and Nb₅Si₃, respectively, in the Nb–18Si–22Ti– x Mo ($x = 0, 10, 20$, and 30) *in-situ* composites prepared by directional solidification technique.³⁾ It is concluded that the micro-Vickers hardness can be evaluated quantitatively from the relationship of H_n and H_v by nanoindentation tests.

The elastic modulus, E , determined by a nanoindenter was 130–200 GPa and 360–420 GPa for the Nb_{SS} and Nb₅Si₃, respectively, as listed in Table 4. The estimated elastic modulus in the present study is in good agreement with reported values, which were evaluated for both phases in SMHC18 with an applied load of 9.81 mN in the previous report.⁹⁾ Considering that the elastic modulus shows almost no dependence upon the applied load in the nanoindentation tests, E obtained from formula (3) could be regarded as reasonable. The estimated elastic modulus of the Nb₅Si₃ phase is somewhat greater than that ($E \approx 326$ GPa) of monolithic Nb₅Si₃ fabricated by the powder metallurgy method (P/M).¹³⁾ The difference would be attributed to the alloying elements in the Nb₅Si₃ phase in the composites. As described in the present study, the nanoindentation technique is applicable to evaluate the hardness and elastic modulus of fine constituent phases of *in-situ* composites.

4. Summary

The evaluation technique of elastic modulus and hardness of constituent phases of finely dispersed *in-situ* composites through nanoindentation tests was proposed. The elastic modulus and hardness of Nb solid solution and niobium silicide in Nb base *in-situ* composites were characterized to verify the proposed method. The obtained results were as follows:

(1) The nanohardness, H_n that is directly determined by

nanoindentation tests, and micro-Vickers hardness, H_v , exhibited a linear relationship for the whole applied load range of 0.1–40 mN. Thus, H_v could be directly determined from the relation that calibrates the obtained H_n under an applied load.

- (2) The elastic modulus showed independence of the applied loads, and therefore it could be directly calculated from the obtained data from nanoindentation tests with Poisson's ratios of tested materials.
- (3) The estimated Vickers hardnesses are H_v340 –700 for Nb_{SS} and H_v1470 –1670 for Nb₅Si₃, and the elastic moduli are 130–200 GPa for Nb_{SS} and 360–420 GPa for Nb₅Si₃, respectively, which is in good agreement with the reported values for both phases. Therefore, it is possible to conclude that the proposed method is useful to evaluate the hardness and elastic modulus quantitatively.

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