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Advanced in situ multi-scale characterization of hardness of carbon-fiber-reinforced plastic

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In situ multi-scale characterization of hardness of carbon-fiber-reinforced plastic (CFRP) is demonstrated by a traditional hardness tester, instrumented indentation tester and atomic-force-microscope (AFM)-based nanoindentation. In particular, due to the large residual indentation and nonuniform distribution of the microscale carbon fibers, the Vickers hardness could not be calculated by the traditional hardness tester. In addition, the clear residual microindentation could not be formed on the CFRP by instrumented indentation tester because of the large tip half angle of the Berkovich indenter. Therefore, an efficient technique for characterizing the true nanoscale hardness of CFRP was proposed and evaluated. The local hardness of the carbon fibers or plastic matrix on the nanoscale did not vary with nanoindentation location. The Vickers hardnesses of the carbon fiber and plastic matrix determined by AFM-based nanoindentation were 340 ± 30 and 40 ± 2 kgf/mm², respectively.

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1. Introduction

Hardness is a functional indicator of characterization of soft or hard materials. Generally, it is the local area mechanical properties of materials response to the overall performances under certain conditions. Hardness is a measurable parameter, mainly used to test the mechanical quality of materials and to determine a reasonable processing technology. Hardness can be measured by a traditional hardness tester1) or instrumented indentation tester.2) Because of residual indentation at the microscale to millimeter range, surface is slightly damaged by the traditional hardness tester. According to international standards of the instrumented indentation technology, the hardness measurement range is divided into micro and nano scales. Generally, it is the local area mechanical properties response to the overall performances of materials,3–5) Atomic-force-microscope (AFM)-based nanoindentation is an effective approach for characterizing the true hardness of composite materials.6–8) In particular, the mechanical properties of monolayer graphene flakes are measured by AFM-based nanoindentation.9,10) Since AFM has a high longitudinal and lateral resolution, it is suitable for measuring nanoscale surface topography. Thus the surface area of residual nanoindentation can be calculated directly by AFM.

Carbon-fiber-reinforced plastic (CFRP)9) is one of the most-advanced composite materials with a high-strength structure. This kind of composite material contains two parts: a carbon-fiber-reinforcement part (which provides strength) and a plastic-matrix part (which binds the reinforcement together). Since CFRP is lighter than aluminum, stronger than iron, and has higher elasticity than titanium, it is widely used for industrial applications such as aerospace,11–13) automobile,14,15) civil engineering,16,17) and sports goods.18) The achievements of modern industry have benefited greatly from testing and evaluation of the mechanical properties of materials. In practice, testing and evaluation of the hardness of materials are a direct bridge connecting material design and industrial applications. Consequently, for developing CFRP in various industrial fields, it is very important to understand the hardness of CFRP.

As commonly known, because single-crystal silicon (001) has high flatness and low surface roughness, it is often used as a standard material for characterizing mechanical properties.19,20) However, composite materials such as CFRP are composed of complex multiphase systems of heterogeneous materials. The hardness of CFRP are related not only to the intrinsic properties of the components, namely, carbon fibers and plastic matrix, but also to the characteristics of the interface region between the carbon fibers and plastic matrix.21–24)

In the present research, the hardness of CFRP are characterized on a multi-scale for the first time. Importantly, the goal of this research is to develop an efficient and reliable technique for characterizing the true hardness of composite materials.

2. Experimental methods

Before the mechanical properties were characterized, CFRP samples (TORAY P2053-17) were cut into rectangular sheets with length of 10 mm, width of 3 mm, and thickness of 1 mm. The cross sections of the samples were then polished by Ar+ beam (a cross-section polisher, JEOL IB-09020CP, 8 kV) for 15 h. Next, the hardness of CFRP were in situ multi-scale characterized in the following steps.

First, the macroscale hardness of CFRP was characterized by a micro Vickers hardness tester (Shimadzu HMV-G21 series). The micro Vickers hardness tester has a standardized automatic length measurement function using a CCD camera. The indentation measurement resolution is 0.09 µm with 40 times objective lens. The tip half angle of a diamond Vickers was 68°. The loading forces were from 9.8 to 245 mN, and were applied for 10 s in each indentation test.

Second, the microscale hardness of CFRP was characterized by the instrumented indentation tester (Hysitron TI950 Triboindenter). The indenter was a diamond Berkovich with tip half angle of 65.3°. The loading forces were from 1.5 to 7.5 mN and applied for 30 s in each indentation test.

Third, the nanoscale hardness of CFRP was characterized by AFM-based nanoindentation (Bruker AXS Multimode 8), which was set in a glove box filled with Ar. The diamond
probe glued onto a rectangular silicon-etched cantilever was used from Artech Carbon. The sample was scanned so that smooth carbon fibers or plastic matrix could be selected. A carbon fiber or plastic matrix were nanoindentation tested. Sample images after the nanoindentation tests were obtained in contact mode. The loading forces applied were from 0.7 to 8 µN.

3. Results and discussion

3.1 Characterization of macroscale hardness by a traditional hardness tester

As shown in Fig. 1(a), marks with different sizes were formed on the silicon. The marks looked like a quadrangular pyramid, namely, the shape of the Vickers indenter. The loading forces were 9.8, 19, 49, and 98 mN, which correspond to the marks from left to right in Fig. 1(a).

Since silicon is very brittle, it is easy to produce a stress concentration on the edge of the pyramid. Two phenomenon, namely, chipping and radial cracks, occurred at the corners of the pyramid. The standard mark without any stress concentration was selected as the one for calculating the Vickers hardness, \(HV\) (kgf/mm\(^2\)), of silicon. The Vickers hardness was determined as loading force, \(F\) (kgf), divided by the surface area, \(S\) (mm\(^2\)), of indentation as

\[
HV = \frac{F}{S}.
\]

If the unit of force is the newton (N), the Vickers hardness can be expressed as

\[
HV = 0.102 \frac{F}{S}.
\]

Kilogram-force is the gravitational unit of force. The average length of the diagonal line of the marks \(d\) (mm), could be read directly by the software of the micro-Vickers-hardness tester. Lastly, the Vickers hardness was calculated as

\[
HV = 0.102 \frac{2F \sin \theta}{d^2} = 0.1891 \frac{F}{d^2},
\]

where \(\theta\) is the tip half angle (68°) of the Vickers indenter, which is the angle between the axis of pyramid and its face. The indentation test on the silicon was done for 15 times. The Vickers hardness of silicon was determined from Eq. (3) as 1050 ± 20 kgf/mm\(^2\). This value fits into a range of Vickers hardness given in the literature, namely, from 918 to 1173 kgf/mm\(^2\).

Cross sections of the CFRP samples are shown in Figs. 1(b) and 1(c). The bright white circles are carbon fibers; other (gray) areas are plastic matrix. A typical diameter of carbon fibers is about 7 µm. The packed carbon fibers after indentation test is shown in Fig. 1(b) under a loading force of 245 mN. The surface area of indentation could not be calculated because there was no a quadrangular-pyramid hole formed on the CFRP. Although the loading force was decreased to 98 mN or even small, the quadrangular-pyramid hole could not be formed yet in Fig. 1(c). The carbon fibers debonded from the plastic matrix at the millinewton range of the applied force.

Due to the large residual indentation and nonuniform distribution of the microscale carbon fibers, it was difficult to measure the hardness at macro scale by the traditional hardness tester. The hardness of CFRP were therefore tried to be characterized at micro scale by the instrumented indentation tester.

3.2 Characterization of microscale hardness by the instrumented indentation tester

As shown in Fig. 2(a), the triangular-pyramid marks formed on the silicon represent the shape of the Berkovich indenter. The marks become enlarged with increasing applied forces up to the millinewton range. The Vickers hardness of silicon (HV) are calculated as 1180 ± 30 kgf/mm\(^2\) by the Oliver–Pharr method\(^{26,27}\) as follows:

\[
HV = \frac{P_{\text{max}}}{A_c},
\]

\[
A_c = 3 \sqrt{3} h_c^2 \tan^2 \theta = 24.56 h_c^2,
\]

\[
h_c = h_{\text{max}} - \frac{e P_{\text{max}}}{S},
\]

\[
S = \left( \frac{dP}{dh} \right)_{h=nh_{\text{max}}},
\]

The relationship between loading force \(P\) and displacement \(h\) is shown in Fig. 3. The slope of the curve in the figure (black-dotted line) is indicative of the contact stiffness.
(S) [given by Eq. (7)] upon unloading and was used to calculate the depth of indentation (hc) [given by Eq. (6)]. Constant ε (which is related to the shape of the Berkovich indenter) was 0.75. The depth was used to calculate the projected area (Ac) of indentation [Eq. (5)], where θ is the tip half angle (65.3°) of the Berkovich indenter, which was between the axis of pyramid and its face. The Ac (hc) function of the Berkovich indenter is calibrated according to a standard procedure on a reference fused silica sample.28

The value of the Vickers hardness of silicon is fairly consistent at macro- (1050 ± 20 kgf/mm²) and microscale (1180 ± 30 kgf/mm²) characterization and fits into the range of Vickers hardness given in the literature from 918 to 1173 kgf/mm².25 Due to the different measurement techniques and calculation methods for the Vickers hardness of silicon between macro- and microscale characterization, 13% difference was reasonable and acceptable.

In order to get clear holes on the carbon fibers, the loading force was decreased from 9.8 to 4.5 mN [Fig. 2(c)], which was the maximum of the indenting apparatus. As shown in Figs. 2(b) and 2(c), the marks of a cross formed on the plastic matrix and carbon fiber are the diagonal lines of a triangular pyramid. Due to the approximate size of the tip half angle between the Vickers and Berkovich indenter, the clear triangular marks could not be formed on the carbon fiber under a loading force of 4.5 mN [Fig. 2(c)]. Therefore the Vickers hardness of CFRP could not be calculated at microscale by the instrumented indentation tester when no holes were formed.

To produce a clear mark on the CFRP, a sharp nanoscale diamond probe of AFM (see Fig. 4) was used in place of the Berkovich indenter. The tip apex was a spherical shape with radius of about 20 nm after the nanoindentation test. The value of the Si(001) hardness was 1173 kgf/mm² (Fig. 5(a)).

Fig. 2. (Color online) (a) AFM images of single-crystal Si(001), (b) plastic matrix (PM), and (c) carbon fiber (CF) after microscale indentation test using a diamond Berkovich indenter. Loading force: (a) 1.5, 3, 4.5, 6, and 7.5 mN; (b) 0.5 mN; (c) 4.5 mN. The bright circles (in the black-dotted circle) are carbon fibers.

Fig. 3. Dependence on applied loading force of displacement of a triangular-pyramid hole formed on the silicon.

Fig. 4. (Color online) Helium-ion-microscope (HIM) images of nanoscale diamond probe. (a) and (b): side views of the probe after nanoindentation test.
Although the polishing caused a nanoscale height difference, it did not have a strong influence on the shape of residual nanoindentation.

Because the hardnesses of the plastic matrix and carbon fibers differ significantly, a nanoindentation test was performed on the plastic matrix and carbon fibers separately. The nanoscale holes formed on the plastic matrix [image (c)] and on the carbon fiber [image (d)] by a diamond AFM probe become enlarged with increasing applied force up to micronewton range. The nano-holes close to and away from the interface (interface region) do not obviously change shape when a constant loading force is applied. In other words, the local hardness of the carbon fiber or plastic matrix on the nanoscale do not vary with nanoindentation location.

The Vickers hardness can be expressed by Eq. (4) as the inverse of the slope in Fig. 6. Vickers hardnesses of silicon (green square), carbon fiber (red circle), and plastic matrix (blue triangle) were successfully determined by AFM-based nanoindentation as 640 ± 60, 340 ± 30, and 40 ± 2 kgf/mm², respectively. Comparing the literature-quoted value between the graphite (from 7 to 11 kgf/mm²) and diamond-like carbon (from 2000 to 9000 kgf/mm²) reveals that the carbon fibers of CFRP is harder than the graphite, and softer than the diamond-like carbon.

The Vickers hardness of silicon on the nanoscale is found to be smaller than that on the macro- and microscales. But roughly speaking, the Vickers hardness of silicon on the multi-scale was the same reported value in order of magnitude (10⁴). Therefore, the consistent hardness of the homogeneous materials could be obtained by a traditional method or AFM-based nanoindentation.

4. Conclusions

In situ multi-scale techniques for characterizing the hardness...
of composite materials such as CFRP were evaluated. Due to the large residual indentation and nonuniform distribution of the microscale carbon fibers, the Vickers hardness could not be calculated by a traditional method, such as micro Vickers hardness tester. Although the loading force was decreased to get a small size of residual indentation on the carbon fiber, the clear marks of residual indentation could be formed. The Vickers hardness of CFRP could not be calculated by the instrumented indentation tester. Accordingly, a technique called AFM-based nanoindentation was found to be an effective approach for measuring the true nanoscale hardness of CFRP. The local hardness of the carbon fiber or plastic matrix on the nanoscale did not vary with nanoindentation location. The clear holes were made by a nanoscale diamond probe. The Vickers hardness of the carbon fiber and plastic matrix were determined by AFM-based nanoindentation as 340 ± 30 and 40 ± 2 kgf/mm², respectively. Our method (AFM-based nanoindentation) can be used for characterization of hardness of composite materials as a standard method. It is no doubt to be used for the homogeneous materials.

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