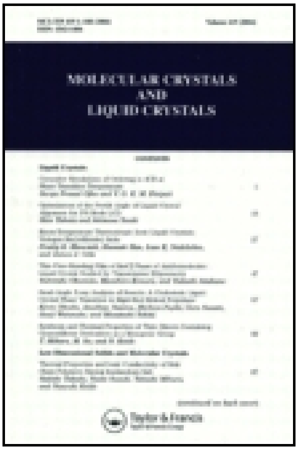
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ELASTO-PLASTIC DEFORMATION OF GLASSY CARBON BY NANO-INDENTATION WITH SPHERICAL TIPPED INDENTERS

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Nano-indentation of glassy carbons (GCs) heat-treated at different temperatures was carried out using spherical tipped indenters with different radii. Analysis of the force-displacement data enabled indentation stress-strain curves to be generated. It was found that the elastic modulus and the yield stress of GCs decreased with increasing the heat treatment temperature. It was also observed that the radius of spherical tipped indenters depended on the yield stress. From a simple the power law fitting of a non-linear elasto-plastic (contact stress-strain) deformation, beyond the initial elastic deformation, a better measure of hardness for brittle materials is proposed.

Keywords: elesto-plastic deformation; glassy carbon; nano-indentation

INTRODUCTION

Nano-indentation was defined as "continuous-depth-sensing indentation testing" [1]. Nano-indentation with several micron radius spherical tipped indenters provides the ability to measure both elastic and plastic deformation of brittle materials. Field and Swain [2] proposed the analysis of the stress ($P/\pi a^2$)-strain (a/R) curves derived from the indentation force (P)-displacement (h) curves with spherical tipped indenters. They also found that the indentation force-displacement behavior for glassy carbon (GC) exhibited almost complete recovery with a significant hysteresis between loading and unloading when indented with a 10 μm radius spherical tipped indenter [3]. From the analysis of the stress ($P/\pi a^2$)-strain (a/R) curves, GC was shown to be elastic at small strains, and non-linear above a threshold contact pressure was observed because of elasto-plastic deformation. In the present work, nano-indentation of GCs heat-treated at different temperatures was carried out using spherical tipped indenters with different radii. The effects of crystal and pore structural changes of the GCs and of radius of spherical tipped indenter on the indentation elasto-plastic deformation behavior are discussed from stress-strain curve of the indentation test results.

EXPERIMENTAL

Two commercially available glassy carbons, GC10 and GC20, heat-treated at 1000 and 2000°C, respectively, (manufactured by Tokai Carbon Co. Ltd.) were used. The surface of the samples, which was cut after carbonization of a block of precursor thermosetting resin, was highly polished using diamond slurry with several micron diameter grains.

Load cycle indentation was performed using a commercial ultra micro-indentation system (UMIS-2000, CSIRO Australia) with sphero-conical diamond indenters with tips of nominally 1, 3 and 10 μm radii. The radius of the spherical indenters and the projected area of the pyramidal indenters were calibrated using silica glass, of which the elastic modulus is 68 GPa and Poisson's ratio is 0.2. Each test was repeated ten times at different positions on the polished surface of the specimen.

For the force-displacement response of a spherical indenter on "elastic" surface, Hertz derived the basic relationship between total penetration, h_t, and applied force, P,

$$h_t = \left(\frac{3P}{4E^*}\right)^{2/3} \cdot \left(\frac{1}{R}\right)^{1/3}, \tag{1}$$

where R is the radius of spherical indenter and E^* the composite modulus. Providing that $h_t \ll R$, the radius of the contact circle may be obtained from $a = \sqrt{R \cdot h_c}$. Thus, Eq. (1) can be rewritten as

$$\frac{P}{\pi a^2} = \left(\frac{4}{3\pi}\right) E^* \cdot \frac{a}{R}. \tag{2}$$

The effective strain associated with a spherical tipped indenter may be written as the ratio of radius of contact circle to the radius of indenter, a/R. A value of elastic modulus E^* is estimated from a linear fitting for the first several points within the linear region on the plots of $(P/\pi a^2)$ versus (a/R).

RESULTS AND DISCUSSION

The stress $(P/\pi a^2)$-strain (a/R) curves of GCs by the indentation with spherical tipped indenters are shown in Figure 1. Average values of elastic modulus of GC10 and GC20 are 31.8 and 27.4 GPa, respectively. Thus, it is pointed out that the elastic modulus of GCs decreases with increasing heat treatment temperature.

A non-linear relation is observed beyond the yielding point after the initial elastic deformation on the plots of $(P/\pi a^2)$ versus (a/R). Since the secondary mechanism, *i.e.*, elesto-plastic deformation, is increasingly more

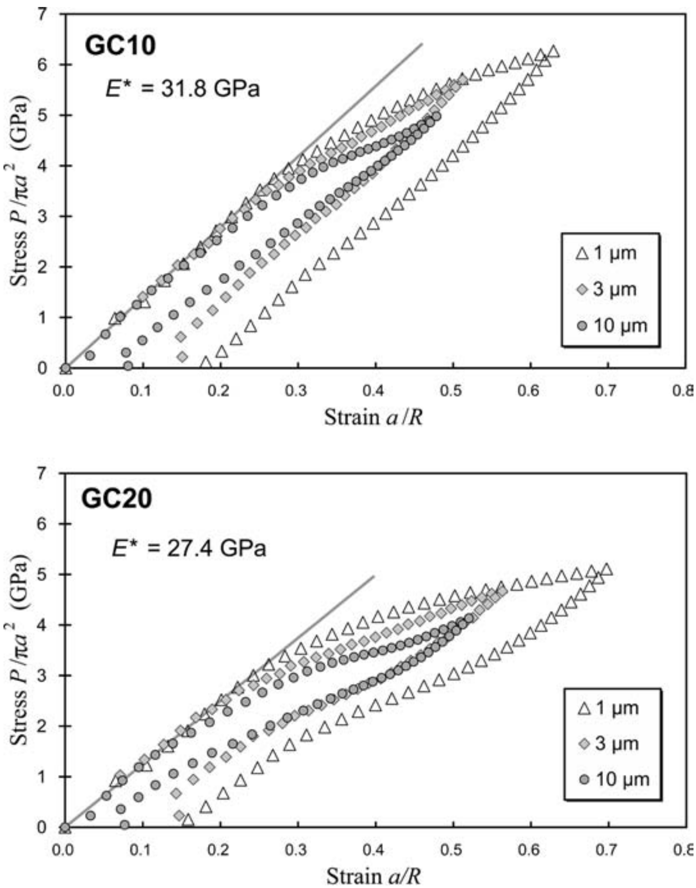

FIGURE 1 The stress $(P/\pi a^2)$-strain (a/R) curves of glassy carbon, GC10 and GC20, by the nano-indentation with spherical tipped indenters with different radii.

obvious at the higher terminal force, it may be described by a simple power law relationship,

$$\frac{P}{\pi a^2} = k \cdot \left(\frac{a}{R}\right)^n . \quad (3)$$

The power law constants, k and n, were estimated from log-log plots of the non-linear portion of the stress-strain curves. By the indentation of both GCs, the values of yield point (yield stress, yield strain and yield contact radius) and the power law fitting constants k and n are summarized in Table 1.

Not only elastic modulus, but also yield stress decreases with increasing heat treatment temperature of glassy carbon. The reduction of the elastic modulus and the yield stress is predicted to result from a corresponding decrease in bulk density of GC with the elevation of HTT and a decrease in shear modulus (rigidity) of the carbon hexagonal network layers with development of the graphitic structure.

It is clearly observed that the yield stress depended on the radius of spherical tipped indenters: the higher yield stress was shown by the indentation with the smaller radius indenter.

For polycrystalline metallic materials, it has been known that the grain size depends on their yield stress, as the Hall-Petch relationship. For nano-indentation of brittle materials, it is reasonably assume that the size of indenter may affect on the yielding behavior. The plots of the yield stress

TABLE 1 Yield Stress, Yield Strain, Yield Contact Radius and Constants of Power Law Fitting Obtained from Stress-strain Curves with Different Radius Spherical Tipped Indenters.

Sample radius of indenter	1 μm		3 μm		10 μm	
			GC10			
yield stress [GPa]	4.1	(0.4)	3.4	(0.4)	2.7	(0.4)
yield strain [–]	0.29	(0.04)	0.25	(0.03)	0.19	(0.03)
yield contact radius [μm]	0.23	(0.04)	0.73	(0.09)	1.72	(0.33)
n [–]	0.60	(0.06)	0.69	(0.07)	0.65	(0.10)
k [GPa]	8.53	(0.50)	8.90	(0.61)	8.30	(1.05)
			GC20			
yield stress [GPa]	3.2	(0.3)	2.5	(0.4)	1.9	(0.4)
yield strain [–]	0.31	(0.04)	0.20	(0.04)	0.15	(0.03)
yield contact radius [μm]	0.25	(0.04)	0.54	(0.11)	1.47	(0.30)
n [–]	0.46	(0.09)	0.56	(0.05)	0.60	(0.10)
k [GPa]	6.21	(0.55)	6.29	(0.40)	6.23	(0.67)

The numbers in brackets are standard deviation.

versus the yield contact radius, a_{yield}, of the spherical tipped indenter, derived from the yield strain $(a/R)_{\mathrm{yield}}$, are shown in Figure 2.

It has been reported that the Meyer hardness, or mean contact pressure during indentation of brittle materials, is not a measure of "plasticity" or yield behavior as for metallic materials, but an elasto-plastic parameter that significantly depends on the geometry of pyramidal indenters [5]. Sakai [6] has proposed the concept of "true hardness", which is a hypothetical perfectly plastic contact with a sharp conical or pyramidal indenter of apical angle $\psi = 0°$, in other words, of face angle $\beta = 90°$, as a characteristic material measure of plasticity.

It was previously proposed that the power law fitting constant k, which is the stress at the indentation strain $(a/R) = 1$ (face angle: $\sin^{-1}(a/R) = 90°$), corresponds to a better measure of "true hardness" for spherical tipped indenter [4]. It is also found for both GCs that the power law fitting constant k is almost independent of the radius of spherical tipped indenters: 8.6 and 6.2 GPa for GC10 and GC20, respectively.

It may be supposed that the plots of the yield stress versus yield contact radius (Fig. 2) can be extrapolated to the constant k derived from the power law fitting by the nano-indentation of each GC, at $a_{\mathrm{yield}} = 0$, though the relation between the yield stress and the indenter size is not persuasively understood yet.

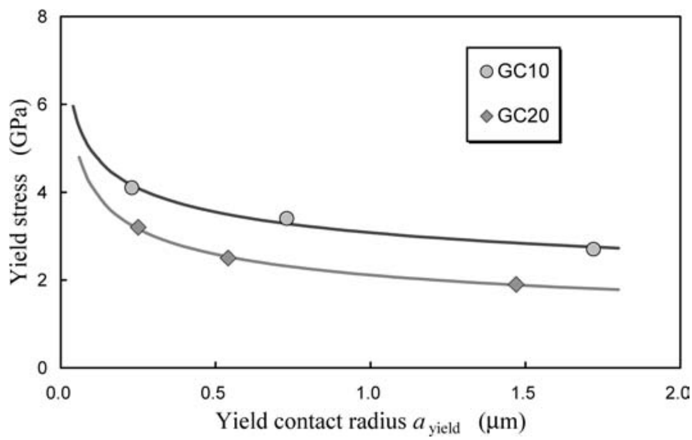

FIGURE 2 Plots of yield stress versus yield contact radius a_{yield} of glassy carbon, GC10 and GC20, by the nano-indentation with spherical tipped indenters with different radii.

CONCLUSION

By nano-indentation with spherical tipped indenters of glassy carbons heat-treated at different temperature, effects of a slightly structural change on their mechanical properties (elastic modulus and elasto-plastic deformation) can be detected. It was found that the elastic modulus and the yield stress of glassy carbons decreased with increasing the heat treatment temperature. Although it was clearly observed that the radius of spherical tipped indenters depended on the yield stress, the constant k obtained from the power law fitting of the non-linear elasto-plastic deformation was almost independent of the radius of spherical tipped indenters. Therefore, it is concluded that the constant k, which is the stress at the indentation strain $(a/R) = 1$ (face angle: $\sin^{-1}(a/R) = 90°$), corresponds to a measure of "plasticity" for the nano-indentation with spherical tipped indenter, namely, the value of k is the characteristic material measure for hardness of glassy carbon.

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