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MICROHARDNESS-DEPTH PROFILES OF Si/SiC LAYERED STRUCTURES PREPARED BY CHEMICAL VAPOUR DEPOSITION

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Abstract. Microhardness-depth profiles of Si/SiC layered structures prepared by chemical vapour deposition are studied by indenting with the pyramids of Knoop and of Vickers. In this way it is possible to reveal strained regions resulting from the large lattice mismatch between Si and SiC.

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1. Introduction

The increasing interest in studying Si/SiC layered structures prepared by Chemical Vapour Deposition (CVD) is due to their potential application in various wide band-gap devices [1]. Having in mind the large ($\approx$20 %) lattice mismatch of Si and SiC, these studies have to include detailed investigation of texture and mechanical properties. The texture evolution in the structures is studied in [1]. As a step in the complex material characterization, in the present work microhardness-depth profiles are investigated.

2. Experimental

2.1. Samples

Layers of SiC and Si were sequentially deposited on Si (001) substrates by atmospheric pressure CVD, using SiH_4 and C_3H_8 for growing the SiC layers

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and SiH_4 for growing the Si ones. The pre-growth preparation of the substrates included hydrogen etches at 1100 °C for 5 min *in situ* to remove the native oxide. Before introducing the mixture of SiH_4 and C_3H_8, the substrate surface layer was converted into SiC by reaction with C_3H_8. The details of the growth procedure are described in [1].

The study of the texture evolution in the Si/SiC multilayers shows that the first SiC layer grown on the Si substrate is monocrystalline and epitaxial while the Si layer deposited onto the SiC one adopts predominantly (110)-oriented columnar structure. The columns extend throughout the layer thickness [1].

2.2. Microhardness Measurements

Microhardness measurements were carried out at room temperature using a PMT-3 tester [2]. Having in mind the possible mechanical stress of the material under load, all the samples were indented in the same manner, initially with the pyramid of Knoop, followed by that of Vickers. The load to the indenter P was varied in the interval 5-200 g in order to vary the penetration depth h. The step of the load variation was 1 g in the vicinity of an expected interface and 5 g in the other regions of the heterostucture. At least five indentations were produced with a given P. The measured values of microhardness H were regarded as composite since all the components of the structures were deformed under the applied load.

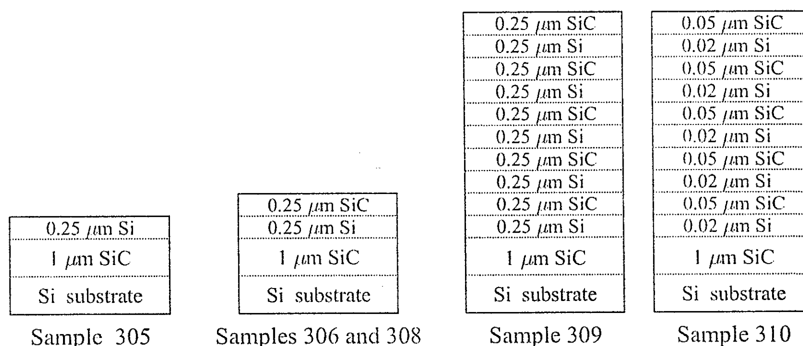

Fig. 1. Schemes of the Si/SiC structures

The configuration of the studied Si/SiC layered structures is shown schematically in Fig. 1. Samples 306 and 308 differ slightly in their preparation regimes.

The experimental points in the microhardness-depth plots (Figs 2-5) are fitted using the computer program TableCurve (Jandel Scientific); the 99 % confidence intervals of the fitting curves are also shown.

3. Results and Discussion

The microhardness-depth profile of sample 305, taken with the indenter of Knoop, is given in Fig. 2. As it can be seen, at small h, in the top Si layer, very high values of H are measured unlike the ones in the Si substrate. This is presumably connected with the Indentation Size Effect (ISE), which is characteristic for measurements at phase boundaries, such as layer surfaces, layer/substrate or layer/layer interfaces, etc. [3]. The observed minimum in the plot at $h \simeq 0.25\ \mu$m illustrates the transition to the underlying SiC layer. A relatively constant microhardness value ($H \simeq 13$ GPa) is registered in the interval $h \in (0.4-0.7)\ \mu$m. This value may be accepted as a characteristic one for the SiC layer. At bigger h, H gradually decreases as the top of the indenter approaches the Si substrate. In the latter, $H \simeq 10$ GPa, which is the microhardness value of the substrate material. In the same figure are presented also the experimental data taken with the indenter of Vickers. Due to geometry, its penetration depth is bigger which makes the subsurface region of the structure inaccessible for measurement.

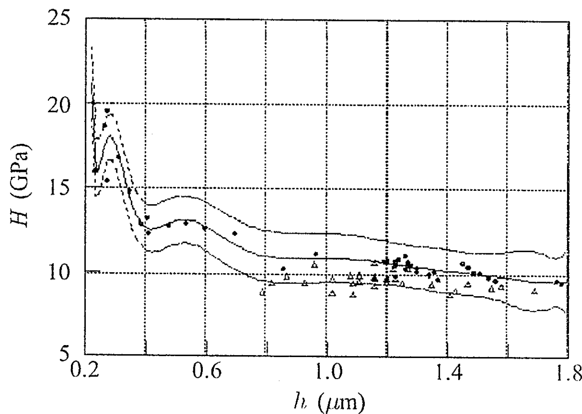

Fig. 2. $H-h$ plot of sample 305
• — Knoop and △ — Vickers data

The adding of a harder top layer of SiC (samples 306 and 308) results in bigger values of H at small h (Fig. 3). At greater h, the plots of samples 305 and 306 (308) are essentially the same.

The $H-h$ profile of sample 309 is presented in Fig. 4. As it can be seen, the first experimental points are taken away from the top surface, at $h > 0.4\ \mu$m. This results from the roughness of the surface of this particular sample that affects the quality of the imprints and makes the measurements in the ISE-governed region unreliable. However, a well-resolved minimum is detected at

$h \simeq 50\ \mu m$, which corresponds to the phase boundary between the subsurface Si layer and the underlying SiC one. Keeping its wavy character, reflecting the variation of the chemical content of the layers, at greater h the dependence shows an overall decreasing of H with h as in the previous samples.

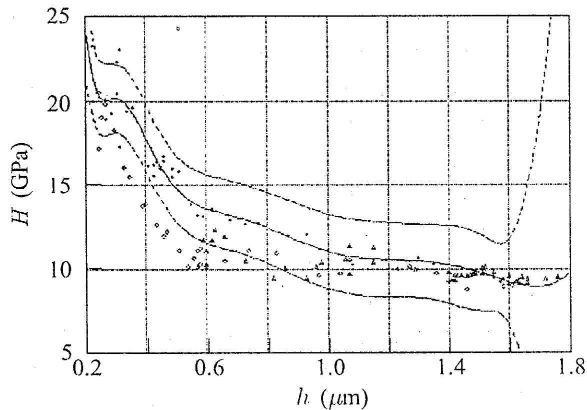

Fig. 3. H-h plots of sample 306 (• — Knoop and △ — Vickers data) and sample 308 (◇ — Knoop data)

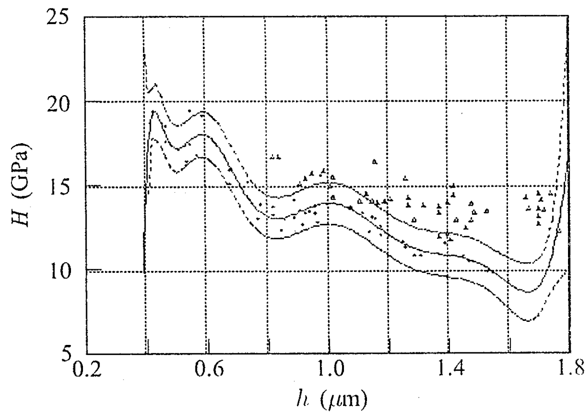

Fig. 4. H-h plot of sample 309
• — Knoop and △ — Vickers data

Having in mind that the total thickness of the Si/SiC multilayers in this sample is $\approx 250\ \mu m$ and that the maximum penetration depth of the Knoop indenter is $\approx 1.73\ \mu m$, it is clear that the plot does not include four layers and the substrate.

As it can be seen in Fig. 4, the Vickers values of H are greater than the Knoop ones. This contradicts the usual situation, observed in the previous samples as

well, in which the Knoop microhardness values are greater as the penetration depth is smaller and the ISE is consequently more pronounced. The observed experimental fact in sample 309 is presumably due to the stresses produced by indentation of this structure which is obviously strained by origin as it consists of a number of layers of Si and SiC with the cited great lattice mismatch.

The H-h profile of sample 310 is presented in Fig. 5. It can be seen that no minima of the curve are observed which implies that the resolution of the used technique is insufficient in the case of very thin layers; presumably, a better resolution would provide the method of nanohardness measurements. At $h \in (0.6-1.2)$ μm, in the thick SiC layer, H is of relatively constant value (10.5 GPa). As in the previous sample, the Vickers values of H are bigger than those of Knoop but the difference is smaller which obviously is an indication of smaller strain.

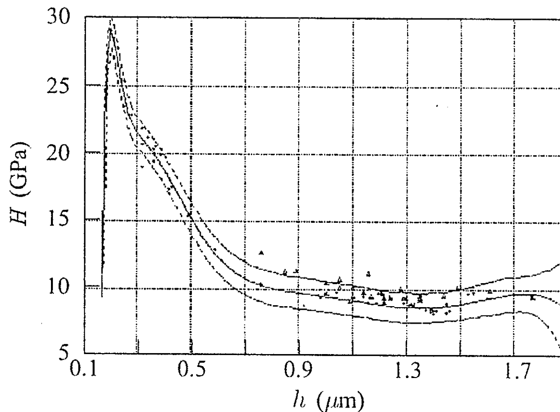

Fig. 5. H-h plot of sample 310
• — Knoop and △ — Vickers data

In this way, the H-h plots may be used to reveal stressed regions in the samples, which may result, as in our case, from a large lattice mismatch.

Microscopy observation of the crack formation in the stressed regions around the imprints was also carried out. It showed that severe cracking of the material takes place at large P, particularly when the tip of the indenter is in the vicinity of a phase boundary.

4. Conclusions

Microhardness-depth profiles of multiyear Si/SiC samples have been studied by indentation with the pyramids of Knoop and of Vickers. Stressed regions have been revealed which result from the large lattice mismatch between Si and SiC.

Acknowledgement

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References

1. L.-O. Björketun, L. Hultman, O. Kordina and J.-E. Sundgren. J. Mater. Res. **13** (1998) 2632.
2. N. Goryunova, A. Borshchevskii and D. Tretiakov. In: *Semiconductors and Semimetals: Physics of III-V Compounds*. Vol. 4 (R. Willardson and A. Beer, Eds, Academic Press, New York 1968) p. 3.
3. M. Baleva, E. Mateeva and E. Trifonova. J. Mater. Sci. **34** (1999) 795.