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Detection of Crystalline Phase in Cross-Section Multilayer Thin Film by High-Resolution Transmission Electron Microscopy

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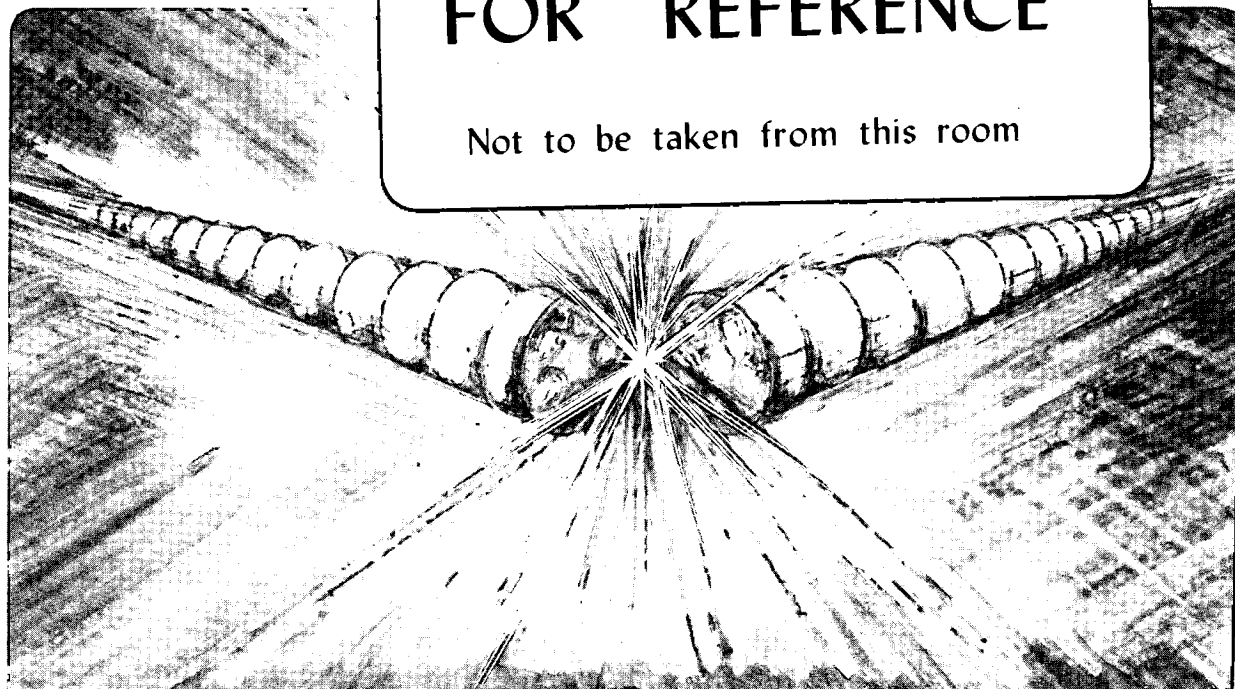
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## DETECTION OF CRYSTALLINE PHASE IN CROSS-SECTION MULTILAYER THIN FILM BY HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY

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High-resolution transmission electron microscopy has proven to be very useful in direct detection of crystalline phases that exist over extremely small volumes, yielding information about structure, orientation, and, under appropriate circumstances, composition.<sup>1</sup> In this paper, we report the detection of a crystalline phase in the tungsten-rich layer of an annealed 7 nm-period tungsten-carbon multilayer produced at the Center for X-Ray Optics at the Lawrence Berkeley Laboratory.

The multilayers were prepared by dc magnetron sputtering at floating temperature.<sup>2</sup> The argon sputter gas pressure was 0.0020 torr. Different techniques were employed to produce cross-section and plan-view samples for TEM. For cross-section samples, 70 bilayers of W and C were sputtered on semiconductor-grade Si (111) wafers. For plan-view samples, the substrates on which the multilayer was grown consisted of 3 mm-diameter 300-mesh copper microscope grids, mounted on glass slide with Crystalbond<sup>®</sup> vacuum adhesive. After a deposition of 4 bilayers of W-C, keeping the same sputtering parameters as those of the Si substrates to guarantee the same layer thicknesses, the glass slide was soaked in acetone to dissolve the Crystalbond<sup>®</sup>, leaving the multilayer spanning the holes of the copper grids. Both the Si-substrate and copper-grid samples were annealed at 500°C for 4 hours under vacuum of 10<sup>-6</sup> torr. The annealed Si-substrate sample was then prepared for cross-section by mechanical grinding, and ion milling in a cold stage at 5kV.<sup>3</sup> The cross-section sample was studied in a JEOL JEM 200CX with ultrahigh resolution goniometer, with the electron beam parallel to the [112] of the Si substrate. The plan-view sample was studied in a Philips 301 operating at 100kV.

Figure 1 shows the overall cross-section configuration of the multilayer, which consists of Si substrate, carbon transition layer, and amorphous carbon layers interleaved with the crystalline layers. The known spacing ( $d = 3.14 \text{ \AA}$ ) of the Si (111) planes in this image was used as a calibration to determine the thicknesses of the individual layers. Because of extensive intermixing between the amorphous carbon and the crystalline layers at their interfaces, it was difficult to determine the exact position of interlayer boundaries. Nevertheless, the amorphous carbon layer was measured to be  $2.68 \pm 0.77 \text{ nm}$ , and crystalline layers  $4.99 \pm 0.77 \text{ nm}$ . The intermixing interfacial layer was estimated to be about 0.77 nm. It is noted that single grains of the crystalline phase can extend for considerable lateral distance.

Figure 2 shows an enlarged region of the multilayer from figure 1. At this magnification, we can see clearly the lattice fringes of the crystalline layers. From its electron diffraction pattern, the crystalline phase was identified to be hexagonal WC, with the (100) planes measured to be  $2.4 \pm 0.1 \text{ \AA}$ . The grain boundaries in the WC crystalline layer (arrows) seem smooth, and there are no abrupt changes across the layer interface at these grain boundaries.

Plan-view bright-field and dark-field images of the same region of the multilayer prepared on copper grids are shown in figure 3 and 4, with the crystalline grains visible against the amorphous carbon background. These images show different crystalline growth morphology occurred during deposition or annealing process. There are grains as large as a few micron.

In this study, high-resolution TEM has clearly demonstrated its capability for detecting and identifying a crystalline phase in multilayer thin films over a 5 nm spatial scale. Further processing is underway to study the crystalline growth in the W-rich layers and the intermixing of the layers at the interface in more detail.<sup>4</sup>

### References

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3. T. D. Nguyen, R. Gronsky, and J.B. Kortright, in *High-Resolution Microscopy of Materials*, edited by W. Krakow, F.A. Ponce, and D.J. Smith (Mater. Res. Soc. Proc. **139**, Boston, MA 1989).
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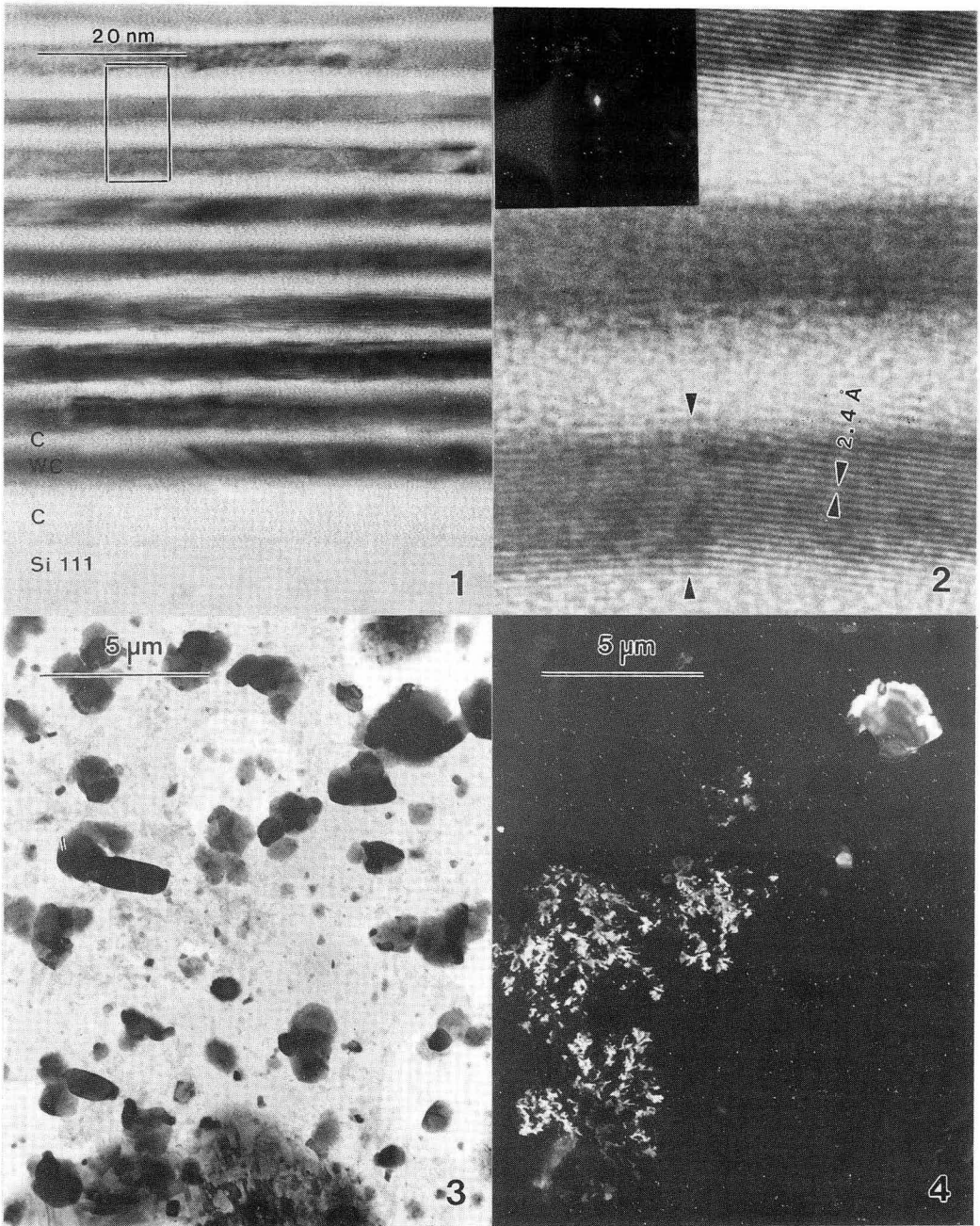


FIG. 1.--Cross section of annealed 7 nm period W-C multilayer structure on Si substrate.  
 FIG. 2.--Enlarged image of region indicated in figure 1.  
 FIG. 3.--Plan-view bright-field image of multilayer prepared on copper grid substrate.  
 FIG. 4.--Dark-field image of figure 3.

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