*Sub-Micron Sectioning Technique for Diffusion Experiments**

There are several experimental techniques [1–5] which have been used successfully to study self-diffusional processes in the solid state. From a theoretical viewpoint, the tracer-sectioning technique involves the fewest mathematical assumptions and permits relative ease of data processing. However, in cases where the self-duffusion coefficient is small and the material being studied is difficult to section, as is the case in transition metal carbides, application of conventional lathe-sectioning techniques is not feasible.

In order to study the slow carbon self-diffusion in dense, polycrystalline tungsten carbide specimens at temperatures in excess of 2000° C it would be desirable to limit the diffusion anneals to less than 12 h, employ the sectioning technique and observe the grain-boundary as well as volume diffusion processes directly. This was made possible by the sub-micron sectioning apparatus shown in fig. 1, suggested by the design of Goldstein [6].

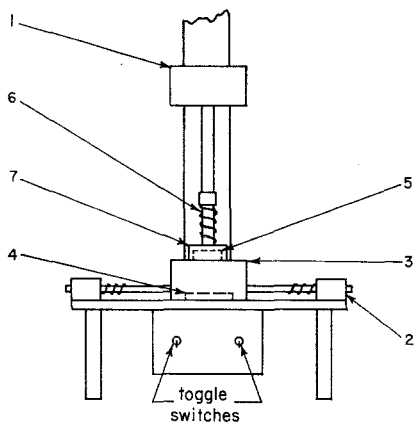

Figure 1 The sub-micron sectioning apparatus. The section, in granular form, was collected in cup (3) and retained for activity and thickness determination.

It was operated as follows: A few drops of lapping oil were placed on the bottom of the cup (3) for lubrication. Then 3 μm diamond dust in paste form was worked onto the top surface of the alumina plate (4), which was put into the cup with the diamond abrasive side up. The sample was secured in the retaining holder (5) by double-stick tape against a rubber backing to prevent sectioning at an angle to the sample face. The sample assembly was then mated to the alumina disc and held firmly against it by the spring (6) and thrust bearing (7). The motor (1) rotated the sample at 1700 rpm while another motor (2) revolved at 2 rpm causing the cup (3) to traverse the stage. This combined action resulted in a slow eccentric motion of the alumina disc (4) within the cup, presenting a new grinding surface continually to the sample. The alumina disc was made by hot-pressing Linde A alumina and then machining the upper and lower surfaces mutually parallel and polishing the upper surface optically flat. The flatness of the alumina plate and sample surface was checked interferometrically after each section removal. After each sectioning the radioactive section was separated by fine particle filtration techniques and the section activity determined by conventional counting techniques.

Operation of the sectioning apparatus for 60 sec. resulted in removal of a WC section of about 0.1 μm thickness, making conventional weighing or micrometer measuring techniques very difficult to apply. Therefore, the section thickness was determined by spectrophotometric analysis of tungsten [7] in the removed section.

Typical results are shown in fig. 2, illustrating

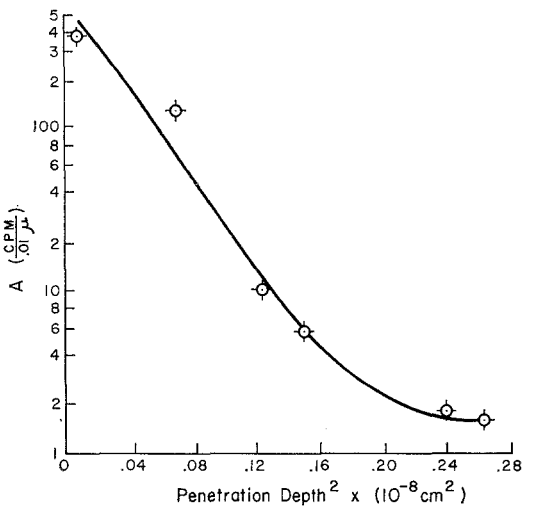

Figure 2 Carbon-14 activity as a function of penetration in the WC sample. Results of diffusion anneal at 2220° K for 8 h. (Activity shown as counts per min, per 0.01 μm sample thickness removed).

the extent of uncertainty in experimental points, as well as the existence of at least two distinct diffusion processes.

Using this method it was possible to separate, graphically, volume and grain-boundary diffus-

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ion phenomena in polycrystalline tungsten carbide [8].

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Growth of Epitaxial Silicon Layers by Ion Beam Sputtering

Sputtering as a method of growing semiconductor epitaxial layers has received very little attention compared with the more widely used methods of chemical transfer and vacuum-evaporation. Silicon layers showing preferred orientation have been grown by triode-sputtering [1], but there is no published account of good quality epitaxial silicon layers grown by any sputtering techniques. Only limited information is available on the growth of epitaxial layers of some of the other semiconducting materials such as Ge [2-6] and GaAs [7]. The twinning and high fault density present in these layers could be due to inherent limitations in the diode- and triode-sputtering techniques used. These limitations are high operating pressures, low deposition rates and limited control of unwanted primary and secondary ions incident on the substrate surface. In the present work, high quality epitaxial silicon layers have been grown by a novel technique of sputtering by an argon ion beam of high current density in a vacuum in the range of 10^{-4} torr and at controlled rates of up to 400 Å per min. The bombardment of the substrate surface by positive ions during deposition, which has been shown to have an adverse effect on the quality of the layers, has been eliminated.

A schematic diagram of the sputtering arrangement is shown in fig. 1. Argon at a pressure of a few tenths of a torr was leaked into the ionisation chamber of an ion-beam source developed from the design of Nelson and Hill [8]. The electrons emitted from the heated tantalum filament ionised the argon atoms which were concentrated along the central axis of the chamber due to the magnetic field of the current through the filament. The extraction electrode at earth potential extracted a fraction of these ions from the ionisation chamber which was held at + 9 kV. A pressure in the range of 10^{-4} torr was maintained in the vacuum chamber by pumping argon at about 180 l/sec with a helium cryopump and by limiting the leakage of argon from the ionisation chamber only through the 2 mm aperture of the ionisation chamber. The ion-source was bakable to over 400° C and was water-cooled during the operation to reduce the contamination from it to a minimum. Contamination from the sputtering of the tantalum filament was minimised by maintaining the filament bias with respect to the ionisation chamber at 20 V, which is well below the threshold voltage for sputtering of tantalum of 26 V [9]. Another source of impurity was found to be due to the sputtering of the edge of the 3 mm aperture of the extraction electrode due to the tendency of the high-current-density ion-beams to diverge. To minimise this divergence, and hence the sputtering of the aperture, optimum values of angles for the electrodes were determined experimentally and were found to be $\phi_1 = 30°$, $\phi_2 = 32°$ and $\phi_3 = 42°$. The emerging ion beam of between 3 and 8 mA bombarded the central region of a silicon target, a slice of silicon 32 mm in diameter and 3 mm thick, at energy of 12 keV and an angle of 60° to the target normal. The silicon sputtered from the target was deposited on a (1 1 1) surface of a single crystal silicon slice 32 mm in diameter and 0.25 mm thick, placed above the target. The slice was heated by focused radiation from outside the vacuum and was held at + 9 kV to prevent it from being bombarded by positive ions. Before deposition, the slice was cleaned by flashing it at 1100° C for 2 min in a vacuum of 10^{-8} torr and the target was