11-Surfaces, Interfaces and Thin Films

PS-11.02.25 HREM STUDIES OF TiN THIN FILMS.
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INTRODUCTION: TiN thin films have been widely used as diffusion barrier between metals and silicon in VLSI as well as coatings for hard metal tools. There has been a great interest in TiN thin films during the past few years, which consequently gives rise to a large scatter in their microstructure. In this paper, the results of TEM analyses of TiN thin films by high resolution electron microscopy (HREM) and selected area electron diffraction (SAED) are reported.

EXPERIMENT: TiN films were prepared by sputtering Ti on chemically cleaned and polished Si(100) wafer, and then annealing at 600 °C and 800 °C in N_2 atmosphere. Through-foil samples were prepared for TEM analyses. TEM was performed on a PHILIPS EM430 transmission electron microscopy, Operated at 300 kV and selected aperture 0.5 μm, with Cs=2 mm and point resolution 2.3 Å.

RESULT AND DISCUSSION: From the morphological view of the films, the resulting TiN films, about 150 Å in thickness, are dense and homogeneous. The sizes of the evenly distributing TiN grains vary considerablely from 100 Å to 250 Å. The interrupted rings patterns of SAED of the films indicate that the TiN grains in the films have some coherent relationship between their crystal orientations.

The TiN[110] HREM images of the films, can tell us the structural features of the films at atomic scale. TiN grains contact closely with narrow grain boundaries, and there is no other phase or amorphous presents in the grain boundaries. Most of the TiN grains are perfect crystals inside the grains. Most grain boundaries are low-angle grain boundaries. According to the compound Mole fringes, and the suggestion that they are formed by two overlapping TiN grains alone the electron beam, we can deduce the orientation difference between these two grains, which has a typical value of 13°. Another kind of titanium nitride phase, though sparse and scattered, is found in the films. HREM images show their structure is different from the cubic TiN, but more close to orthogonal structure and smaller crystal constants. We believe they are orthogonal Ti_2N grains. Both Ti_2N and TiN have similar structure and close crystal constants, so they can contact closely without a third phase.

All the above results show that the resulting TiN thin films have a texture structure in [110] direction. That is, the TiN grains in the films orient about the TiN [110] direction, and distribute irregularly around [110] axis within a small spherical angle. Thus most grain boundaries of the TiN grains in the films are low angle grain boundaries. Consequently form the closely contact TiN grains and narrow grain boundaries structures, and the dense films. This is the very reason why TiN film can effectively bar the metal and semiconductor atoms which mainly diffuse through grain boundary.

REFERENCES: 1. J.Stimmell, "Properties of D.C. Magnetron Reactively Sputtered TiN", unpublished.
2. J.-E. Sundgren, Thin Solid Films 128, 21(1985).

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