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In-situ Observation on Electron Beam Induced Chemical Vapor Deposition by Transmission Electron Microscope

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A novel method for directly applying the desired material on only a very small area of a substrate (selective deposition) at low temperature has been reported by applying an electron beam induced chemical reaction at a gas-substrate interface[1]. Nanometer structures can be fabricated by electron beam Chemical Vapor Deposition (CVD) using electron beam induced surface reaction[2]. Computer controlled direct writing also is possible for electron beam CVD. Metals, semiconductors, and inorganic materials can be deposited by this means. When an electron beam is irradiated onto a substrate in electron beam CVD, source gas molecules adsorbed on a substrate are dissociated into nonvolatile and volatile materials. Nonvolatile materials are deposited on a substrate, while volatile materials are evacuated. The deposited material growth process can be observed in-situ by transmission electron microscope (TEM), if source gas molecules adsorbed on a TEM specimen. This paper presents results obtained for TEM tungsten electron beam induced CVD observation in-situ, using the tungsten hexafluoride gas source.

Figure 1 demonstrates an experimental arrangement. Tungsten hexafluoride gas molecules were introduced into the TEM specimen chamber at 0.1Torr and evacuated soon after. The gas molecules adsorbed on fine silicon particles, about 100nm in size, which were on TEM specimen grid. The gas molecules were excited by the TEM electron beam and dissociated into tungsten metal and fluorine gas. Tungsten metal was deposited on the silicon surface and growth was formed. The growth process was observed in-situ by TEM and was recorded by VTR system. Tungsten clusters, as initial state of the growth, coalesced and grew under electron beam irradiation. Figure 2 shows an electron micrograph of the grown tungsten clusters on fine silicon particles. The grown tungsten clusters (2-3nm in size), irradiated by high energy and high current density electron beam (120kV, 100A/cm^2), were beta-tungsten crystal. Clusters often show beta-tungsten (110) or (100) lattice image, as demonstrated in Fig. 2b. Figure 3 shows an electron micrograph of a tungsten rod on a fine silicon particle. Tungsten rod was made by focused electron beam (3nm in diameter) scanned on the silicon surface manually at about 1×10^{-6}Torr source gas pressure. The rod radius was about 15nm. Bright contrast for the inner rod shows that the center of the rod was probably tungsten silicide. Tungsten silicide growth process at a tungsten-silicon interface can be clarified and nanometer structures can be fabricated, using this technique.

This experiment was made by modified EM-002A equipped with gas introducing system and VTR system in the KURODA Solid Surface Project.

1) S. Matsui and K. Mori, J. Vac. Sci. Technol. B4, 299 (1986).
2) A.N. Broers,W.W. Molzen, J.J. Cuomo, and N.D. Wittle, Appl. Phys. Let. 29, 596 (1976).

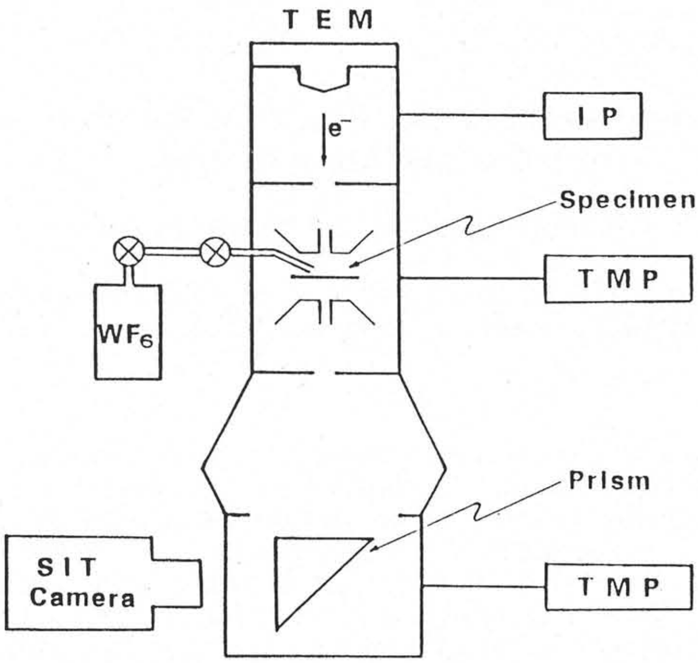

Fig.1 Experimental arrangement diagram. Gas introducing system and VTR system are equipped.

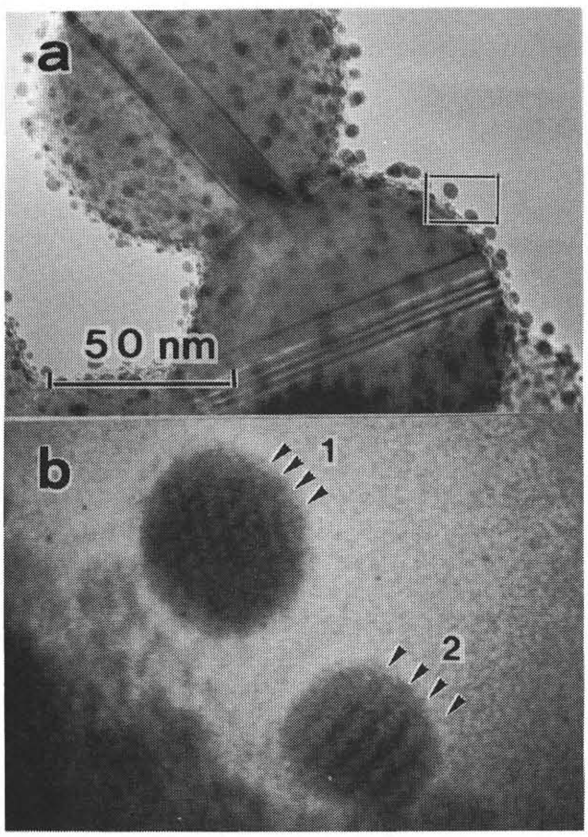

Fig.2 Electron micrograph of Tungsten clusters deposited on fine silicon particles. Arrowhead 1 shows beta-tungsten (110) lattice plane. Arrowhead 2 shows beta-tungsten (100) lattice plane.

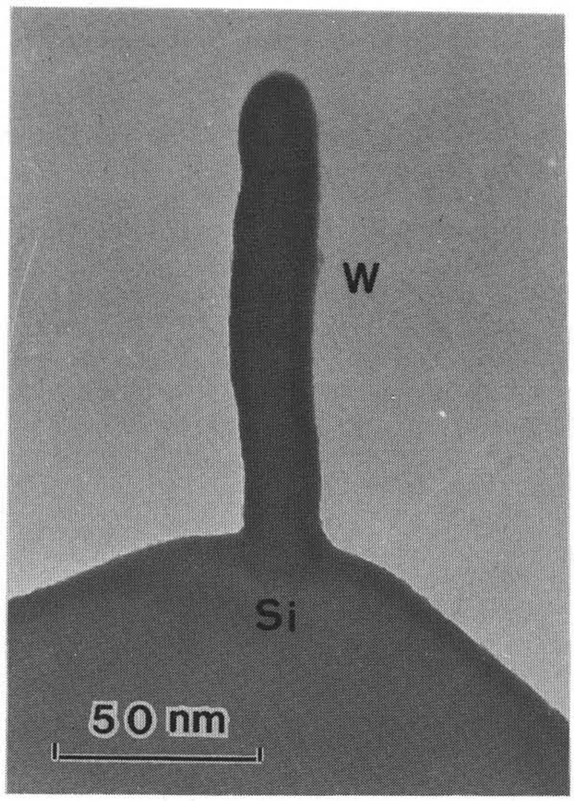

Fig.3 Electron micrograph of a tungsten rod. The rod is 15nm thick. The center of the rod (bright contrast) is probably tungsten silicide.