Transmission Electron Microscopy (TEM) Specimen Preparation Technique using Focused Ion Beam (FIB):
Application to Material Characterization of Chemical Vapor Deposition of Tungsten (W) and Tungsten Silicides (WSi_x)

Yih-Yuh Doong*, Jui-Mei Fu, Yen-Chu Huang

*Worldwide Semiconductor Manufacturing Crop. , Hsinchu, Taiwan
United Microelectronics Corp., Hsinchu, Taiwan

Abstract

The specimen preparation technique using focused ion beam (FIB) to generate cross-sectional transmission electron microscopy (XTEM) samples of chemical vapor deposition (CVD) of Tungsten-plug (W-plug) and Tungsten Silicides (WSi_x) was studied. Using the combination method including two axes tilting[1], gas enhanced focused ion beam milling[2] and sacrificial metal coating on both sides of electron transmission membrane[3], it was possible to prepare a sample with minimal thickness (less than 1000 Å) to get high spatial resolution in TEM observation. Based on this novel thinning technique, some applications such as XTEM observation of W-plug with different aspect ratio (1 ~ 6), and the grain structure of CVD W-plug and CVD WSi_x were done. Also the problems and artifacts of XTEM sample preparation of high Z-factor material such as CVD W-plug and CVD WSi_x were given and the ways to avoid or minimize them were suggested.

Introduction

Cross-sectional transmission electron microscopy (XTEM) specimen preparation using focused ion beam (FIB) milling have been proven as the most promising technique to characterize the specific site and small geometry structure which of critical dimension is close to XTEM specimen thickness (equal or below 1000 Å). With the aid of computer-controlled stage, navigation software, and its in-situ high resolution imaging capability, FIB system can drive the specimen to the specific region with the accuracy of um range and mill micron even sub-micron scale features. The application of FIB assisted XTEM specimen preparation provides a promising way to use TEM as a routine microanalysis tool. However, FIB assisted XTEM specimen preparation still encountered some problems that need to be address: (1) to prepare XTEM specimen of the ultimate thin and uniform thickness. (2) to prepare XTEM specimen containing the material with different etching or sputtering rate, such as refractory metal and their silicides, W-plug and etc.

In the application of focused ion beam micro-machining (FIBM), ion beam induced chemical etching (IBICE) has been widely used to increase etching rate, reduce redeposition of sputtered particle and decrease the cycle time of specimen production. Using halogen-based gas, such as I_2, Cl_2, and XeF_2, the etching rate is enhanced up to 10 times depending on the materials and gas species. For examples, the etching rates of Si, SiO_2, and Al with gas assisted etching are 2.6, 0.4 and 0.6, respectively [4]. Therefore, the streaking or masked effect [5] could be enhanced using IBICE when XTEM specimen preparation.

In this article, we demonstrate a novel scheme of FIB assisted XTEM specimen preparation to get the high quality XTEM micrograph. This scheme combine and optimized the different specimen preparation techniques to get a acceptable cycle time of sample preparation and minimize artifacts.

Experiment

The specimens are chemical vapor deposition of Tungsten (W) plug and Tungsten Silicides (WSi_x) which are typical material used as via interconnects and gate of ULSI. The technique includes two major steps: slice preparation and FIB process. The slice preparation followed the specific XTEM specimen preparation developed by Morris, S. et al [6]. The specific sites which could be electrical failure or key process structure are marked by laser cutter or FIB, were cleaved, lapped down to 20 ~ 50 μm thickness, and mounted to a modified copper grid prior to XTEM specimen preparation using FIB process. The differences between Morris’s and this technique are the shape and position of modified copper grid which prevent the gas nozzle hitting the TEM sample (Figure 1, 2). Before FIB fabrication, the TEM sample was coated with metal film of several hundred Å thick as a conductive path preventing charging effect and thermal damage.

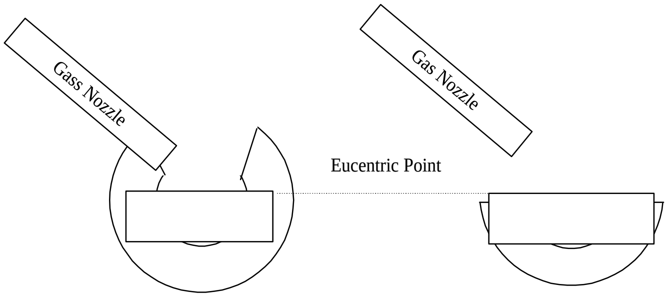

Figure 1: The left is used by Morris, S. et al. and the right is used in this work. The right can prevent the gas nozzle hitting the sample but the left.

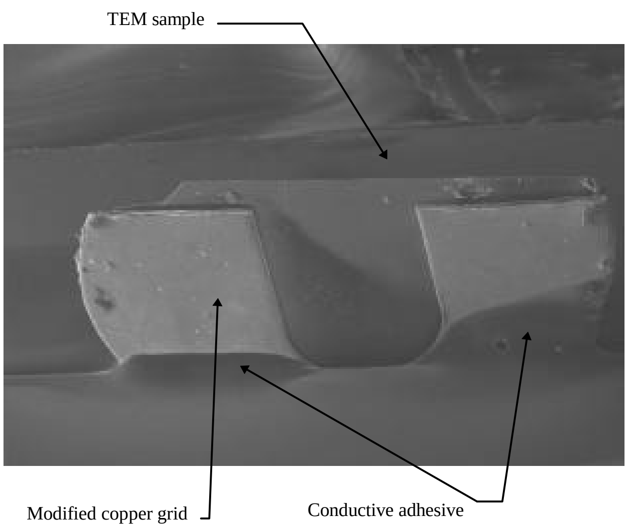

Figure 2: SEM micrograph of TEM sample mounted on the modified copper grid.

The FIB fabrication procedure of this work is illustrated in Figure 4. Two trenches were milled on the both sides of the XTEM specimen by FIB with Halogen gas assisted [4]. Two kinds of Halogen gas are available for this work: XeF_2 and I_2. Due to the high etching rate of XeF_2, XeF_2 is not suitable for longer time etching. FIB milling using I_2 gas with larger current mode (870 pA) was performed until the thickness of XTEM specimen reached 1μm. After initial trenches milled, the successive steps with progressively reducing the ion beam current (300 pA, 150 pA, and 60 pA, respectively) were enacted to mill membrane down to 200 ~ 300 nm thick. To remove the skirt of the tapered membrane, the incident angle of the ion beam was set 5^o ~ 3^o off from the vertical to the specimen surface. To get the ultra fine membrane, the membrane was coated with sacrificial Pt film on the both sides by FIB induced Pt deposition at 60 ~ 10 pA current mode. The thin Pt film can compensate the thickness variation between inter-metal/inter-layer dielectric (IMD/ILD) and W-plug / WSi_x because the sputtering rate of Pt is more close to Tungsten (W-plug) / Tungsten silicide (WSi_x). Therefore, the uniform thickness membrane with W-plug / WSi_x gate structure could be obtained.

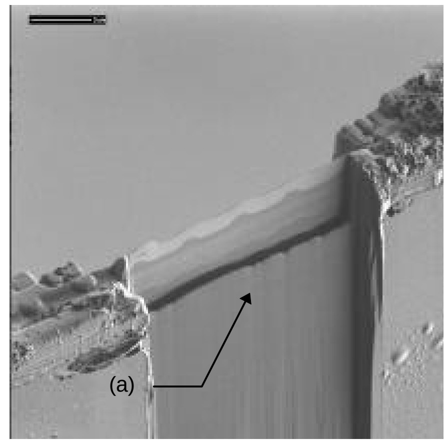

Figure 3: Scanning ion micrograph of XTEM membrane fabricated by FIB. (a) With the aid of Halogen gas assisted etching, the streaking / masked effect induced by IBICE can be obviously observed.

Two types of STEM specimens were prepared in this study. One was W-plug structure for the XTEM observation of glue layer and grain; the other was fabricated to observed the W-silicide gate structure. XTEM observation was performed using JEOL TEM 2010 with accelerating voltage 200 KeV.

Results

(1) Ion beam shift induced by charging effect

The factors of beam shift and drifting during the FIB milling were mechanical stage relaxation, ion beam instability and electrically isolated conductive material which is structure dependent. In this work, coating a conductive film (Pt film was used in this work) conquered charging effect causing ion beam shift on both sides of membrane. The sacrificial Pt film can be good thermal conductive film as well as conductive film minimizing the thermal damage and charging effect during FIB fabrication [3]. Figure 5 is a scanning ion micrograph of XTEM membrane before sacrificial Pt film coating. Due to the charging effect, the membrane of W-plug / W-silicide gate were charged up and deflected ion beam, which resulted of the thickness variation between the electrically isolated metal and oxide film. Figure 6 shows a scanning ion micrograph of XTEM membrane after sacrificial Pt film coating. Based on passive voltage contrast technique, Figure 6 shows that the membrane of electrically isolated W-plug / W-silicide gate was ground.

2) Streaking / masked effect of FIB fabrication

The streaking / masked effect of XTEM specimen preparation using FIB is a commonly seen artifact. Especially, the material of the different sputtering / etching rate, surface topography variation, and IBICE will enhance this phenomenon. To compensate the sputtering / etching rate difference of W-plug / W-silicide gate, a sacrificial Pt film was adopted in this work. A. Yamaguchi [2] reported that conventional FIB milling without gas assisted etching will induce InP amorphous layer of 31 nm thick and 2~5 nm by IBICE, respectively. Therefore, low current mode (60~10pA) was used to deposit sacrificial Pt film when thickness of XTEM membrane was 200 ~ 300 nm thick and I_2 was used as the gas source for the reason of lower etching rate of Si / SiO_x. Figure 7 is a bright-field XTEM micrograph of high aspect ratio W-plug fabricated by FIB without IBICE. The streaking / masked effect on FIB trimmed surface can be obviously observed at the bottom of W-plug. Figure 8 shows a bright-field XTEM micrograph of high aspect ratio W-plug fabricated by FIB with ICIBE. The thickness of W-plug bottom (Si-substrate) was almost uniform. The keyhole was not observed at this micrograph because they were filled up during sacrificial Pt coating.

3) Hole formation resulted from two axes tilting

To remove wedge profile and achieve the ultra thin membrane, two axes tilting technique was commonly used. In this work, the incident angle of the ion beam was set 5^o ~ 3^o off from the vertical to the specimen surface at the different steps of FIB process. At the final step, the offset angle was set to 0^0, specific site milling was performed at the location of W-plug / W-silicide gate, and a lower current mode 10 pA with ICIBE was used. Figure 9 is a XTEM micrograph of CVD W-plug. The hole under Al line was formed during two axes tilting FIB milling and the different thickness of membrane was resulted from specific site FIB milling. Figure 10 shows the complete W-plug structure with visible grain.

Examples

The following figures are images obtained from two type Tungsten specimen prepared by this novel FIB process then analyzed with XTEM. Figure 11 is a XTEM micrograph of high aspect ratio W-plug. Figure 12 shows XTEM micrograph of W-silicide gate of flash memory cell. Both them were fabricated under the optimized condition of FIB machine, operation and the different techniques described previously. The images shows less radiation damage and uniform thickness without hole formation at the bottom of membrane.

Conclusion

By trading off the side effects induced by the different kinds of XTEM specimen preparation using FIB, we proposed this novel technique to get a membrane with less artifact and thickness of ultimate thinning and uniformity. This novel technique provides a timely, reliable, and high yield method to enact XTEM analysis as an ULSI process characterization tool. Based on this process flow, specific site TEM sample preparation can be routinely done and more analyst-independent. The total analysis can be done under working day and even within5 hours including lapping, mechanical polishing (1 ~ 1.5 hrs) and FIB fabrication (3 ~ 4 hrs).

Acknowledgments

The authors would like to show thanks to Dr. Wang, Yuh-lin, Dr. Hsieh, Yong-Fen, Mr. Peng, Yuan-ching and staffs of material analysis laboratory at UMC for their valuable discussions and efforts to this work.

Reference:

1. C. W. Snyder, M.R. Frei, D. Bahnck, L. Hopkins, R. Hull, L. Harriott, T.Y. Chiu, T. Fullowan, and B. Tseng, “Combining transmission electron microscopy with focused ion beam sputtering for microstructural investigations of AlGaAs/Ga/As heterojunction bipolar transistors” , JVST B. 13(4), Jul/Aug 1995, p1514-1518
2. Akira Yamaguchi and Masahiro Shibata, ¡ §Transmission electron microscopy specimen preparation technique using focused ion beam fabrication: application to GaAs metal-semiconductor field effect transistors ¡ ¨, JVST B. 11(6), Nov/Dec 1993, p2016-2020
3. Y. Y. Doong, ¡ §Method for preventing charging effect and thermal damage in charged-particle microscopy ¡ ¨, pending patent #:306033, Taiwan, ROC .
4. R.J. Young, J.R.A. Cleaver, and H. Ahmed, “Characteristics of gas-assisted focused ion beam etching”, JVST B. 11(2), Mar/Apr 1993, p234-241
5. A. J. Leslie, K. L. Pey, K. S. Sim, M. T. F. Beh, ¡ §TEM sample preparation using FIB: practical problems and artifacts¡ ¨, ISTFA`95, p353-362
6. S. Morris, S. Tatti, E. Black, N. Dickson, H. Mendez, B. Schwiesow, and R. Pyle, ISTFA`91, p.417-427 (1991).

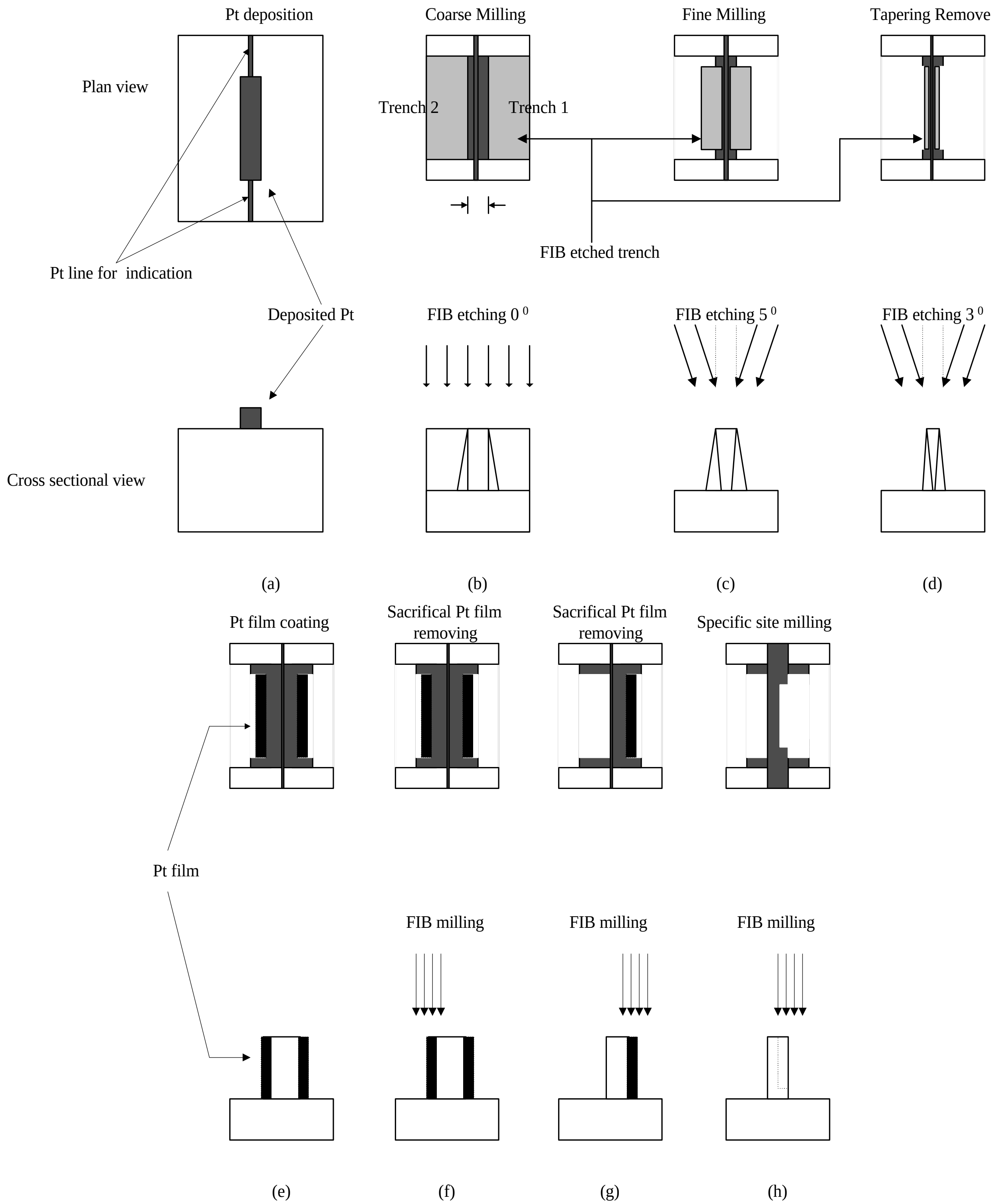

Figure 4: FIB fabrication procedure. (a) Pt deposition and marking. (b) Coarse milling: Ion beam vertical milling trench with I_2 gas until thickness reaches 1 λm. (c) Fine milling: Ion beam set 5^0 off from vertical without I_2 gas. (d) Tapering remove: Ion beam set 3^0 off from vertical without I_2 gas. (e) Both-side sacrificial Pt film coating: Current less than 60 pA. (f) First-side sacrificial Pt film etching. (g) Second-side sacrificial Pt film etching. (h) One-side specific site Etching: 10 pA current With I_2 gas.

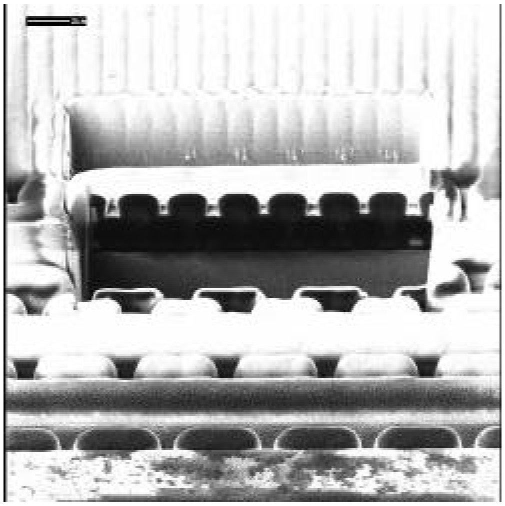

Figure 5: FIB cross-sectional micrograph of selective area before sacrificial metal film coating.

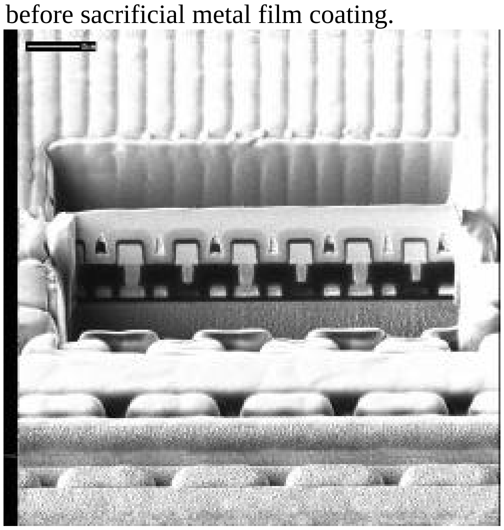

Figure 6: FIB cross-sectional micrograph of selective area after sacrificial metal film coating. The charging effect was minimized and the fine milling could be performed without suffering charging effect induced beam shifting.

Figure 7: XTEM micrograph of CVD W-plug with high aspect ratio (6:1). The streaking or masked effect was obviously observed.

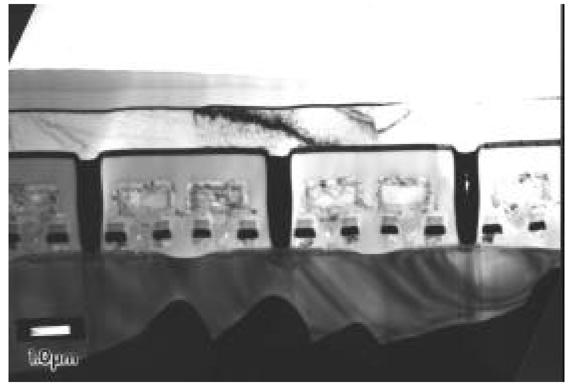

Figure 8: XTEM micrograph of CVD W-plug with high aspect ratio (6:1). The streaking or masked effect was almost eliminated.

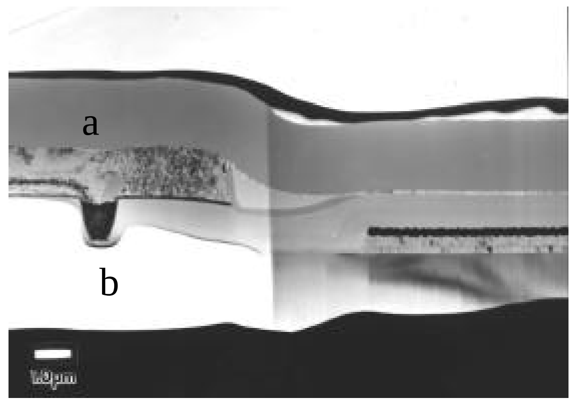

Figure 9: XTEM micrograph of CVD W-plug. By using two axes tilting and selective area etching, hole due to melting of materials under the Al line is clearly visible.

(a) Selective etching area of the fine FIB milling.
(b) Hole induced from two axes tilting milling.

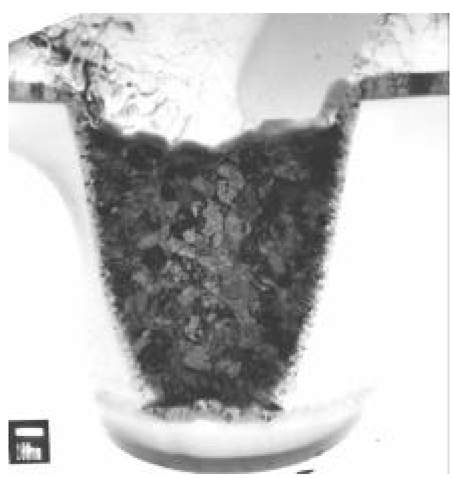

Figure 10: XTEM micrograph of CVD W-plug. The complete plug structure was preserved and grain was observable.

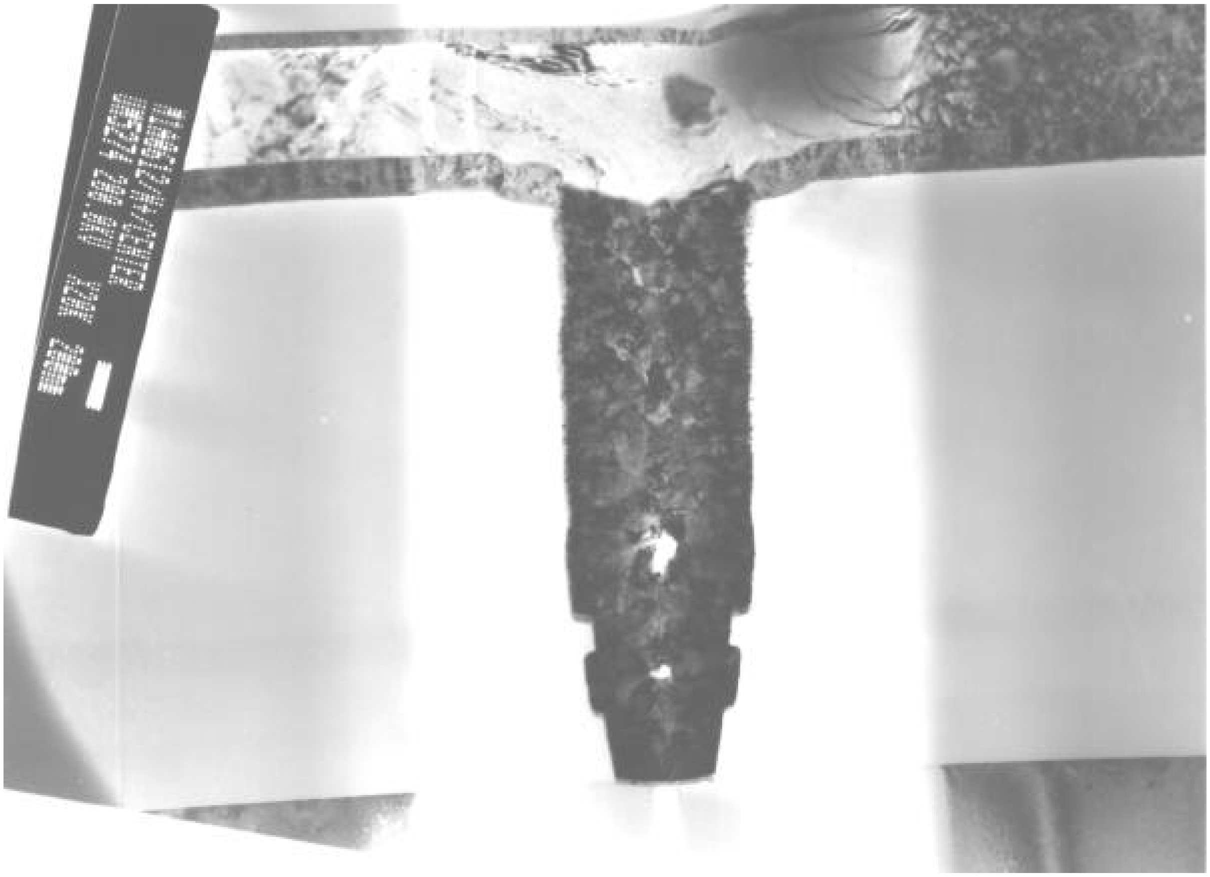

Figure 11: XTEM micrograph of high aspect ratio W-plug.

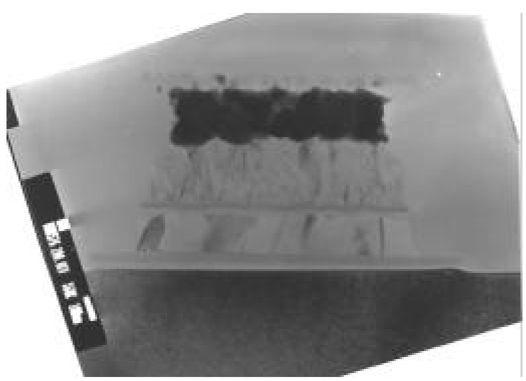

Figure 12: XTEM micrograph of gate of flash memory. WSi_x grain is visible.