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Physical and chemical analysis of advanced interconnections using energy filtering transmission electron microscopy

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Abstract

Our newest method for interconnection analysis using focused ion beam (FIB) specimen thinning and energy filtering transmission electron microscopy (EFTEM) is presented. It is shown that using the site-specific capability and the controlled thinning effect of the FIB in addition with the high spatial resolution of the EFTEM technique, fast chemical analysis of materials with nanometre spatial resolution can be obtained. This is the only method for the observation of very thin diffusion barriers and interfaces in the presence of drastic topography. Application examples are given concerning firstly, the in-depth analysis of tungsten aluminum technology, barrier integrity and interdiffusion of elements near interfaces and secondly, the surface contamination of copper in copper interconnection technology with high aspect ratio contacts. In this case, photoresist spin-coating is carried out prior to FIB thinning. This method is an alternative to surface analysis techniques and offers the best spatial resolution without topography limitations.

Keywords: Energy filtering transmission electron microscopy; Chemical analysis; Focused ion beam; Microelectronics; Metal interconnections

1. Introduction

In new interconnection process technology, very thin diffusion barriers are deposited in high aspect ratio contact holes fabricated using reactive ion etching (RIE). Physical and chemical characterization of diffusion or contamination is increasingly challenging due to topography. The transmission electron microscopy (TEM) analysis technique is more extensively used to overcome this difficulty in association with the focused ion beam (FIB) specimen preparation technique [1]. Recently, we have shown the advantages of adding electron energy loss spectroscopy (EELS) to FIB–TEM analysis for chemical characterization [2]. In this paper, we present the newest technique to overcome this physical and chemical analysis challenge using FIB sample preparation allowing localized site specific thinning with thickness in the range of 100 nm and energy filtering transmission electron microscopy (EFTEM) observations. The EFTEM technique allows high-resolution compositional mapping using

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imaging of core level ionization edges [3]. We show applications of FIB–EFTEM for bulk analysis of tungsten–aluminum technology interconnection, interface or diffusion barriers observation and surface contamination observation of contact etching in copper technology.

2. Experimental details

The specimens were thinned using the FIB technique as already described [2]. The FIB system is a MICRION model 9500 EX using a gallium ion beam of 50 keV maximum energy with a 5-nm minimum spot diameter. In the particular case of the contamination control in copper interconnection technology, a special technique was used to allow FIB cross section of contacts just after oxide etching (i.e. before barrier deposition and metal filling). A conventional I-line photoresist solution was spin-coated over the contacts and let to dry at room temperature in order to imbed and protect, the surface which is intended to be analyzed in the TEM, from the ion gallium irradiation.

The thinned samples were observed at 200 keV electron energy using a TEM PHILIPS CM200 FEG equipped with an electron energy filter (Gatan Imaging Filter GIF200). The energy filtering equipment uses a magnetic prism and a set of electromagnetic lenses [3] allowing energy filtration of transmitted electrons (tunable energy loss and energy window). These electrons of particular energy are used to form, on a CCD camera, a magnified image of the thinned specimen with a resolution better than 1 nm.

3. Results and discussion

3.1. Tungsten aluminum interconnection technology analysis

The first series of FIB–EFTEM application examples concerns bulk analysis of tungsten, titanium nitride diffusion barrier and aluminum interconnection technology. The purpose of Fig. 1 is to illustrate the improvement due to EFTEM in the high-resolution physical imaging of materials.

Fig. 1 shows TEM bright field images of a contact thinned using FIB. This contact has intentionally been shifted with respect to the lower aluminum line in order to study the consequences of photolithography misalignment. Fig. 1a presents an unfiltered TEM image and Fig. 1b shows the same contact imaged using filtration on the zero loss energy. Fig. 1a corresponds to what is currently obtained in the classical TEM technique. All electrons are collected and, due to the chromatic aberration of TEM lenses, those who have loss energy are coarsely focused on the image. This effect induces a diffuse background, which affects contrast and resolution. On the contrary, in the image of Fig. 1b, only the electrons which have not lost energy are collected and the effect of chromatic aberration is suppressed. Small details, which are not visible in Fig. 1a, can now be observed in Fig. 1b. Particularly the TiN anti-reflecting coating (ARC) on top of the aluminum can be seen as composed of three layers. The two lower layers are Ti and TiN sputter deposited; the upper layer is probably formed during the intermetal dielectric deposition.

Another important purpose of energy filtering in the TEM is to allow elemental compositional maps with high spatial resolution. This is obtained using core level ionization edges imaging combined with electron background subtraction (i.e. three window method) [4]. In the resulting compositional image,

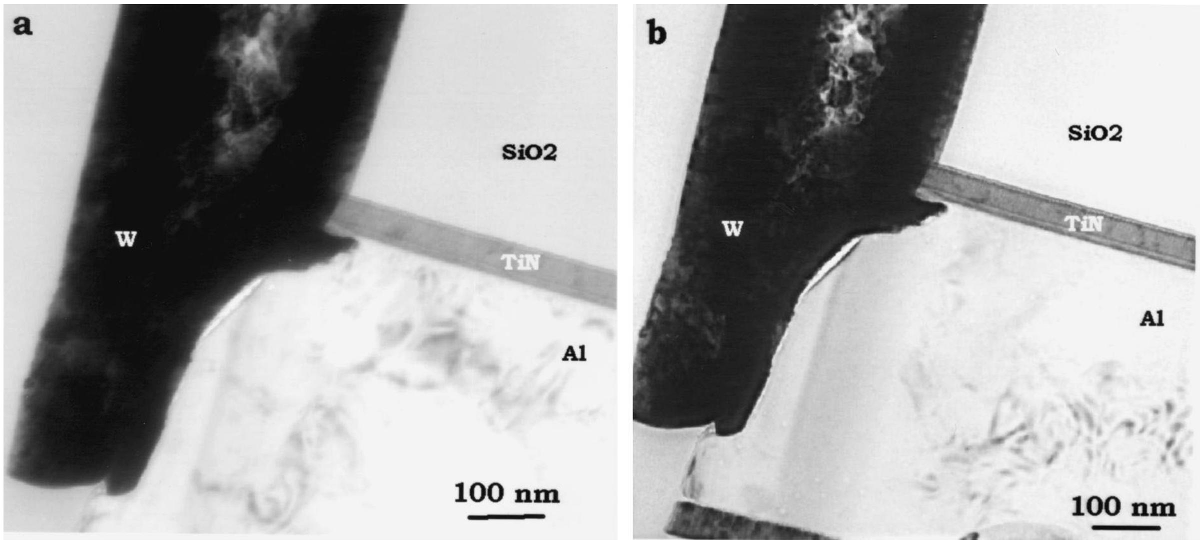

Fig. 1. TEM images of a tungsten contact presenting a negative overlap with respect to the lower aluminum line. (a) Unfiltered classical TEM bright field image. (b) Zero loss energy EFTEM image.

the intensity roughly increases with atomic concentration: dark contrast for zero concentration and bright contrast for higher concentrations.

Fig. 2a–c present respectively: the zero loss image, the compositional map of titanium (Ti_L ionization edge) and the compositional map of fluorine (F_K ionization edge). These images were obtained on a contact such as that shown in Fig. 1. Despite the improved contrast and quality of the zero loss image, the gray level intensity is not directly related to the local chemical composition of the materials. Faintly contrasted interfaces may not be visible at low magnification such as in the ARC

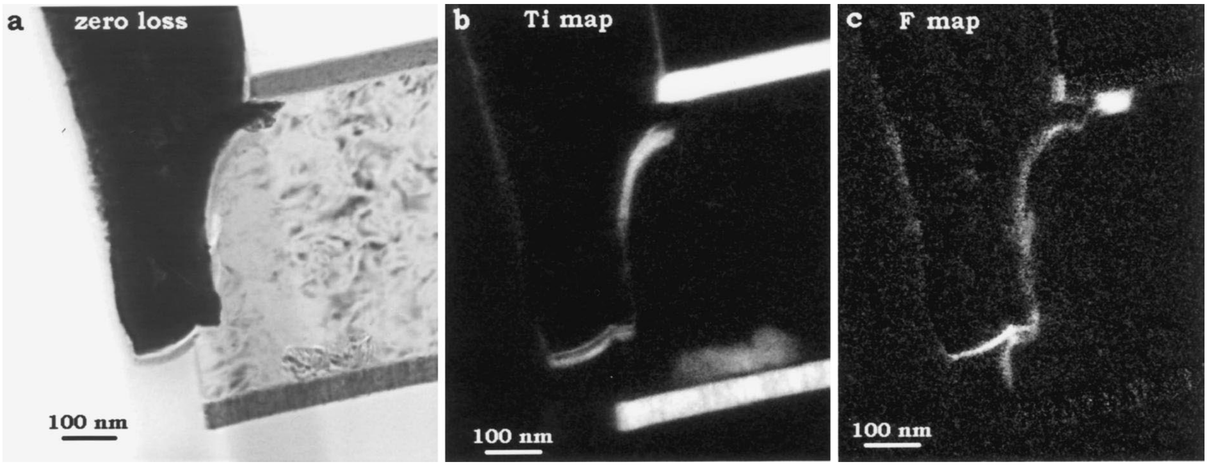

Fig. 2. EFTEM images of the same contact type as that presented in Fig. 1. (a) Zero loss energy EFTEM image. (b) Compositional map of titanium (Ti_L ionization edge). (c) Compositional map of fluorine (F_K ionization edge).

layer as seen in Fig. 1. Also, the diffusion barrier, which is critical for the contact quality, could not be visualized on the image of Fig. 2a. On the contrary, the titanium map of Fig. 2b allows a clear visualization of the TiN barrier. This barrier is not continuous below the tungsten plug. The fluorine map of Fig. 2c shows fluorine accumulation on the bottom of the plug and below the anti-reflecting coating.

Different gray level compositional maps can be combined in a color map. Three images can be easily mixed, each one representing one basic color component of the color map (i.e. green, red and blue). Fig. 3a shows a color map of the plug with oxygen in green, titanium in red, fluorine in blue, and Fig. 3b a color map of the same plug with nitrogen in green, titanium in red, oxygen in blue. Both images are obtained using color mixing of images similar to the one presented in Fig. 2b and c. Some complex layers are better interpreted than using only the separate images of Fig. 2b or c. In particular, at the left bottom of the contact, a three level layer composed of titanium nitride, a titanium fluorine rich compound and titanium is evidenced. In Fig. 3b, the TiN above and below the aluminum line appears in yellow, which is the addition of green and red. As a consequence of this analysis, the physical observation of the three layers of the TiN anti-reflecting coating, shown with high resolution in Fig. 1b, is confirmed and improved by the chemical analysis of each layer. In Fig. 3a, evidence is shown that the fluorine (in blue) is present where the diffusion barrier is discontinuous.

The main advantage of the color compositional maps is that all materials and atomic elements are present in the same image and their localization with respect to each other is more evident.

3.2. Copper interconnection technology analysis

In this part we show that this volume analysis technique can be used as a surface analysis tool in the case of drastic surface morphology. Using a light material, as a protective layer, the surface is embedded and the application of this technique is illustrated on copper interconnections.

Fig. 4 shows EFTEM pictures of a contact hole opened in SiO_2 using a selective SiO_2–SiN plasma etching process. The etching process has stopped in the SiN layer above the copper line. In the zero

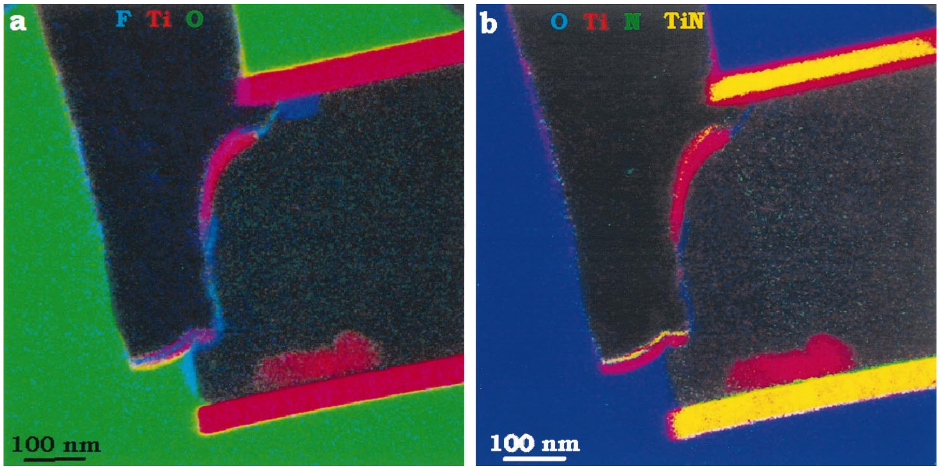

Fig. 3. EFTEM compositional color maps of the same contact as that presented in Fig. 2. (a) Fluorine (blue), titanium (red) and oxygen (green). (b) Oxygen (blue), titanium (red), nitrogen (green) and TiN appears yellow (green plus red).

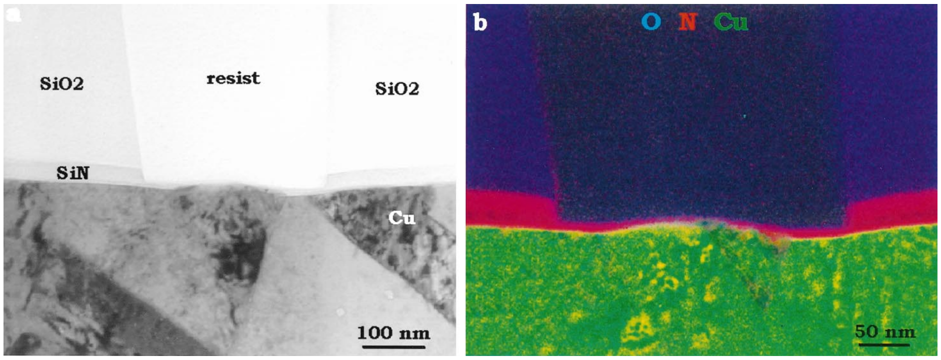

Fig. 4. EFTEM images of a contact hole opened in SiO_2 using a selective SiO_2–SiN plasma etching process. (a) Zero loss energy EFTEM image. (b) Compositional color map of oxygen (blue), nitrogen (red) and copper (green).

loss image presented in Fig. 4a no surface contamination is evidenced either on the bottom or on the contact sides. The compositional color map presented in Fig. 4b (nitrogen in red, oxygen in blue and copper in green) confirms this result, except for some traces of nitrogen on the bottom left of the contact hole.

Fig. 5a shows a zero loss EFTEM picture of another contact after SiN removal using plasma etching. Dark residues are now observed at the bottom and on the edges of the contact hole. These residues are composed mainly of copper as shown on the EFTEM compositional color map of Fig. 5b in which copper is in green color, oxygen in blue and nitrogen of the SiN layer in red.

Fig. 6 presents EFTEM pictures of a similar contact than in Fig. 5 but after wet cleaning using hydrofluoric acid. Fig. 6a presents the zero loss image showing the removal of the residues observed in Fig. 5a. The copper removal is confirmed on the color compositional map of Fig. 6b.

The analysis presented here is typically an experimental problem which could be solved using surface analysis techniques: Auger, electron spectroscopy for chemical analysis (ESCA) or secondary

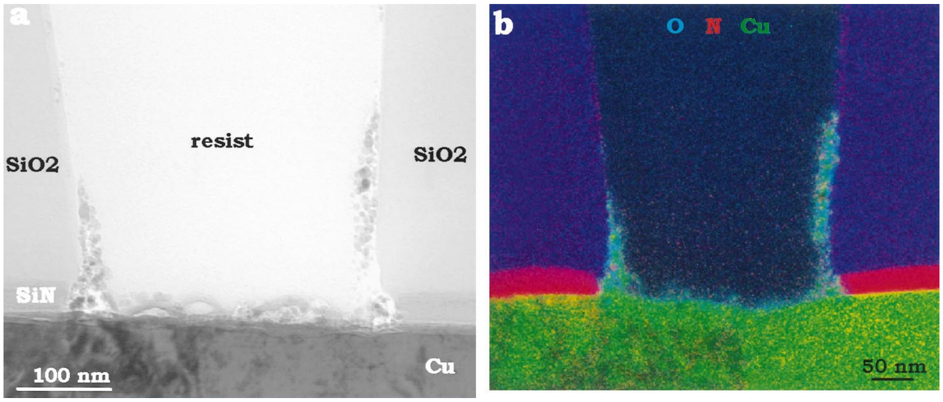

Fig. 5. EFTEM pictures of the same contact hole as that shown in Fig. 4, but after SiN removal using plasma etching. (a) Zero loss energy EFTEM image. (b) Compositional color map of oxygen (blue), nitrogen (red) and copper (green).

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