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Oxygen ion beam-induced abnormal surface topographic development at Ta/Si interface

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We report on an abnormal surface topographic development at a Ta/Si interface, which is believed to be one of the major sources of the irregular interface artifacts in secondary ion mass spectrometry depth profiling by oxygen ion beam. Round crater type topographic development was observed at the interface by sputtering with a 7 keV O_2^+ ion beam and was found to have been formed by blister formation and gradual opening of its center. We suggest that the driving force for this abnormal topographic development is a buildup of compressive stress caused by a volume increase of the Ta layer near the interface, due to oxidation. *© 1996 American Institute of Physics.* [S0003-6951(96)00443-3]

In sputter depth profiling analysis, the ion beam bombardment generally modifies surface composition and structure, which in turn affects the depth resolution. In secondary ion mass spectrometry (SIMS) depth profiling, where an oxygen ion beam is used to increase positive secondary ion yields, the severe matrix effect often makes the profiles quite different from the original ones. The effects are greatest at interfaces where atomic mixing and the consequent SIMS matrix effect are inevitable. Surface topographic development during sputtering is known to be one of the most important factors which contribute to depth resolution in sputter depth profiling.[1–3] Though the subject has attracted much attention, further investigation seems necessary for a better understanding of the complicated roughening processes.[4–9]

In this letter, we report on an abnormal topographic development at a Ta/Si interface in SIMS depth profiling by oxygen ion beams. This has been correlated with the irregular interface artifacts observed in the profiles. Large craters, 400 nm diameter, develop at the interface during the profiling process. From systematic investigations using various surface analysis techniques [including x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), scanning electron microscopy (SEM), and atomic force microscopy (AFM)], we suggest that the driving force for this abnormal topographic development is the buildup of compressive stress[5,6] produced as a result of a volume increase in the Ta layer near the interface, due to oxidation.

The depth profiles are analyzed by a multitechnique XPS/AES/SIMS surface analysis system. The SIMS unit is based on a quadrupole mass spectrometer and a duoplasmatron ion gun. The incident angle of the ion beams can be controlled without affecting the ion energy. The change in the chemical state of surface atoms, due to oxygen ion bombardment, is studied by *in situ* XPS measurements. The base pressure of the system is below 2×10^{-10} Torr. Ta thin film is grown on a Si(100) wafer by sputter deposition of Ta target (99.99% stated purity) by 1 keV Ar^+ ion beams.

SIMS depth profiles of 100 nm thin Ta film on Si using a 7 keV O_2^+ ion beam are shown in Fig. 1 as a function of various incident angles. Abnormal variations in Ta^+ and Si^+ secondary ion intensities near the Ta/Si interface are observed for all the SIMS depth profiles. The Si^+ secondary ion signal increases abruptly for all incident angles before reaching the interface, and fluctuates quite differently at each incident angle. The location of the interface was assumed to be at the final Si intensity increase. For a normal incident angle, the Si intensity shows only a small fluctuation around

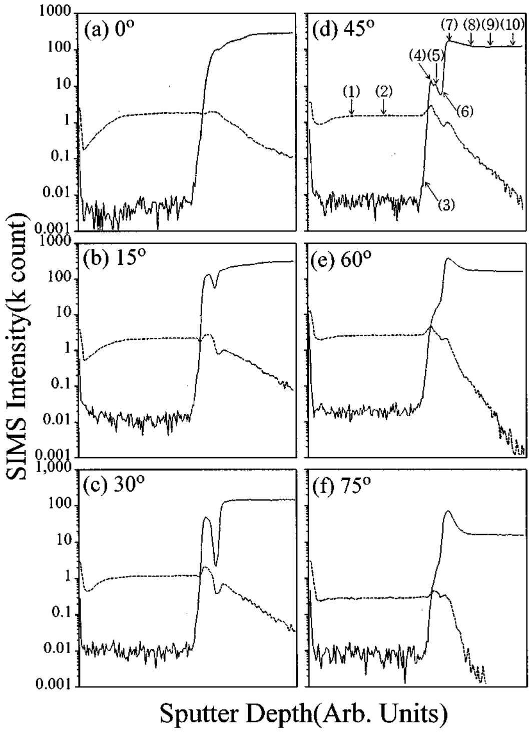

FIG. 1. SIMS depth profiles of a Ta thin film on Si using a 7 keV O_2^+ ion beam for various incident angles (solid line: Si^+, broken line: Ta^+).

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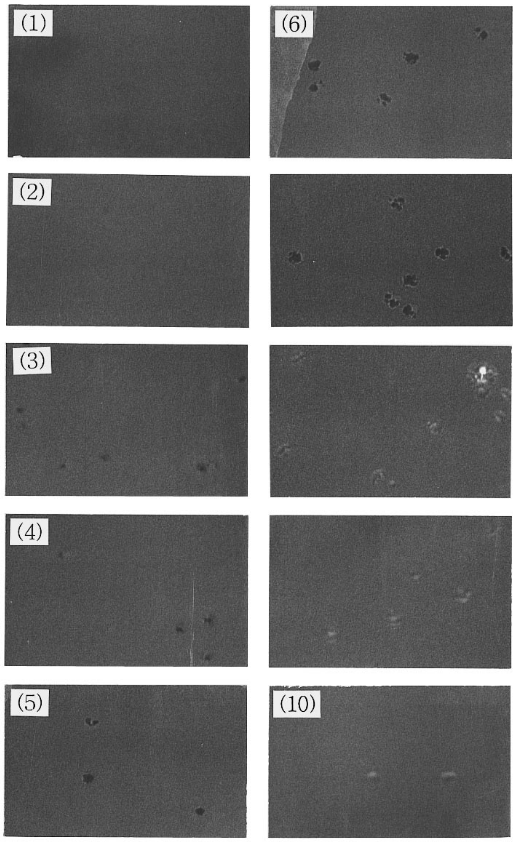

FIG. 2. SEM images at 10 crater bottoms formed by sputtering using a 7 keV O_2^+ ion beam of 45° incident angle as indicated in Fig. 1(d).

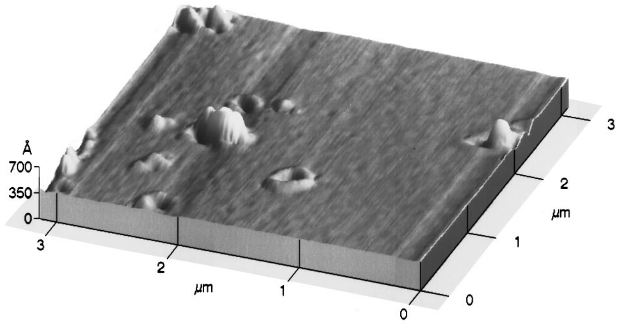

FIG. 3. Three-dimensional AFM image of craters and blisters formed at the Ta/Si interface by sputtering using a 7 keV O_2^+ ion beam for 45° incidence angle (step 6 in Fig. 2).

the interface. The intensity increases abruptly and then decreases before reaching the interface and then increases again at the interface region for a 15°–45° incident angle. For glancing incident angles of 60°–75°, the Si signal increases and then levels off, but then increases again abruptly near the interface. The initial increase of the Si^+ signal arises at a shallower depth from the interface as the angle of incidence increases. These interface artifacts are quite different from the simple form[10] expected by atomic mixing and the subsequent matrix effect near the interface. We have also observed similar features from SIMS depth profiling of very thin 20 nm Ta film on Si using identical analysis conditions. These interface artifacts are not observed using argon ion beam sputtering for either the 100 nm or 20 nm Ta thin film on Si. We therefore propose that the interface artifact is caused by interactions between the oxygen ion beam and the interface.

In order to study topography development as a function of the sputter depth and its relevance to the SIMS interface artifact, we have made ten craters each with a different sputter depth, and have taken SEM images for each crater. Topographic development was observed at the interface for all the incident angles. An incident angle of 45° was chosen to study the topographic development process in detail, because at this angle the topography had the greatest clarity. Figure 2 shows SEM images of crater bottoms corresponding to the different sputter depths as indicated in Fig. 1(d). At stages 1 and 2, topographic development has not occurred, but at the initial increase stage 3, it starts to appear as very small dark spots. These spots occur due to abrupt topographic development near the interface. The dark spots further increase in size if sputtering is continued through stages 4–6 and then open completely at the interface.[7] In SEM, the brightness indicates a secondary electron emission yield by electron beam irradiation. The secondary electron yield of Ta is quite different from that of Si. The dark spots are therefore identified to be the Si substrate produced as a consequence of surface exposure. It is worth pointing out here that the topography size is quite large (~400 nm in size, ~20 nm in height) and round as shown in Figs. 2 and 3. At the stages after the interface,[8–10] the border lines of the topographies disappeared and the dark spots become vague because the Ta thin film has been completely sputtered away and the silicon substrate exposed at the surface. From a point AES spectrum at the dark spot region of a crater,[6] a strong Si signal is observed as shown in Fig. 4 (curve b). But at the outer regions of the dark spot, only a weak Si signal is observed (curve a). These findings suggest that the silicon substrate is exposed at the surface of the dark spots.

The chemical state of the oxygen ion bombarded Ta surface layer is analyzed with a 3 keV O_2^+ ion beam using a 30° incident angle as discussed previously.[11] Under this sputtering condition, the oxidized Ta layer appears at the rising point of the Si^+ ion intensity before reaching the Ta/Si interface. It may cause the topographic development.

These results lead us to propose the following plausible

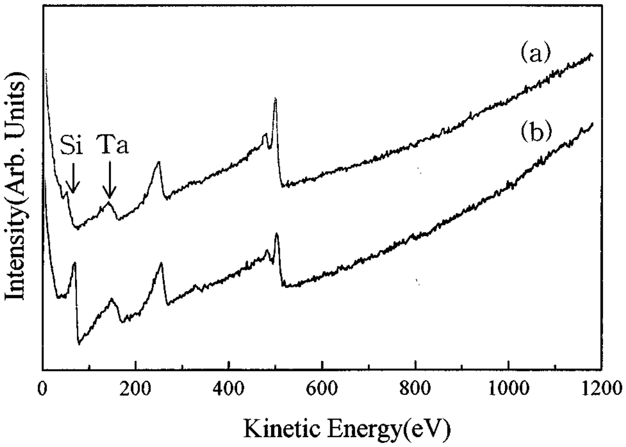

FIG. 4. Point AES spectra at outside (a) and inside (b) of dark spot at stage (6) in Fig. 2.

mechanism for the topographic development. It has been reported that surface stress by ion bombardment can produce dome shaped surface topography.[5,6] By sputtering the Ta thin film on Si with an oxygen ion beam, a Ta oxide layer develops near the interface and its volume should also increase. The thin oxidized Ta film must therefore be under compressive stress. As this stress increases and the thickness of the film decreases, the stressed thin film can be lifted and forms blisters with void formation occurring at the Ta/Si interface to release the stress. With further sputtering, the top regions of blisters may be opened up and result in a crater type of topography. These phenomena are clearly demonstrated in Figs. 2 and 3. Since the blisters do not show up in SEM images because of their relative low heights comparing with the sizes, only the development of craters are shown in Fig. 2 whereas in Fig. 3, the blister formation is observed by AFM. The details of the AFM study will be presented in a forthcoming work.

The variation of Si^+ ion intensity near the interface correlates well with the blister opening process. Secondary ion yield of an element depends on the chemical state or composition of the matrix. In general, the secondary ion yield of an element is higher when it is emitted from an oxidized matrix than from a metallic matrix. For example, Si ion intensity from SiO_2 is about 100 times higher than that from Si. Therefore, during the initial opening process, Si intensity abruptly increased because the Si surface (already oxidized by implanted oxygen ions) is exposed to the surface through the open hole. The Si intensity decreases as this oxidized surface layer is further sputtered. The final rise of Si^+ ion intensity corresponds to the stage where the remaining Ta layers have been completely sputtered away at the real interface.

In summary, abnormal interface artifacts were found using SIMS depth profiling analysis of a Ta/Si interface using an O_2^+ ion beam. They were well correlated with the irregular topographic developments at the interface. SEM and AFM, surface morphology studies, showed quite large crater type topographic development just around the interface, and also showed that the center of the craters gradually opened and exposed the Si substrate to the surface through the exposed area. This report showed that the interface artifacts often found in SIMS depth profiling could be due not only to the atomic mixing and subsequent matrix effect, but also to topographic development around the interface.

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