Three-Dimensional Microanalysis of Solid Materials using Ion and Electron Dual Focused Beam Apparatus

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We developed a novel 3D microanalysis method which is applicable to samples with arbitrary shapes and heterogeneity. Our method employs a combination of micro-cross-sectioning of a sample using a Ga focused ion beam (Ga FIB) and Auger mapping of the cross sections using an electron beam (EB). We realized this method by constructing an ion and electron dual focused beam apparatus. In the apparatus, a Ga FIB and an EB are perpendicularly directed to a sample to define simply the 3D coordinates from the steering of the two beams. An experiment on 3D Auger mapping of a bonding pad of an IC showed well-coordinated 3D elemental maps of Al and Si.

1. Introduction

A true three-dimensional (3D) microanalysis method is desired for the precise characterization of non-flat and inhomogeneous samples like ICs and microparticles. In conventional 3D analysis method called "image depth profiling", analytical surfaces are crater bottoms created with ion beam etching [1]. The depth scale of 3D data is determined from the total etched depth by assuming a constant etching rate of the sample. This definition of the depth scale is available only for flat and homogeneous samples.

First of all, 3D analysis of samples with arbitrary shapes and heterogeneity requires a clear and unique definition of the depth scale. We have developed a novel 3D microanalysis method by means of cross-sectional Auger mapping where the depth scale is determined by the cross-sectioning position using a gallium focused ion beam (Ga FIB). In this paper, we describe the concept of the 3D analysis method, the instrumentation of an ion and electron dual focused beam apparatus, and a result of 3D Auger mapping of a bonding pad of an IC.

2. Novel 3D microanalysis using ion and electron dual focused beams

2.1 Concept

Conventional "image depth profiling" is performed by a combination of ion beam etching of a crater and two-dimensional mapping by scanning Auger microscopy (SAM) or secondary ion mass spectrometry (SIMS) [2,3]. Analytical surfaces are the crater bottoms which define x-y planes. The depth (z) is determined from the final crater depth by assuming a constant etching rate. This determination is applicable only to flat and homogeneous samples, because non-flat or heterogeneous samples cause uneven etching during a measurement. Some efforts were made for the correction of depth scale by post-data processing [4] or direct measurement of crater bottoms using atomic force microscopy during an analysis [5]. These methods, however, require known compositions of a sample and complicated analytical procedures, respectively.

It is the most indispensable condition for realizing a true 3D analysis that the analytical surfaces are always flat and well-positioned at

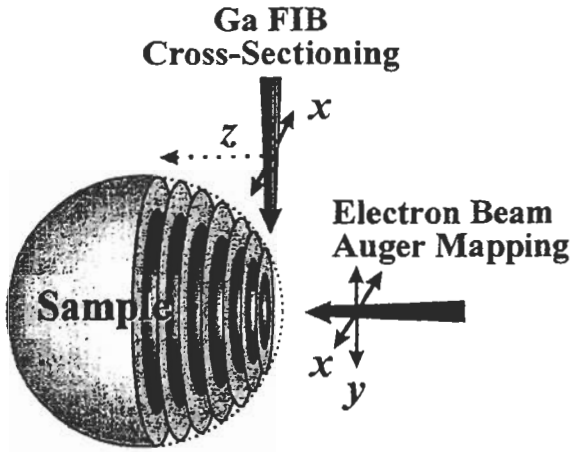

Fig. 1: An illustration depicting three-dimensional microanalysis of a particulate sample using ion and electron dual focused beams.

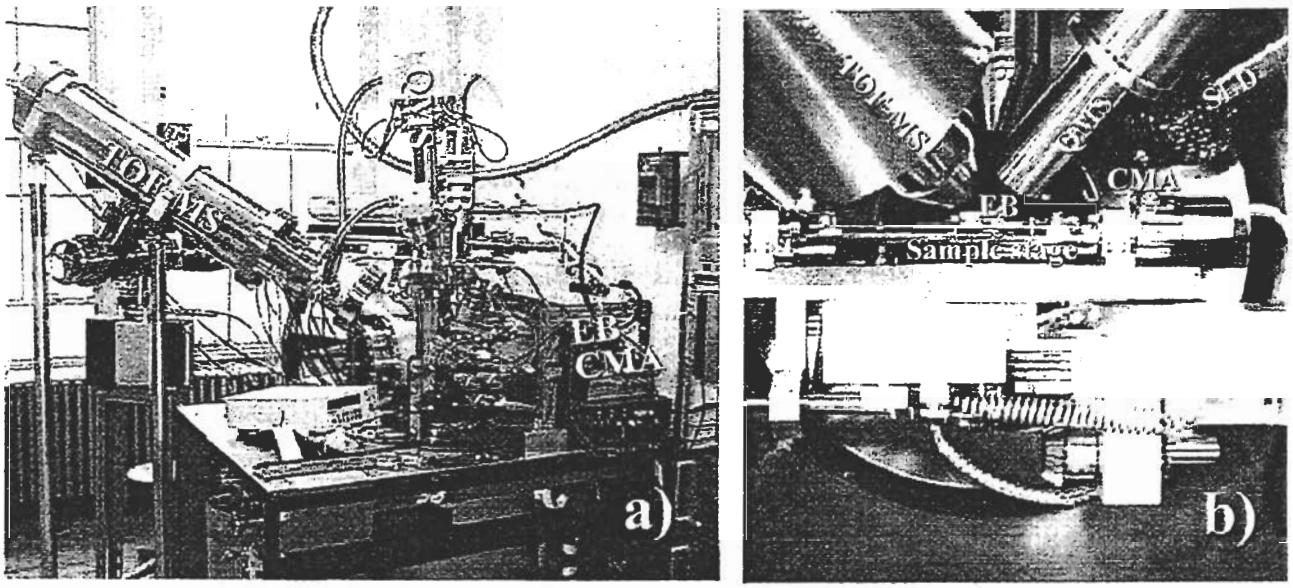

Fig. 2: Photographs of the ion and electron dual focused beam apparatus, a) whole of the analytical chamber, b) analytical components around the sample stage.

aimed depths. We previously developed a precisely positioned cross-sectioning technique using a Ga FIB [6]. We named this technique "Shave-off cross-sectioning". In a Shave-off cross-sectioning, a Ga FIB is scanned like a line scan mode. A line scanning Ga FIB is moved very slowly from the top of an area to the bottom. During a Shave-off scan, a sample within the area is continuously cross-sectioned at the vertical scan position of the Ga FIB.

Our 3D analysis method employs Shave-off cross-sectioning to obtain analysis surfaces at precisely controlled depths. As shown in Fig. 1, a 3D microanalysis of a particulate sample is performed by the repetition of intermittent Shave-off cross-sectioning using a Ga FIB and Auger mapping of the cross section using an electron beam (EB). The depth (z) of a mapping surface (x-y) is easily defined by the cross-sectioning position. Since the cross-sections are almost parallel to the Ga FIB axis, the x-y-z coordinates are defined by the beam steerings of the Ga FIB and EB, if the EB is arranged perpendicularly to the Ga FIB axis.

Spatial resolution within the x-y plane is identical to the Auger mapping resolution reaching a few 10 nm by recent Auger apparatuses. The Ga FIB cross-sectioning determines the z resolution. In this case, the spot diameter of the Ga FIB is not necessarily a limiting parameter for z resolution, because the cross section is not created with the whole of the spot, but its edge part. Recent result of Shave-off cross-sectioning in combination with SIMS analysis showed a resolution better than 10 nm using a Ga FIB with a 100 nm diameter [7]. Therefore, 10 nm resolutions for x, y and z directions are expected if state-of-the-art analytical components are used.

As for the contamination on the cross section, our measurements by means of Auger electron spectroscopy showed that there is less implanted Ga and redeposited materials compared with conventional raster scanning ion beam etching. Details of these results are described elsewhere [8].

2.2 Instrumentation

We realized the 3D analysis method by constructing an ion and electron dual focused beam apparatus shown in Fig. 2. The apparatus mainly consists of a Ga FIB column (Eiko-Engineering, FI-1000), a LaB_6 EB column (PHI) coaxially arranged with a cylindrical mirror analyzer (CMA, PHI), a quadrupole mass spectrometer (QMS, Hiden Analytical, EQS-300) and a time-of-flight mass spectrometer (TOF-MS, homemade). The Ga FIB, EB and CMA are used for 3D Auger mapping, while two mass spectrometers are used for dynamic-SIMS or TOF-SIMS. As shown in Fig. 2 b), the Ga FIB and EB are perpendicularly arranged. The minimum spot diameters of the Ga FIB and EB are 0.1 μm (at 25 keV energy and 60 pA current) and 0.5 μm (at 10 keV, less than 1 nA), respctively. The analytical chamber is maintained at 10^{-9} Torr with four

sputter ion pumps. The control system consists of a scanning Auger microscope controller, a Ga FIB controller, QMS and TOF-MS controllers and two computers for remote control and data acquisition.

In a 3D analysis, first, a cross section is created with the Ga FIB. Secondly, Auger maps of the cross section are acquired while the Ga FIB is blanked. We named these two operations one "stage". Following stages are performed while moving the cross-sectioning position towards z direction with an arbitrary step. 3D Auger maps are easily reconstructed from acquired cross-sectional Auger maps by stacking them along z axis.

3. Experimental

We performed a 3D analysis using a 64 Kbit EP-ROM IC as a sample. Prior to the experiment, a Au film was deposited on the IC surface to prevent charge-up problems. The 3D analysis was applied to one of the bonding pads of the IC. The bonding pad consisted of an Al bonding wire, an Al pad and a Si substrate. The analysis region and z step of one stage were set to be $70(x)\text{x}70(y)\text{x}50(z)$ μm^3 and 1.6 μm, respectively. Total number of stages was 32. The apparatus was operated under a computer control mode. In each stage, Al_{LVV} and Si_{LVV} Auger maps (128x128 pixels) were acquired by taking secondary electron images at Auger peak energies and background energies. The Ga FIB and EB were operated under conditions of 20 keV energy and 2.17 nA current, and 4 keV and 1 nA, respectively. The sample was tilted 20 deg with respect to the Ga FIB axis. Total analysis time was about 22 h consisting of 640 min for cross-sectioning and 660 min for Auger mappings.

4. Results and Discussion

An EB-induced sample current image of the bonding pad before the analysis is shown in Fig. 3. The 3D analysis was started near the tip of the bonding wire. Secondary electron images of the bonding pad at stages 5, 15 and 25 are shown in Fig. 4. At stage 5, the tip of the bonding wire was started to be cross-sectioned. At stages 15 and 25, the structure of the Al wire, Al pad (dark layers within the cross sections) and Si substrate (bright layers) are recognized.

From 128 energy-selected secondary electron images, 32 Auger maps of Al_{LVV} and Si_{LVV} were obtained, respectively. Reconstructed 3D Auger maps of Al and Si are shown in Fig. 5. The Al wire on the Al pad (Fig. 5 a)) and Si substrate (Fig. 5 b)) are successfully shown in a 3D space. From the experimental condition, one voxel size is calculated to be $0.55(x)\text{x}0.55(y)\text{x}1.6(z)$ μm^3.

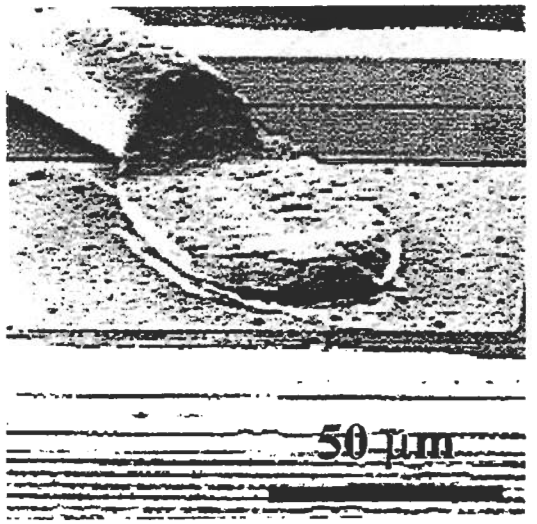

Fig. 3: EB-induced sample current image of the bonding pad before the 3D analysis.

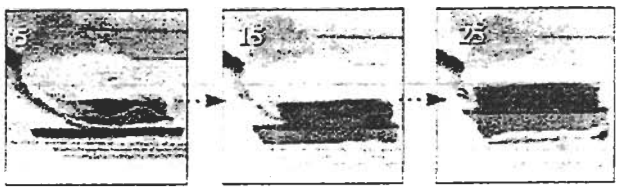

Fig. 4: Secondary electron images at stage 5, 15 and 25 during the 3D analysis.

In the Si map, one can notice the unexpected object with a shape of the tip of the Al bonding wire on the Si substrate. This was caused by the redeposition of sputtered Si atoms onto the underside of the overhanging tip viewed from the Ga FIB. We can safely neglect this object, because it is not assigned to a certain depth in the cross-sectional Auger mapping.

In general, mapping analysis requires long analytical time and frequently causes a problem due to sample stage drift. In this experiment, drift lengths of 6 μm for 22 h both for x and y direction were observed from the acquired secondary electron images. We tried to correct x-y coordinates by choosing a reference point outside the cross sections. A raw 3D Auger map (an overlay of Al and Si maps) is shown in Fig. 6 a). The stage drift resulted in the bend of the interface of the Al pad and Si

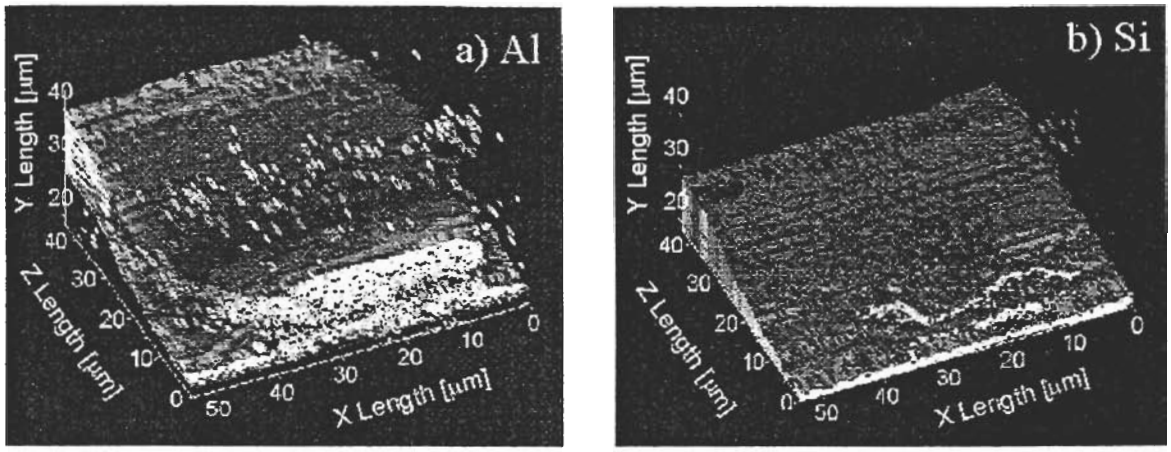

Fig. 5: 3D Auger maps of the bonding pad, a) Al_{LVV} and b) Si_{LVV}.

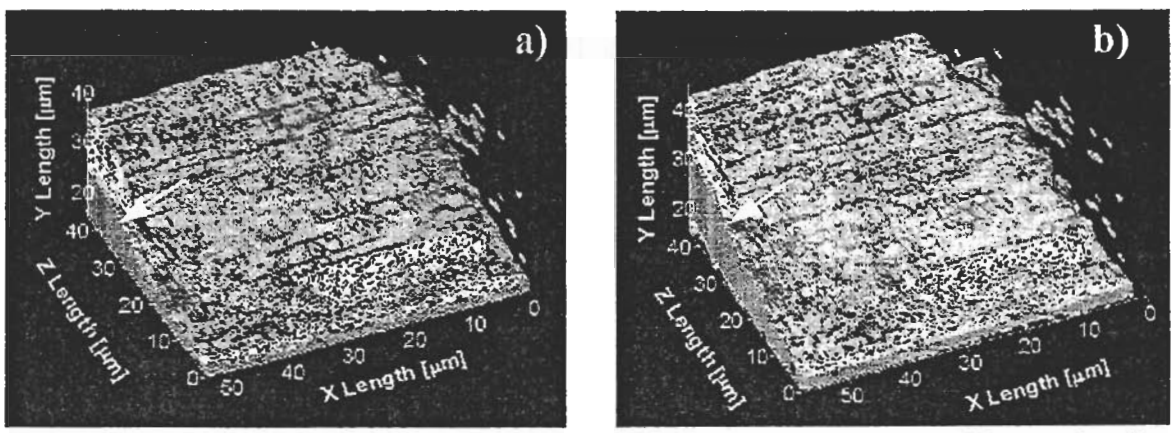

Fig. 6: Overlay 3D maps of Al and Si, a) a raw 3D map and b) a 3D map after the drift correction for *x*-*y* coordinates. White allows indicate the interfaces of Al pad and Si substrate.

substrate indicated by an arrow. After the correction for x and y coordinates, the interface was properly reconstructed as a straight line in Fig. 6 b). The correction accuracy depends on the image resolution (about 1 μm in this experiment). Correction for z direction will be possible if Ga FIB-induced images during cross-sectioning are acquired.

5. Conclusion

We described a novel 3D analysis method and the instrumentation of the ion and electron dual focused beam apparatus for 3D analysis.

The 3D analysis was applied for a bonding pad of an IC. The result showed well-coordinated 3D Auger maps with the aid of the coordinate correction for the stage drift. The analysis required 22 h because of relatively large volume of the sample. If the Auger mapping resolution of the apparatus is improved, we can apply this method to smaller volume down to 1x1x1 μm^3 with much shorter analysis time, because the time required for the cross-sectioning is largely reduced.

6. References

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