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High-resolution x-ray tomography using laboratory sources

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ABSTRACT

X-ray computed tomography (XCT) is a powerful nondestructive 3D imaging technique, which enables the visualization of the three dimensional structure of complex, optically opaque samples. High resolution XCT using Fresnel zone plate lenses has been confined in the past to synchrotron radiation centers due to the need for a bright and intense source of x-rays. This confinement severely limits the availability and accessibility of x-ray microscopes and the wide proliferation of this methodology. We are describing a sub-50nm resolution XCT system operating at 8 keV in absorption and Zernike phase contrast mode based on a commercially available laboratory x-ray source. The system utilizes high-efficiency Fresnel zone plates with an outermost zone width of 35 nm and 700 nm structure height resulting in a current spatial resolution better than 50 nm. In addition to the technical description of the system and specifications, we present application examples in the semiconductor field.

Keywords: x-ray microscopy, computed tomography, Fresnel zone plate.

1. INTRODUCTION

Structure determination of crystalline materials is routinely performed by x-ray and neutron diffraction techniques capable of atomic resolution. However, in the absence of long-range order imaging techniques are required for structural analysis. Visible light or electron microscopic techniques are commonly employed and widely available. The use of shorter wavelength electromagnetic radiation such as x-rays extends the theoretical resolution limit for imaging from hundreds of nm for visible light to the sub-nm regime for x-rays. In addition, multi-keV x-rays interact weakly with matter compared to electrons, which allows them to penetrate deep into samples and does not require extremely thin samples as is the case for electron microscopy. In the last two decades significant progress has been made in the field of x-ray microscopy utilizing Fresnel zone-plates as objective lenses, now achieving better than 15 nm resolution at synchrotron light sources [1-8]. Zone-plate x-ray microscopes both in scanning and full-field imaging geometry now exist at more than twenty international synchrotron facilities and new ones are being constructed or in the planning phase. High resolution x-ray microscopy at multi-keV x-ray energies opens up new avenues to nondestructively explore the internal structure of optically opaque solids with submicron resolution previously inaccessible by other analytical techniques.

Large scale studies using x-ray microscopy require laboratory based instruments that provide easy access and reliable operation with performance comparable to synchrotron based instruments. Recently first soft x-ray energy standalone microscopes for biological applications in the high contrast "water window" (284-560 eV) x-ray energy range have been reported [4,8]. These instruments take advantage of the development of high-powered laser excited plasma sources to generate soft x-rays. For applications with inorganic materials, such as semiconductor samples, more energetic x-rays are required to achieve better x-ray transmission through up to 100 um of material [2,3,5,6]. For this purpose we developed a hard x-ray tomographic x-ray microscope based on a commercial x-ray generator using the Cr-Kα emission line (5.4 keV), which was commercialized in 2003 for semiconductor failure analysis as the primary application [2]. This system enables 3D tomographic imaging of Cu interconnects through up to 30 μm thick integrated circuit (IC) silicon dies.

Sample preparation of ICs for the 5.4 keV x-ray energy XCT system requires thinning of the silicon die to 30um thickness or less due to the limited penetration of 5.4 keV x-rays through silicon. While suitable preparation techniques for thinning the silicon substrate such as dimpling are well established and widely used, it is highly desirable to use a higher x-ray energy to simplify sample preparation and increase penetration for higher angle projections in computed tomography. In order to increase the transmission in Si from 30 μm up to 100 μm, we have chosen to adopt a

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commercial x-ray generator with a copper target producing Cu-Kα x-rays with an x-ray energy of 8 keV. Since the absorption contrast diminishes rapidly with x-ray energy ($\sim 1/E^3$), we have equipped the XCT system with Zernike phase contrast optics utilizing the slower drop-off (1/E) of phase shift with increasing x-ray energy.

2. DESIGN AND PERFORMANCE OF FULL FIELD X-RAY MICROSCOPE

Figure 1 shows schematically the main components of the tomographic x-ray microscope. The microscope is configured as a full-field transmission microscope much like a transmission electron microscope or a regular visible light microscope. A commercial x-ray generator with ~100 μm diameter source size and copper anode is employed in conjunction with an energy filter to reduce x-ray intensity which does not fall into the Cu-Kα emission line (E=8.05 keV, wavelength λ=1.54 Å). A reflective condenser optic refocuses the x-rays onto the sample. This optic has the advantage over Fresnel zone plate condensers that it can have a numerical aperture higher than the objective lens and that it has much higher focusing efficiency. The sample is located on a high-precision rotation stage that allows to acquire 2-D projection images for a large range of rotation angles (up to +/- 80 degrees). The key element, the objective, is a Fresnel zone plate. Fresnel zone plates are diffractive optical elements, which focus x-rays by means of diffraction not refraction [10]. The Fresnel zone plate objective creates a magnified image of the sample on the high-resolution x-ray detector. For a compact microscope it is advantageous to employ a very high spatial resolution x-ray detector in order to minimize the sample to detector imaging distance. In addition, it is very important to use a detector with very high quantum efficiency in order not to loose valuable x-ray photons in the detection process. We have developed a detection system with >50% quantum detection efficiency and resolution better than 2 um based on a scintillation screen coupled to a CCD detector. For implementation of Zernike phase contrast a transmissive phase ring is added in the back focal plane of the objective zone plate. The phase ring is fabricated out of gold and has a thickness designed to produce a phase shift of 3π/2 of the beam not diffracted by the sample. This phase shift produces negative phase contrast, i.e. features with strong phase shift appear dark. The Rayleigh resolution δ of a x-ray microscope is a function of the outermost zone width Δr_n of the objective zone plate:

$$\delta = 1.22\Delta r_n \qquad (1)$$

This formula is strictly only valid for coherent illumination (parallel light), and better resolution is routinely obtained using incoherent illumination (illumination from a range of angles) [13]. Using the simple Rayleigh formalism, the theoretical resolution of the objective zone plate with Δr_n=35 nm is computed to be δ=42.7 nm using Equation 1. Note that the resolution of the zone-plate based x-ray microscope is independent of x-ray source spot size and ultimately limited by the outermost zone width of the zone plate (finer zones give higher resolution). As a consequence, the resolution of a x-ray microscope is inherently coupled to the progress of the fabrication technology of Fresnel zone plates. While it is relatively simple to pattern high-resolution structures using electron beam lithography, it is very challenging to fabricate these fine features very tall, which is needed in x-ray focusing to have sufficient focusing efficiency.

One big advantage of using 8 keV x-rays for imaging is that the working distance (distance between sample and objective) is quite large, which originates in the proportionality to x-ray energy *E* of the focal length *f* of a zone plate:

$$f = \frac{D\Delta r_n}{\lambda}, \qquad (2)$$

where *D* is the diameter of the Fresnel zone plate objective. As an example, the working distance of the microscope operating at 8 keV with a zone plate of diameter <100 μm can be greater than 20 mm, which is 40 times more than typical working distances for "water-window" soft x-ray microscopes (typically <<1mm working distance).

The price to pay for a long working distance L_1 is that the imaging distance L_2 can be quite large since the x-ray magnification *M* of the microscope has to be quite large due to quite poor spatial resolution of area x-ray detectors. The geometric magnification is given as:

$$M = \frac{L_2}{L_1} \tag{3}$$

L_2 and L_1 are distances that satisfy the well-known thin lens imaging condition

$$\frac{1}{f} = \frac{1}{L_1} + \frac{1}{L_2} \tag{4}$$

To obtain an imaging resolution of 42 nm, the sample has to be oversampled by a factor of ~2.5, leading to a physical pixel size of 17 nm in the sample plane. Using the high resolution x-ray imaging detector with ~2 µm physical pixel size the required x-ray magnification is M~40 and therefore the image distance L_2 gets close to 1 m.

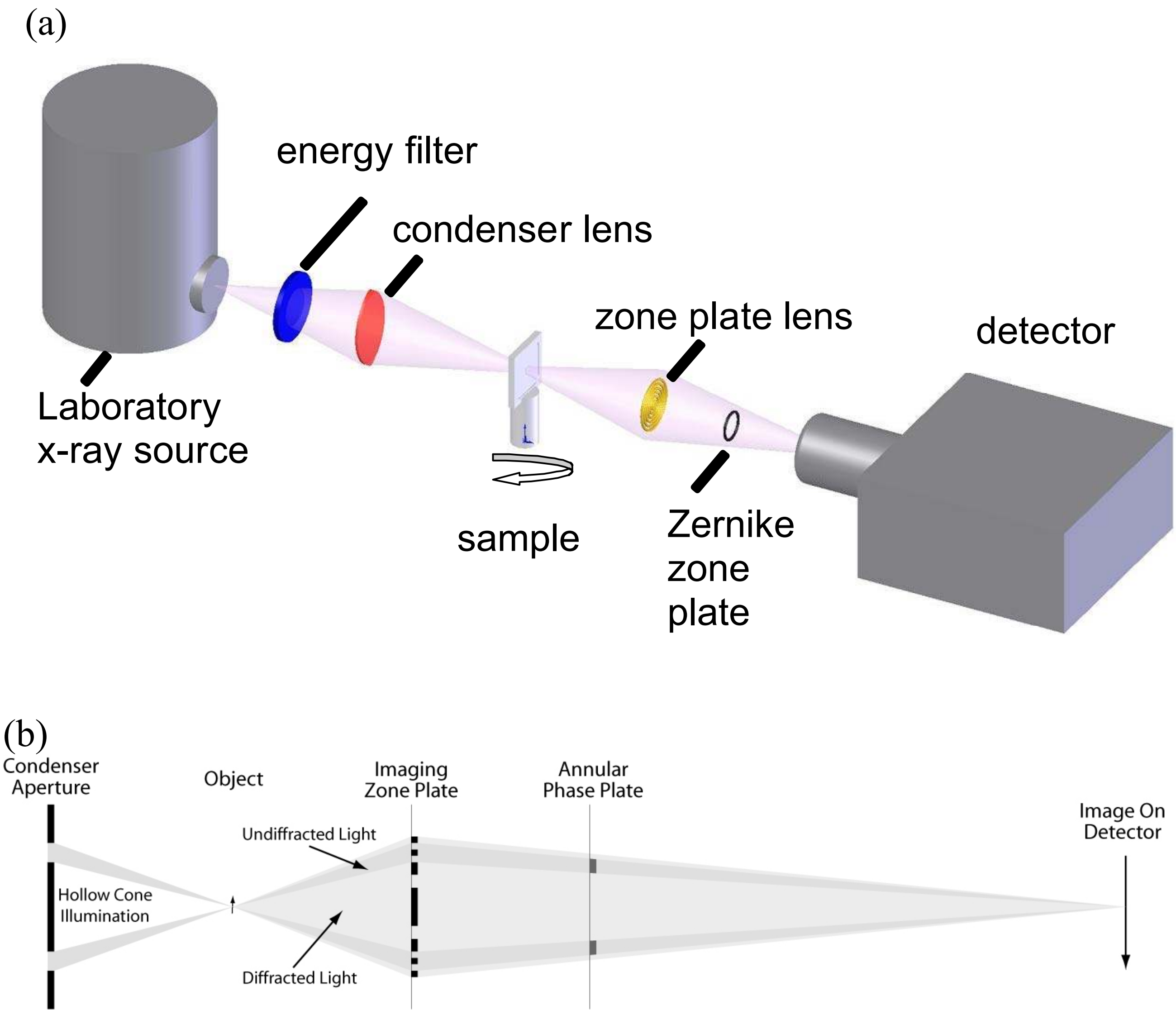

Figure 1: a) Schematics of the zone-plate based x-ray microscope operating in Zernike phase contrast mode. b) Imaging geometry.

A second big advantage of a hard x-ray microscope over a "water-window" soft x-ray microscope is the long depth of focus, which is needed for tomography. The requirement for computed tomography based on back-projection is that the whole reconstructed volume has to be within the depth of focus of the microscope. Quantitatively, the depth of focus *DOF* is given by:

$$DOF = \pm \frac{\lambda}{2NA^2} = \pm \frac{2\,\Delta r_n^{\;2}}{\lambda}\,, \tag{5}$$

where *NA* is the numerical aperture of the zone plate defined by:

$$NA = \frac{\lambda}{2\Delta r_n} \tag{6}$$

, where λ is the x-ray wavelength and Δr_n the outermost zone width of the objective zone plate. For example for 8 keV x-rays (λ=0.154nm) and a zone plate with 35 nm outermost zone width, the corresponding numerical aperture is 2.2 mrad, and *DOF* is approximately 32 μm, which is very comfortable for high-resolution tomography. In contrast using the same outermost zone width zone plate of 35 nm with soft x-rays, e.g. 3 nm wavelength, the *DOF* is severely reduced to less than 2 μm, which clearly poses a problem for high-resolution tomography of larger volumes. Figure 2 depicts the transmission image of Au resolution test pattern 700 nm high (Au structure appear with dark contrast). Smallest spacing between the tips of the Au lines inside the most inner circle is 50 nm.

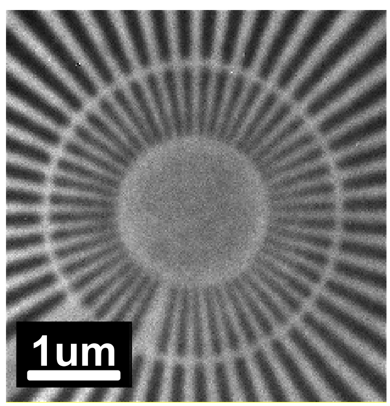

Figure 2: Transmission image of 700 nm thick Au pattern where the line spacing at the tips of the inner most circle is 50 nm. Image was taken with x-ray microscope operating at 8 keV in Zernike phase contrast mode using objective zone plate with 35 nm outermost zone width.

Large *DOF* and large working distances of L_1~20 mm are very well suited for high-resolution tomographic 3D imaging of macroscopic objects, such as semiconductor chips. Computed tomography is a well-known imaging technique to reconstruct a 3D volume of the object from a series of 2D projections from a variety of rotation angles [12]. The x-ray microscope described in this work takes advantage of high penetration of 8 keV x-ray photons into Si. For example, sufficient transmission can be achieved through up to 100 um of solid Si, which allows 3D volume reconstruction of all metal layers of Cu interconnects inside modern IC chips with sub-50 nm resolution.

The contrast *K* of a small particle of material 1 imbedded into material 2 is defines as

$$K = \frac{I_1 - I_2}{I_1 + I_2} \tag{7}$$

, where I_1 is image intensity of the small particle of material 1 and I_2 is image intensity of the surroundings which consist of material 2. In addition to absorption mode where the contrast is due to differences in attenuation length of material 1 and material 2, the described x-ray microscope takes advantage of the Zernike phase contrast mode using a phase ring (see Fig. 1) in the back focal plane of the zone plate [11]. The thickness of the phase ring is chosen to advance the phase of the x-rays not diffracted by the specimen by $3\pi/2$ (negative phase contrast), which interferes with the x-rays diffracted by the sample in the detector plane. As a result a significant contrast boost can be obtained. Following the simple formalism of Rudolph et al. [1], which does not account for the limited aperture of the objective lens and finite phase ring width, the absorption and phase contrast of 50 nm thick Cu features imbedded into Si was calculated and presented in Fig. 3. It can be seen that at 8 keV x-ray energy, the contrast is increased by a factor greater than 80 times. 1% contrast is typically sufficient for tomographic reconstruction algorithms to clearly resolve features.

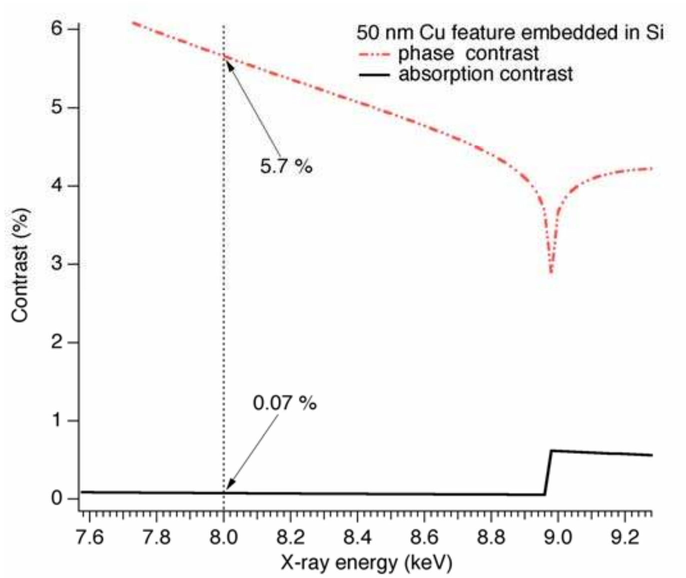

Figure 3: Calculated absorption and phase contrast for a 50 nm thick Cu feature embedded in Si following Ref. 1.

3. EXPERIMENTAL RESULTS

The described XCT system is used in a variety of different applications, including R&D and failure analysis of interconnects in semiconductor ICs, fuel cell R&D, fission and fusion R&D, and bio-medical applications. We are presenting here some of the results from imaging Copper interconnects in ICs. Figure 4 shows a 2D full field transmission image through an IC chip with nine Cu metallization layers embedded into Si die. The image was taken with a Cu laboratory source (8 keV x-ray energy) in Zernike phase contrast mode. The size of the smallest Copper feature in the image is 90 nm and the Si die was backside thinned down to ~70 um thickness to increase the x-ray transmission. The collapse of all metal layers into a single projection image makes it very difficult to discern the geometrical structure of the Cu lines and vias connecting the individual layers from a single image. The XCT technique enables the 3D volume reconstruction and subsequent separation of all metal layers for individual inspection. A series of such projections was taken with a rotation range of +/-70 degrees (limited by sample holder shadowing). Figure 5 shows a volume rendering of the computer-reconstructed data from 140 projection images.

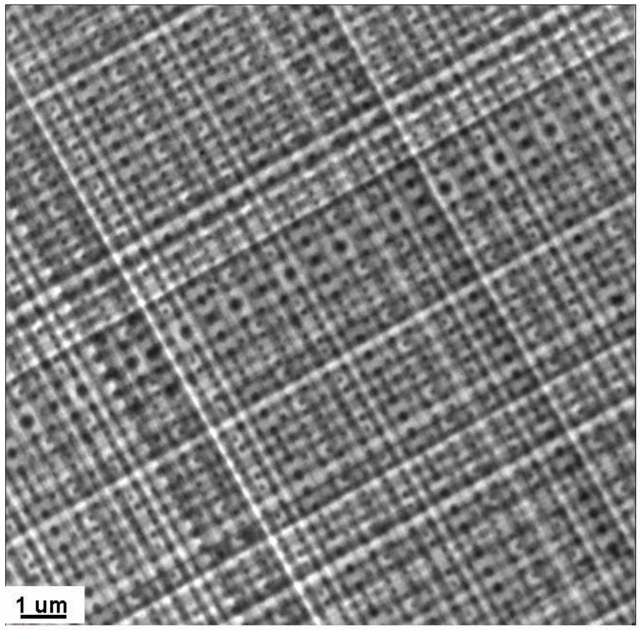

Figure 4: 2D projection image of a 70 um thick Si integrated circuit (IC) with 9 Cu metallization layers at normal incidence (0 degree).

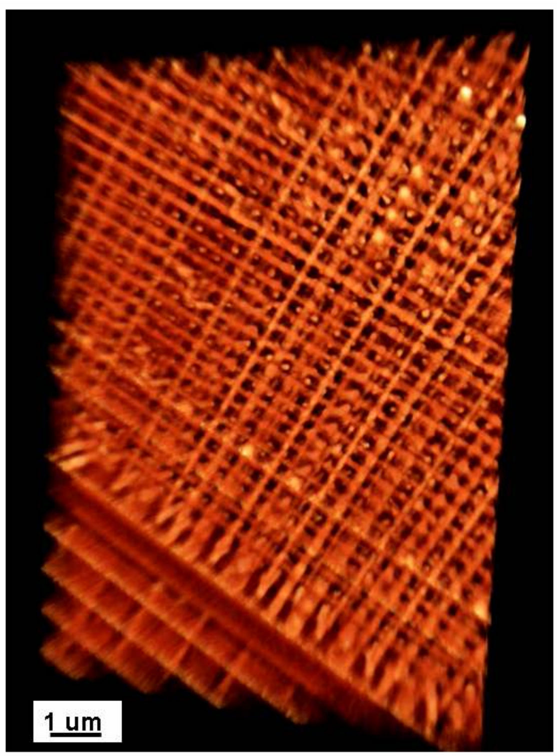

Figure 5: Volume rendering of an IC chip with 9 Cu layers from 140 2D projections.

The reconstructed 3D dataset of the Copper interconnect stack of the IC subsequently can be “virtually delayered” and individual layers were extracted as shown in Figure 6.

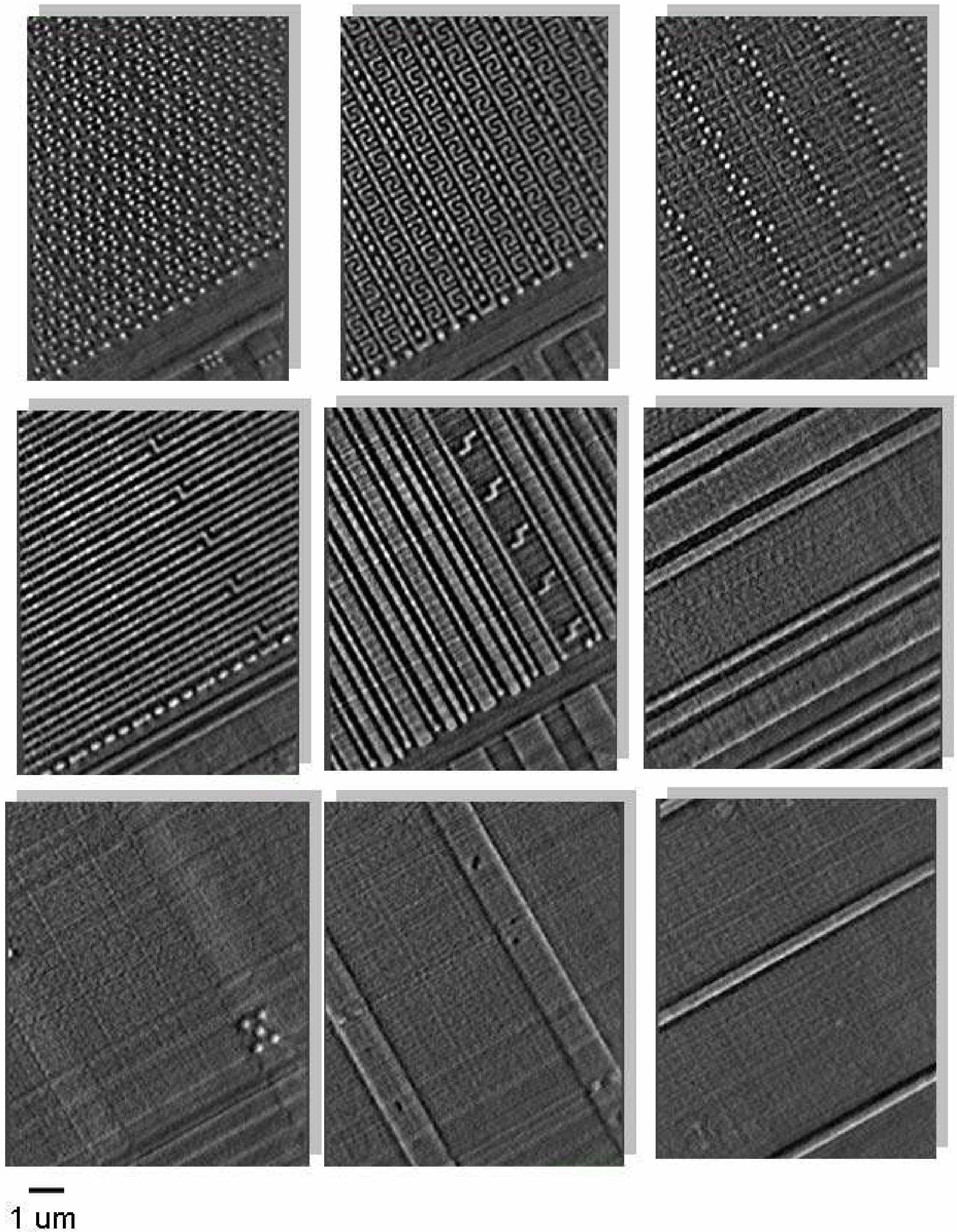

Figure 6: Virtual delayering of nine Cu metallization layers from M1 (top row left image) to M9 (bottom row right image). Sub-100 nm voids in broad Cu lines (bottom row center image) are clearly observed.

Cu lines are shown with bright contrast while Si and voids appear dark in the images. The results shown in Fig. 6 clearly demonstrate the power of the tomographic imaging of Cu interconnects with 50 nm resolution using hard x-rays for direct visualization of defects in the Copper interconnect stack. Since the Cu is imaged with x-rays whose energy is below the Cu absorption edge, the main contribution to the image contrast is attributed to the Zernike phase contrast enhancement as illustrated in Fig. 3.

4. CONCLUSIONS AND OUTLOOK

We have demonstrated sub-50nm resolution tomographic nondesturctive 3D imaging of multi-level Cu interconnect structures through thick Si integrated circuit with a hard x-ray microscope based on a laboratory x-ray source operating at 8 keV x-ray energy in Zernike phase contrast mode. The feasibility of such a laboratory system is based on our development of high performance, high efficiency x-ray optical components, namely the x-ray condenser lens, the x-ray objective zone plate, and the x-ray detector. Virtual delayering of reconstructed data offers a completely nondestructive way to inspect multiple layers from the 3D reconstructed volume for visualization and failure analysis. In the future, the continued advancement in zone plate resolution and efficiency in the hard x-ray energy range promises even higher throughput and spatial resolution. The development of novel laboratory x-ray sources may also open new opportunities for faster, higher resolution x-ray microscopy in the more distant future. It is expected that hard x-ray XCT will proliferate into many areas of R&D and industrial applications as a standard nondestructive microscopy technique that bridges the gap between visible light and electron microscopy.

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