Dark Field Image of Full-Field Transmission Hard X-ray Microscope in 8-11 keV

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ABSTRACT

We have demonstrated dark-field imaging using a full-field hard x-ray microscope by using a custom capillary-based condenser. The condenser provides illumination with a numeric aperture about 3-mrad with high efficiency. This high illumination angle allows full-resolution imaging using a 50 nm hard x-ray zone plate. The zeroth order beam from the condenser is well out of the zoneplate range - which allows a high signal-to-noise ratio in the image plane. Small particles with high scattering power, such as colloidal gold markers used in biology are well-suited for dark-field imaging. Combining with high brightness source from NSRRC BL01B, the dark field image can be acquired within several minutes with high contrast ratio. In this paper, the dark field image of IC and the zoneplate defect will be demonstrated and studied in different energy under dark field mode.

Keywords: Transmission X-ray Microscope, Dark field, Contrast.

1. INTRODUCTION

The transmission x-ray microscope (TXM)[1-3] is growing rapidly in last decade. Currently, 15nm spatial resolution of transmission soft x-ray is demonstrated[4]. Meanwhile, the resolution of tomography is also demonstrated to be close 60nm[5]. The future trend of x-ray microscope is aiming for the resolution down to 10nm[6] and the resolution will be further down to 1nm with the upcoming fourth generation source. Despite the endless completion of the spatial resolution, the contrast of image is even more important because the interaction between x-ray and material is low compare to the electron and material. The dark field image gives the high contrast ratio in the x-ray microscope for high small particles with scattering power, which can be a promising way to investigate the gold markers in biology sample. Actually, this technique has been widely deployed and studied on the soft x-ray microscope[7,8]. Combining the information from bright field and dark field, the position of the marker can be found and prevent the misleading for the absorption by thick sample.

1.1 The TXM in NSRRC

The similar idea can be implemented in hard x-ray microscope. The dark field image is based on a TXM[5] which has already been built in NSRRC in 2005. The microscope is installed at Beamline BL01B[9] of NSRRC on a superconducting wavelength shifter (SWLS) source, which provide x-rays from 6 keV to 20 keV. After the focusing mirror and Ge (111) double crystal monochromator, the x-rays are further shaped by a condenser. Irradiated by the x-ray of cone shape from a condenser, the sample is magnified by an objective zone plate and the image is acquired by a scintillation/CCD composite camera. The spatial resolution of the microscope is 60 nm using the first diffraction order of the objective zone plate**.** The optical layout of the system is shown in fig1.

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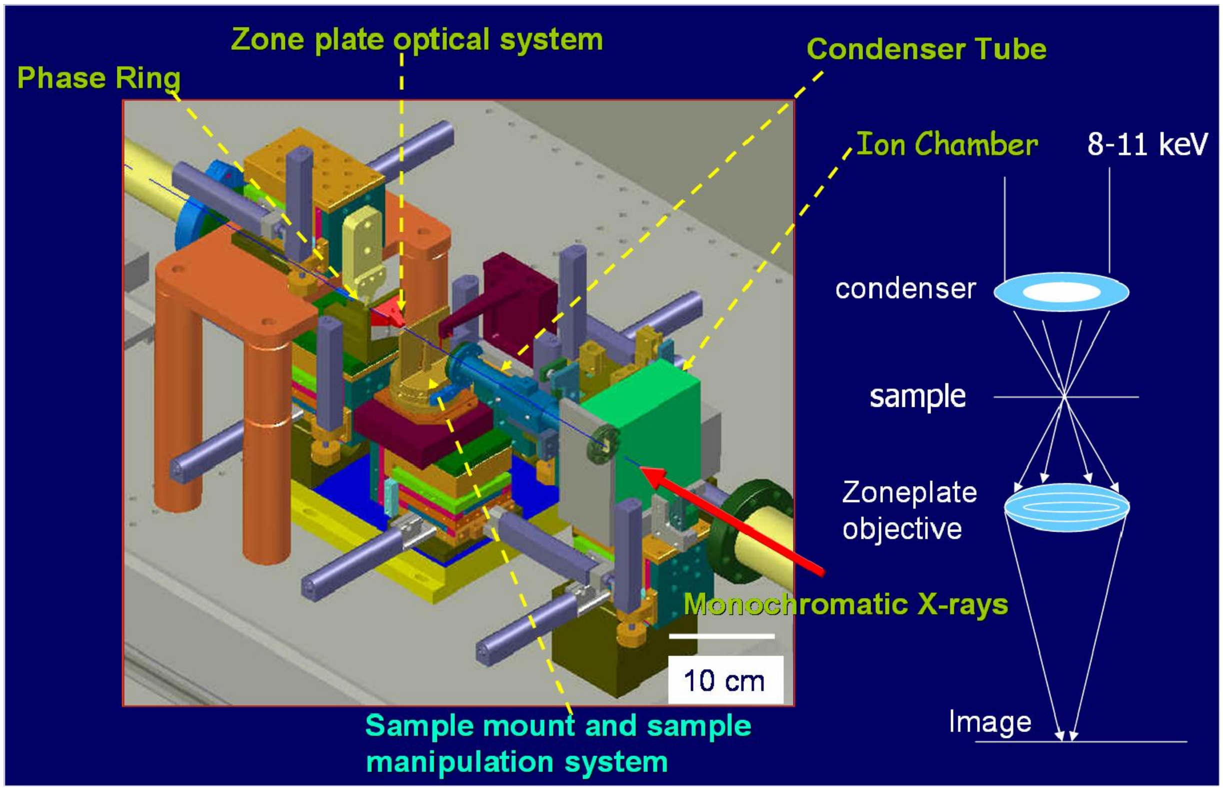

Fig. 1. The setup of TXM in NSRRC. The microscope is mainly composed of three key components, which are the condenser, zoneplate objective and image detection as shown in the right part in this figure. This microscope is operated at the energy range from 8 to 11 KeV with 60nm spatial resolution.

1.2 The optics of the TXM

The optical system of the NSRRC-TXM is composed of 3 capillary condensers[10], a sample manipulation/positioning system, 3 objective zone plates, 3 phase rings (when operating in phase contrast mode), and a scintillator, a 20 times objective, and CCD composite imaging system. For a Fresnel zone plate, the diffraction-limited resolution is $1.22 \cdot \Delta r$, where Δr is the outermost zone width. This TXM is operated at photon energy from 8 to 11 KeV, equipped with zoneplate sets with 900nm thickness of gold and 50nm out most zones, high brightness source, and condenser sets with different fill and illumination angle. With this TXM, We demonstrate the dark field image by a using a condenser that has a three times larger than the numerical aperture (NA) of this microscope.

2. IMPLEMENT THE DARK FIELD IN TXM

2.1 Two ways for implementing the dark field in TXM

The dark field of X-ray microscope can be formed while the principle light does not enter the detector. One way to implement the dark field is to block the principle light in the optical system, and the high spatial frequency is enhanced. As shown in fig.2 (b), because of hollow cone illumination in TXM, the way to generate a dark field is to set a beam stop at the back focal plane, which usually the way to implement the dark field in soft x-ray microscope. The other way to implement the dark field is to use the high angle illumination condenser, as shown in fig. 2(a). In this setup, the principle light is out of the zoneplate range; therefore, only the x-ray of high frequency of the diffraction and the scattering from the sample will enter the optical system which results in a high contrast image.

For the first method, there are some problems to fabricate the ring stop in hard x-ray region. First, the ring stop should be thick enough in order to block the principle light. For example, to order to block 99 percent of 8 keV x-ray, it requires a gold layer of 10 μm thick, which is hard to fabricate by electroplating. The second issue is that the ring shape is not easy to match for the beam shape in the back focal plane. The reason is that the emittance is different in horizontal and vertical because of the natural of the synchrotron source.

For the second method, it requires that the condenser has higher NA than the NA of objective lens. In principle, if the condenser is made by zoneplate and the NA of zoneplate is limited by the wavelength and the outmost zone width as given by $NA_{zoneplate} = \frac{m\lambda}{2\Delta r}$. Where the m is the diffraction order of zoneplate, λ is the wavelength and Δr is the width of outmost zone of zoneplate. In TXM, the wavelength should be the same in the system, and Δr is the same for the fabrication technique. Of course, it is possible to use higher order of diffraction; however, the efficiency is generally too low.

A high NA condenser can be made by capillary, which utilizes the internal reflection x-ray. It has two advantages, one is the better optical efficiency and the other one is high NA could be provided. The capillary is made of glass-based material and the critical angle in 8 KeV is around 3mrad[11]. The NA of zoneplate in 8 keV is around 1.5mrad, for the outmost zone is 50nm. Thus, the capillary condenser could be used to generate higher NA than the zoneplate system and it is good for generating the dark field in TXM.

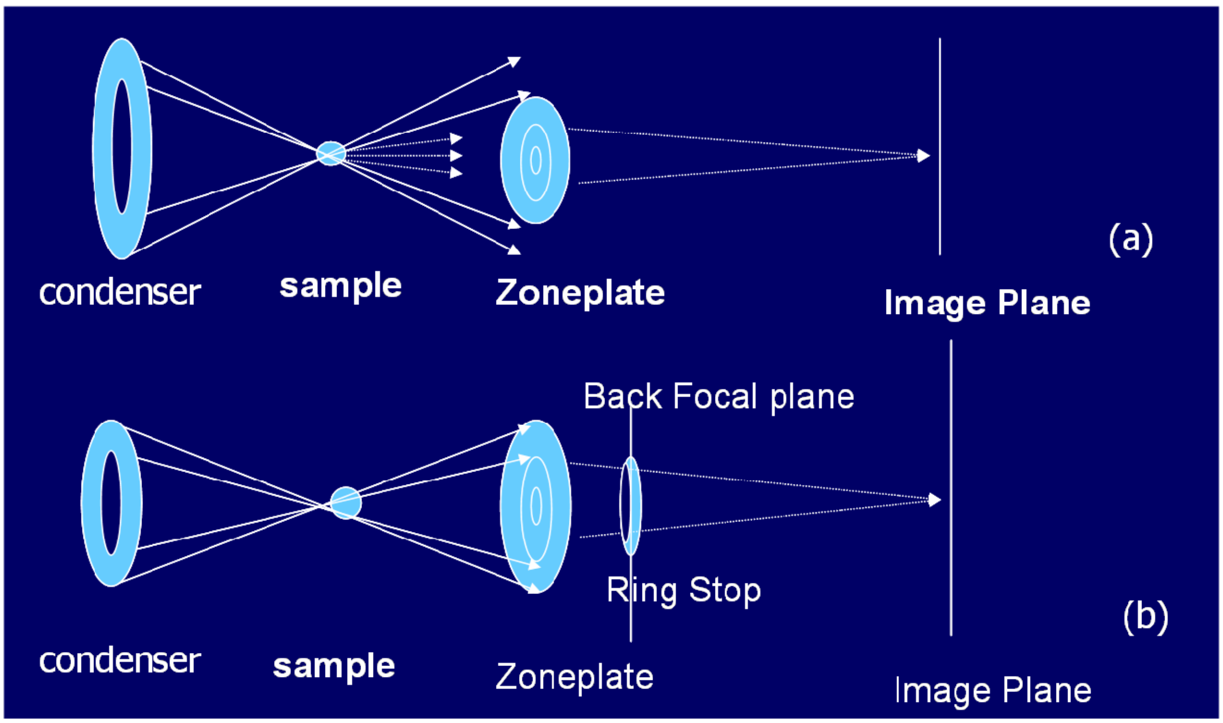

Fig. 2. Two methods implement the dark field in TXM. (a) The dark field is generated by a high angle illumination which can be provided by a capillary-based condenser (b) The dark field is generated by using a beam stop at back focal plane, and the shape of beam stop is a ring because the illumination in TXM is hollow cone. The beam stop is hard to fabricate in hard x-ray region.

2.2 The frequency response of the dark field

We consider the system by frequency because the image is the convolution of input signal and the system response. Thus it is easy to explain the behavior of the image in the frequency domain. The frequency response of the dark field is based on the method of high illumination which is illustrated in fig 2(a). The frequency response is as shown in the Fig3. The vertical axis is intensity (not in scale), and horizontal axis is for the frequency (not in scale). The solid blue line indicates the system response. The bright field information of a Seimen's star of 30nm is plotted as a dotted yellow line which is flat in low frequency, and decreases while the spatial frequency is close to 30nm. The solid yellow line indicates the bright field image in frequency domain, which is a convolution of system response and bright field information of Seimen's star. Followed with the same idea, the dark field image is also the convolution of the system response and the dark-field information from the sample, which is indicated in dotted red line. The dark field information of the dark field is different from that of the bright field because the signal comes from the higher order diffraction and scattering from the sample. Thus, a peak at high frequency can be found, and the position of the peak is varies with the illumination angle. The intensity of the peak is not in scale with the intensity of bright field. Actually, the signal from the dark field is weak compare to that of the bright field. The dark field image in frequency in frequency domain is plotted as solid red line.

The frequency response of the dark field image is low in the low spatial frequency and increase at high frequency then decrease quickly while close to the system response. The dark field seems to have better performance of frequency than the bright field; however, in real case, the long exposure time leads to the noise, vibration and thermal drift always lower down the spatial resolution. According to our experiment result, the dark field image doesn't increase the spatial resolution.

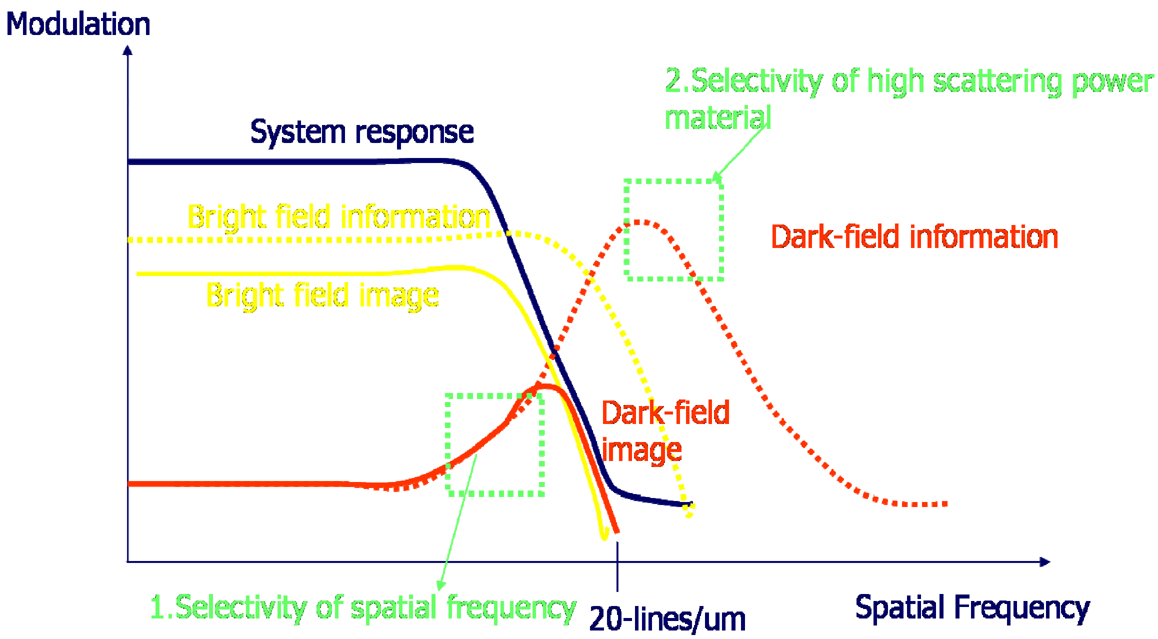

Fig. 3. The frequency response for the dark field and bright field. The solid blue line indicates the system response. The dotted yellow line and dotted red line indicate the input information from bright field and dark field respectively. The solid yellow line and solid red line show the frequency response of the bright field and dark field respectively. Notice that the dark field image has two major points. One is the selectivity of the high spatial frequency and the other one is the selectivity of the high scattering material.

The frequency response of the dark field has two major points. One is the selectivity of the high spatial frequency and the other one is the selectivity of the high scattering material. For the selectivity of the high spatial frequency, one can see the different response with spatial frequency, which is useful to distinguish the different size of objects. For the selectivity of the high scattering power material, it is mainly depends on the scattering power of the material, which is useful to distinguish the high Z and low Z material. In the following sections, the two arguments will be verified by experiments.

3. EXPERIMENT

3.1 The Siemens star

The Siemens star is used to test the frequency response of the dark field image. The Siemens star is made of 150nm thick gold electroplating on the silicon nitride membrane. The half pitch period of inner most zones is 30nm. The result is shown as Fig. 4. In Fig.4 (a) is the bright field of the Siemens star, there are two red circles indicates the half pitch period of 30nm and 50nm respectively. The image shows the absorption contrast with reasonable uniform contrast after the flat field process. In Fig.4 (b) is the dark field image of the Siemens star. The area on the sample is almost the same with the image of bright field. The image is brighter in horizontal than vertical because that the characteristic of source. The image of dark field is not processed by the flat field, so that some defects on the scintillator and uniformed illumination are observed.

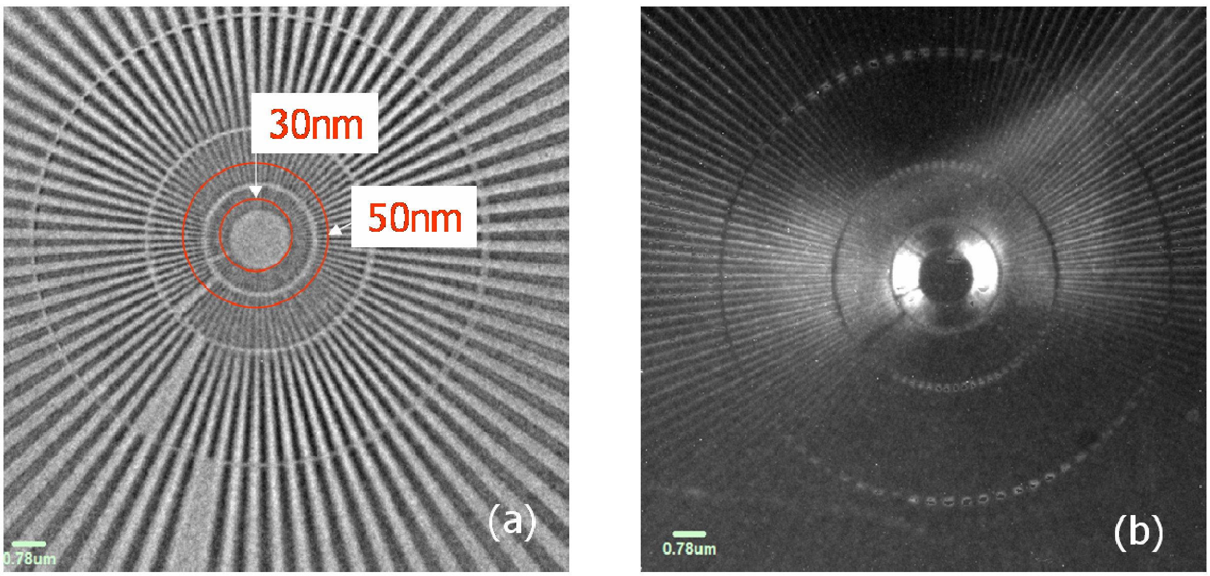

Fig. 4. The image of a siemens star in (a) bright field and (b) dark-field. The half pitch period of most inner part is 30nm, as indicated in the inner red circle. The outer circle indicates the half pitch period of 50nm. The dark field image is able to be recognized in 50nm and completed smelled out at 30~50nm region.

The dark field image shows the edge of the bright field, which is good verification that high frequency is enhanced and low frequency is suppressed. The inner most part of the dark field image is swelled out- which means the resolution does not enhance in the dark field. The high spatial frequency signal becomes a white noise background.

In order to have better under standing of the frequency response, 2D Fourier transform is applied to the two images. The result is shown as Fig5. Fig.5(a) is the modulus of Fourier transform of the bright field image. The center is for zero frequency, the high frequency is away from the center. The picture shows a flat response with low frequency, with a hole in center, which is a discontinuity of frequency of Siemens's star because the low frequency part of the Siemens's star is not shown in the field of view. Fig 5(b) is the modulus of Fourier transform of the dark field image, which shows the higher response at high frequency. This is good verification of the argument mentioned in section 2.

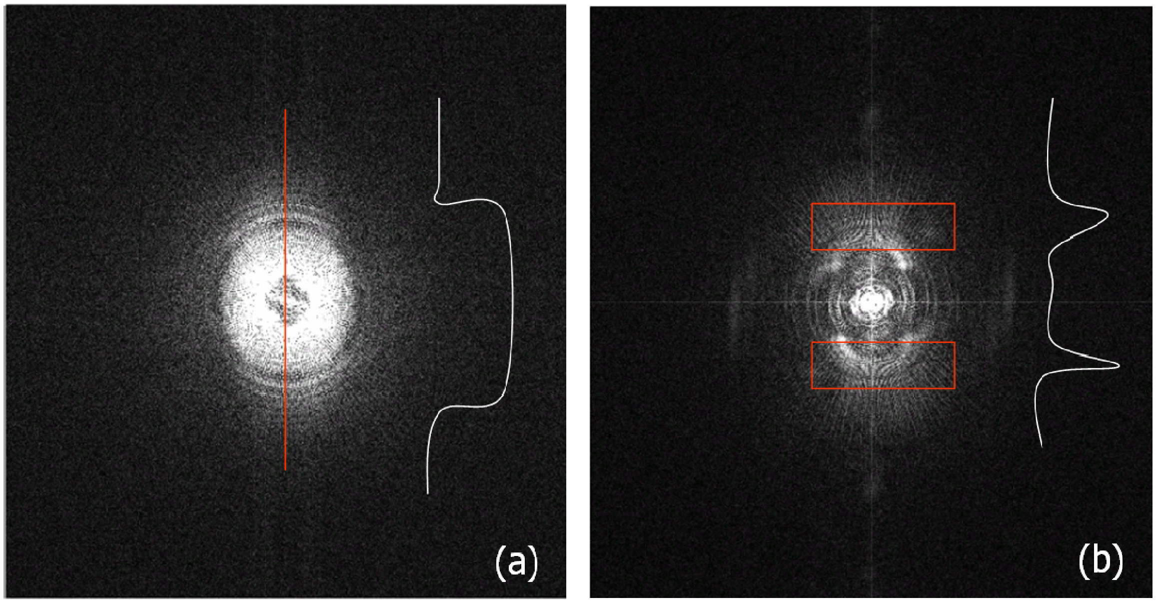

Fig. 5. A Siemens star in (a) Bright field and (right) Dark-field after 2D Fourier transform.

3.2 The used zone plate

The used zoneplate with collapse zone is used to demonstrate the selectivity of spatial resolution in the dark field image. This zoneplate is made of 900nm gold and out most zones width of 50nm. The imaged area is at the edge of the zoneplate. At the edge of the zoneplate, there are support zones outside the zoneplate. The support zones are for supporting and adjust the condition of electroplating. The width of support zones is thicker than that of out most zones. The contrast of support zones in bright field and dark field are obvious. In Fig 6(a), the bright field of the zoneplate, the support zones shows no contrast difference between the out most zones. However, in Fig 6(b), the dark field image shows a contrast difference between the support zones and the out most zones. The support zones in the dark field region become darker because that the support zone is wider than out most zone, which is the selectivity of the spatial frequency.

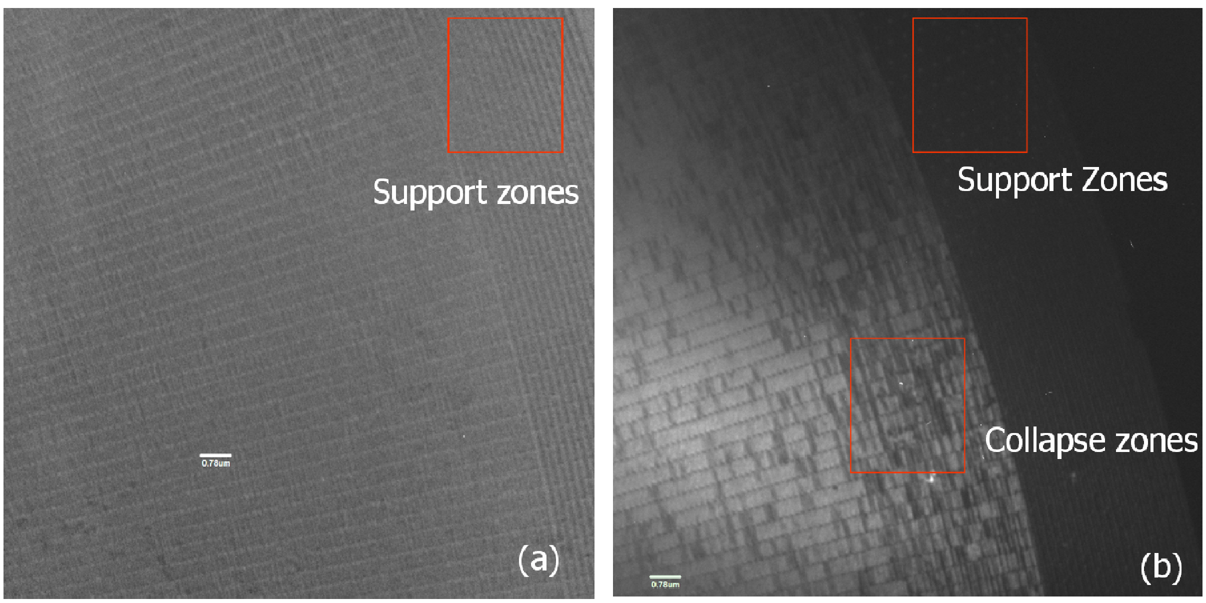

Fig. 6. The dark field of used zone plate in (a) Bright field and (b) Dark-field .

We take the used zone plate as the sample is because the collapse zone is a good demonstration for the dark field. The collapse zones, which means the structure of the zone plate is no longer at the position it should be, but falls on the other zones. Therefore, the effective width is larger while the zone is collapsed. Actually, the collapse zone does not vanish and still causes the absorption. This is the reason that bright field does not have a good contrast of the collapse zones. In Fig 6(b), the collapse zone is clearly shown by the dark area in the dark field and has no contrast in bright field.

3.3 The tungsten plug as the inter connection in memory chip

The tungsten plug is used to demonstrate the material selectivity. The memory chip contains multi-layer structures which are composed of tungsten, alloy of copper and alumni. In Fig. 7, we have demonstrated the high contrast ratio of tungsten plug. The alloy of copper and alumni is disappeared because the low scattering power at 10.5 keV. The material selectivity is shown.

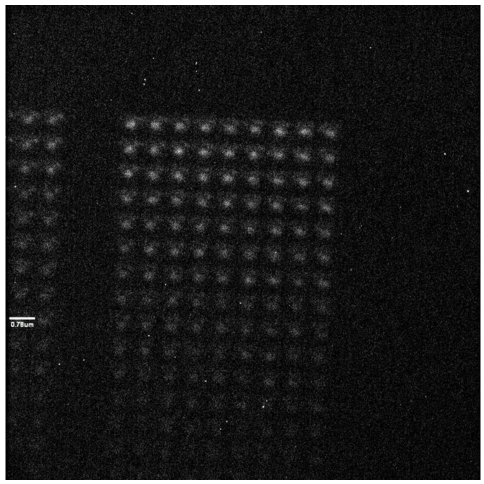

Fig. 7. The dark field image of a tungsten plug in the memory chip.

4. CONCLUSION

We have demonstrated the dark field in the TXM by high angle illumination condenser at photon energy from 8 to 11 KeV. The frequency response for dark field is analyzed and studied by images of Siemens's star. Two major functions of the dark field are found and estimated. One of the functions is the selectivity of spatial frequency which is verified by the used zone plate, in which the collapse zone is unclear in bright field but clearly observed in dark field mode. The other function is the selectivity of the material, which is verified by investigating the tungsten plug in memory chip- a high contrast of tungsten plug is obtained in the dark field image.

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