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CONSTRUCTION OF A ZONE PLATE IMAGING MICROSCOPE AND A ZONE PLATE PHOTOELECTRON SCANNING MICROSCOPE USING MONOCHROMATIZED CIRCULARLY POLARIZED SOFT X RAYS

Higher brilliance synchrotron radiation (SR) with circular polarization and Fresnel zone plates with an outermost zone width as small as a few hundred angströms have become available in recent years. This availability has made it possible to build a zone plate microscope having a sub-100 nm resolution and application in circular polarization.

This dissertation describes the design, construction and experiment of two types of Fresnel zone plate microscopes using circularly polarized soft x rays emitted from a helical undulator available at the beamline NE1B of the TRISTAN Accumulation Ring (AR) in National Laboratory for High Energy Physics *(KEK)*. One is a ***full-field transmission imaging microscope*** equipped with a visible light pre-focus unit aiming at studying the performance of an optical system using a fine zone plate. Another is a ***scanning photoelectron microscope*** aiming at detecting magnetic domains using circularly polarized soft x rays on the basis of magnetic circular dichroism.

Both microscopes need monochromatic illumination because of strong chromatic aberration of zone plates. Since a linear monochromator, which is popularly used in zone plate microscopes using SR from a bending magnet, does not necessarily match to SR from the undulator, a grazing incidence grating monochromator has been introduced and installed at the beamline NE1B to provide monochromatic illumination for two microscopes. It meets the monochromaticity requirement of the zone plates used in the microscopes. This arrangement may be suitable for a zone plate microscope combining with the next generation synchrotron radiation in the future which will provide a higher brilliance in a very low emittance ring.

The optical system of the imaging microscope consists of a ϕ500 μm pre-pinhole, a condenser zone plate (CZP), a ϕ20 μm pinhole, and an objective zone plate (OZP). The outermost zone width of the OZP is 50 nm. The pre-pinhole as a monochromatic secondary source is placed at the post-focused point of the beamline optics. The CZP generates a reduced image of the pre-pinhole onto an object through the ϕ20 μm pinhole. This object is imaged by the OZP with a magnification about 1000. An x-ray image is recorded by a photographic film or converted into a visible image by a microchannel plate (MCP). The working wavelength is chosen at 2.37 nm which is within "water window" region.

The object distance of the OZP acting as magnification is very short and close to its focal length (0.84 mm). Its focal depth is only $\pm$2.1 μm. The distance between the pinhole and an object should be as small as possible to obtain a proper illumination for the object. In addition, the sizes of the OZP (40 μm) and the pinhole (20 μm) are also small. Therefore, it is difficult to align the x-ray optical system precisely without any monitor and measurement implement. For this reason a visible light pre-focus unit has been developed and introduced into the x-ray microscope. It mainly consists of a coarse adjustment objective which is perpendicular to the x-ray axis and a fine adjustment objective which is on the x-ray axis. There is a ϕ2 mm hole on the centre of the fine adjustment objective to make x rays pass through. The focal depth of the fine adjustment objective is designed to be about ± 1.5 μm (shorter than the focal depth of the OZP). Its working distance is designed as long as 10 mm. Therefore, the distance between the pinhole and an object and the object distance of the OZP can be measured and adjusted quickly, easily and precisely with the pre-focus unit. Such a visible light pre-focus unit will be indispensable for a high resolution zone plate microscope.

The imaging properties of this microscope have been evaluated numerically by diffraction limit resolution and modulation transfer function (MTF) on the basis of diffraction theory. The resolution is expected to be about 50 ~ 60 nm. On the other hand, an experimental resolution has reached 55 nm by imaging a fine zone plate in a resolution test, which is in good agreement with the result of numeral evaluation. It suggests that the resolution of the microscope has almost reached its diffraction limit. The numeral evaluation also shows that monochromaticity of illumination has almost no influence on resolution. However, MTF deteriorates with the monochromaticity becomes poor at the same spatial frequency. Furthermore, a MTF curve obtained by an illumination with a relatively wide bandwidth will drop more dramatically than the one obtained by that with a relatively narrow bandwidth along with increment of spatial frequency.

In addition, some dry biological specimens have also been observed by the microscope in spite of a poor contrast due to a low photon flux. That implies that our microscope has a potentiality of observing biological specimens.

The optical system of the scanning photoelectron microscope consists of a pre-pinhole a zone plate with a central stop (D=160 μm, r_1=5 μm and D_{stop}=33 μm), and a ϕ25 μm pinhole. The pre-pinhole acts as the same way as that in the imaging microscope. The zone plate generates a reduced image of the pre-pinhole as a microprobe which irradiates a sample at a grazing incident angle of 30°. Photoelectron yield emitted from the sample is counted by a channel electron multiplier. The sample is scanned across the microprobe two-dimensionally with a scanning system consisting of a two-dimensional coarse stage driven by pulse motors with a scan step of 0.5 μm/pulse and a two-dimensional fine stage driven by piezoelectric transducers with a resolution of 10 nm. The resolution of the scanning microscope has been evaluated to be about

1.2 μm by analyzing an image of an edge of a copper mesh (#2000), which is in good agreement with the ideal resolution of 0.9 μm.

Magnetic domains with dimensions of 20 μm recorded on a piece of a video tape have been imaged at the $L_{2,3}$ absorption edges of cobalt by the scanning microscope using right circularly polarized soft x rays. The magnetic layer of the tape is formed by evaporation with its component of Co:Ni=80:20 which is overcoated with 200 Å-thick CoO and a tens Å-thick organic lubricant. The contrast in the images arises from that x-ray absorption mainly depends on the relative orientation of the helicity of photon and magnetization direction of the sample, i. e., magnetic circular dichroism (MCD). The observed pattern exactly corresponds to the recorded one. The results demonstrate that magnetic domains can be imaged even in a buried layer by detecting secondary photoelectron yield using the scanning microscope combining with MCD. It is also found the contrast in an image taken at 793 eV (the Co-L_2 edge) is inverse in comparison with the contrast in an image taken at 778 eV (the Co-L_3 edge). It is in agreement with the Co-$L_{2,3}$ MCD spectrum. As a result, the contrast can be enhanced by subtracting images obtained at the L_3 and L_2 edges. On the other hand, the images taken at 770 eV (below the Co-L_3 edge) and 803 eV (above the Co-L_2 edge) do not exhibit magnetic contrast. It suggests that the magnetic contrast will take place at the characteristic absorption edge. Therefore, such an observation of magnetic domains is characterized by elemental specificity.

論文の審査結果の要旨

王　李東氏の論文は、高エネルギー物理学研究所のトリスタン入射蓄積リングのヘリカルアンジュレータ（NE1B）からの高輝度の円偏光軟X線が利用できることに着目し、また近年数10nmの最外輪帯幅の高性能なフレネルゾーンプレートが軟X線光学素子として使用できるようになったことから、生物試料の直接的観察を目標とした数10nmの高い空間分解能を持つ結像型軟X線顕微鏡、および磁性材料の評価に貴重な情報を与えると考えられている円偏光軟X線を用いた走査型の光電子顕微鏡の開発研究を行ったものである。

結像型軟X線顕微鏡の開発研究の主要部は、これまで非常に困難とされてきたピンホールと対物ゾーンプレート、試料の高精度な光軸調整（数μm）を、軟X線顕微鏡内に組み込まれた可視光を用いた光軸調整ユニットを設計製作することにより、短時間に効率的にしかもX線照射による試料の損傷無しに実現する方法を考案したことにある。従来このような光軸調整は、研究者の経験にたより、しかも実際にX線を試料に照射しながら行っていた。軟X線顕微鏡自体の開発目標が生きたままの生物試料の観察にある点を考えると、このような装置と方法論の開発は、この分野の発展にとって重要な寄与であると考えられる。本軟X線顕微鏡の結像特性を実験的に検証し、分解能が55nmであり理論値にほぼ一致していることが確かめられた。この分解能は世界最高水準にある。また本軟X線顕微鏡のもう一つの特徴は、アンデュレーター光を回折格子で分光した単色光を用いている点にあるが、本論文では入射X線の単色性の結像に対する影響を数値計算により考察し、入射X線の単色性の重要性を指摘している。さらに本装置を用い、生物試料の観察を行い、実用化の可能性があることを示した。

光電子顕微鏡の開発においては、ゾーンプレートにより最小ビームサイズ1.2μmの軟X線マイクロビームを得、試料を走査することにより、２次元像を得る方法を開発した。特にアンジュレータからの円偏光軟X線を用いて、$CoL_{2,3}$吸収端の磁気円２色性（MCD）を利用することにより、ビデオテープに記録された磁区の観察に成功した。円偏光アンジュレータを用いた走査型の光電子顕微鏡は世界的にも初めての試みであり、軟X線マイクロビームの微小化とともに円偏光光電子顕微鏡の有力な手法となると考えられる。

以上のように、本論文では、結像型軟X線顕微鏡を開発し、その高精度な光軸調整方法に新しい手法を導入し、また結像特性に関する検討も行っている。さらに光電子顕微鏡の開発において、円偏光軟X線によるMCDを利用した走査型光電子顕微鏡が有力な手法であることを示した。本研究は、軟X線顕微鏡の今後の研究開発に資するところが大きいと考えられる。以上の研究成果は博士学位論文としての内容に値すると判断した。