Determination of focal spot size of high-energy microfocal X-ray source based on HfO$_2$-coated single-bounce ellipsoidal glass monocapillary X-ray condenser

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Abstract. A method based on the single-bounce ellipsoidal monocapillary X-ray condenser (SEGMXC) was improved for determining the focal spot size of the high-energy microfocal X-ray source. HfO$_2$ was selected as the high-density material ($\approx$9.68g/cm$^3$) and coated on the inner surface of the SEGMXC by the atomic layer deposition method. The focal spot size of a microfocal spot X-ray source was obtained as $33.14 \pm 0.02$ μm and the relative deviation of the effective size given by the manufacturer was 5.3%.

1. Introduction

X-ray sources play an important role in the science of material analysis [1-2]. Although high-energy X-ray source are available [3-6], the actual dimensions of many focal spots are larger than the nominal sizes given by the manufacturers [7]. Therefore, the accurate determination of the focal spot size of the X-ray source is vital for the design and application of X-ray sources [8-9]. The pinhole method is a traditional method of measuring the focal spot size of the X-ray source [7,10]. However, the pinhole method requires multiple shots and large magnification. In addition, due to the influence of the penumbra, the pinhole should be smaller than the focal spot size and that increases the requirement of the manufacture [10-13], which makes this method has no advantage on the measurement of the microfocal spot X-ray source. The slit and resolution pattern method are other two common methods for determining the size of the focal spot. The scanner is not required in these methods and the exposure times are shorter. However, the slit method requires a spread function of the slit and the resolution pattern method requires low contrast measurement [10-14]. The size of the focused beam of the polycapillary X-ray collimator is related to the size of the focal spot of the X-ray source. The polycapillary X-ray collimator, therefore, can be used in measuring the focal spot size, especially for the microfocal spot X-ray source [15-16]. The focal spot size and the focal depth can be obtained simultaneously by the single-bounce ellipsoidal monocapillary X-ray condenser (SEGMXC) [17]. However, it is hard to manufacture the SEGMXC into an ideal ellipsoidal shape. Duo to the penetration of the high-energy X-ray, none of the above methods are suitable for measuring the focal spot size of the high-energy X-ray source.

According to the principle of the total reflection of the X-ray, the critical angle of the total reflection of the X-ray is proportional to the density of the reflector [18]. Therefore, to determine the focal spot size of the high energy X-ray source. The atomic layer deposition (ALD) was used to deposit the high-
density material on the inner surface of the SEGCMXC. Due high conformality and good chemical control of ALD, it is widely used for the deposition of protective on miniaturized, highly reflective surfaces [19-22]. Compared with the electrophores deposition method, ALD implies a lower roughness. Therefore, ALD has the advantage in depositing high-density materials on the inner surface of the SEGCMXC.

In this paper, the method based on the SEGCMXC was improved for determining the focal spot size of the high energy microfocal X-ray source. HfO₂ was selected as the high-density material (~9.68g/cm³) and coated on the inner surface of the SEGCMXC by the ALD method. The focal spot size of a microfocal spot X-ray source was obtained through this method.

2. Theoretical basis
The SEGCMXC is commonly used as a condenser device of X-rays, the incident X-rays undergo repeated total external reflection until they exit out of the SEGCMXC [23]. Total external reflection can occur as long as the grazing angle  \( \theta \) is not greater than the critical angle of total reflection (figure 1) and the critical angle of total reflection \( \theta_c \) can be estimated as follows:

\[
\theta_c = \frac{20.3 \sqrt{\rho}}{E_i(\text{keV})} \text{(mrad)}
\]

where \( E_i \) is energy of the incident X-ray, and \( \rho \) is density of the reflector.

The HfO₂ film was coated on the inner surface of the SEGCMXC, according to Eq. (1), \( \theta_c \) was increased by 2.1 times (the density of the glass is ~2.23 g/cm³), which meant that the highest energy that the SEGCMXC could focus was increased by 2.1 times. Generally, the glass capillary X-ray condenser can focus the X-ray with energy below 35 keV [24]. Therefore, the HfO₂-coated SEGCMXC can focus the X-ray with energy up to 74 keV.

The X-ray source \( S \) is positioned at the focus of ellipsoid and the image \( I \) of the focused X-ray by the SEGCMXC is at the other focus of the ellipsoid (figure 1).

The relation between the size of the X-ray source \( Z_s \) and the image size \( Z_i \) is as follows [24]:

\[
Z_i = C \left( \frac{L + L_s}{F} \right) Z_s + 2(L + \frac{L_s}{2}) \alpha
\]

where \( C \) is a correction factor and \( \alpha \) is the slope error of the SEGCMXC. These two parameters can be measured based on an X-ray source with a variable focal spot size [24]. Therefore, Eq. (2) can be reduced to the following form:

\[
Z_s = \frac{F}{C(L + L_s)} Z_i - 2(L + \frac{L_s}{2}) \frac{F}{(L + L_s)} \alpha
\]
Here $Z_i$ can be obtained by the knife-edge scanning method, and for a certain SBEGMXC, Eq. (3) is a linear function. $F \frac{L}{C(L+L_s)}$ can be considered as the proportionality coefficient $K$, while $-2(L + \frac{L_s}{2}) \frac{F}{(L+L_s)} \alpha$ can be considered as the intercept $B$. Therefore, Eq. (3) can be reduced to

$$Z_s = KZ_i + B$$

(4)

Thus, the value of $Z_s$ can be obtained by measurement and calculation.

3. Experimental

3.1. HfO$_2$-coated SBEGMXC

A commercial Ke-Micro T-ALD 100A setup was used for HfO$_2$ depositing, Hf(N(CH$_3$)$_2$)$_4$ was selected as the hafnium precursor, deionized water as the water precursor and the purity nitrogen with a flow rate of 14 standard cubic centimeters per minute (sccm) as the carrier and purging gas.

The parameters of one ALD cycle are listed in Table 1:

| Parameters of one ALD cycle | Pulse time of Hf(N(CH$_3$)$_2$)$_4$ (s) | First purging time (s) | Pulse time of H$_2$O (s) | Second purging time (s) | Reaction temperature (°C) | Deposition pressure (torr) |
|-----------------------------|----------------------------------------|------------------------|--------------------------|---------------------------|---------------------------|---------------------------|
| 0.13                        | 50.00                                  | 0.02                   | 50.00                    | 250                       | 0.1                       |

1700 ALD cycles were set to deposit HfO$_2$ on the inner surface of the SBEGMXC.

In this paper, a SBEGMXC was fabricated by drawing tower method [25-26], and the SBEGMXC was coated before it was cut into the design length. After coating, the SBEGMXC was cut into the design length, and the scanning electron microscope (SME) method was used to obtain the thickness of HfO$_2$ film at the cut section (figure 2).

![Figure 2. SEM image of the cut section of the HfO$_2$-coated SBEGMXC.](image-url)
According to the results of SME, the HfO$_2$ film thickness is about 34 nm, and X-rays did not penetrate beyond this thickness outside the capillary wall [27].

The parameters of the HfO$_2$-coated SBEGMXC (figure 3) are listed in table 2.

**Table 2. Parameters of SBEGMXC.**

| SBEGMXC         |                |
|-----------------|----------------|
| Effective length (mm) | 50.000         |
| Input radius (mm)   | 0.380          |
| Output radius (mm)  | 0.491          |
| Major semi-axis (mm) | 500.000  |
| Minor semi-axis (mm) | 0.600    |

![Figure 3. Photo of SBEGMXC.](image)

3.2. Determination of C and $\alpha$

In this paper, a microfocus X-ray source with a W target (L9631 HAMAMATSU, Japan) and a high-energy X-ray detector system (X-123 CdTe AMETEK, USA) were used to obtain $C$ and $\alpha$ in Eq.(2) [21]. The focal depth of this X-ray source was 16.5 mm, and the focal spot size of this X-ray source was continuously adjusted from 15 to 80 $\mu$m by changing its output power (figure 4).

![Figure 4. The relation between the output power and the focal spot size of the X-ray source.](image)
The voltage of the X-ray source was set as 65 kV and the current was changed to obtain the different focal spot size. The $C$ and $\alpha$ were obtained as 1.2 and 53.7 $\mu$rad, respectively.

### 3.3. Experimental setup

An X-ray source with a W target (MAX-ULI-L-8-2-10-W, TRUFOCUS, USA) was used as a sample of the measurement. The focal depth $F_d$ is given as 15.7 mm and the effective size given by the manufacturer as 35 $\mu$m. The voltage of the X-ray source was set as 65 kV and the current was set as 10 mA. The high energy X-ray detector system (X-123 CdTe AMETEK, USA) was used as the detector. The X-ray source, the HfO$_2$-coated SBEGMXC and the detector was positioned on the same axis (figure 5). A lead knife edge (2 cm in thickness) was used to scan the focal spot of the SBEGMXC.

![Figure 5. Sketch of the experiment.](image)

$F_b$ is the distance between the beryllium window and the entrance of the SBDGMXC.

4. Results

The counts of $K_\alpha$ characteristic line (58.0keV) of the W target was selected to obtain $Z_i$ by knife edge scanning method and took the FWHM of the scanning curve as the size of the high energy focal spot. In this experiment, $Z_i$ was measured by from direction (figure 6) and the average value of $Z_i$ was $309.1 \pm 0.2 \mu$m.

![Figure 6. Sketch of the knife-edge scanning directions.](image)
Other parameters are listed in Table 3.

**Table 3. Parameters obtained by the experiment.**

| Parameters | Value  |
|------------|--------|
| L (mm)     | 393.0  |
| L₀ (mm)    | 50.0   |
| F₆ (mm)    | 41.3   |
| F₄ (mm)    | 15.7   |

According to the experimental results and Eq. (4), Zᵢ was obtained as $33.14 ± 0.02 \mu m$. The relative deviation of the effective size from the one given by the manufacturer was 5.3%. This discrepancy may be due to deviation of the measurement of the slope error of the SBEGMXC and the error of knife-edge scanning, as well as the deviation of the optical alignment. We also used the knife-edge scanning method [7] to measure the focal spot size of the X-ray source, which was assessed as $46.24 ± 0.02 \mu m$. The relative deviation of the effective size from the one given by the manufacturer was 32.11%.

5. Summary

The ALD method can efficiently deposit the high-density material and improve the SBEGMXC to focus higher energy. The HfO₂-coated SBEGMXC can obtain the high-energy microfocal spot size of the X-ray source effectively. The comparative analysis of the results obtained via the HfO₂-coated SBEGMXC method and the knife-edge scanning method proved the superiority of the former method. The difficulties of measurement caused by X-ray penetration are effectively mitigated by the HfO₂-coated SBEGMXC method, while the SBEGMXC shape is not constrained by the X-ray source size. This method has potential value in the fabrication and calibration of high-energy microfocal X-ray sources.

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