Paper

Measurement of Complex Refractive Index of Tungsten by Using Ellipsometry

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In development of tungsten microcavity illuminants and tungsten cluster illuminants, the Complex Reractive Index of tungsten at the operating temperature of 2000-6000 K is necessary to evaluate their luminous efficacy and lifetime. Little is, however, known about the complex index of tungsten in such a high temperature region.

In this study, we have determined the complex index of tungsten using an ellipsometer at room temperature. In measurement, a He-Ne laser of 633 nm and a Nd:YAG laser of 532 nm were used as the light sources. The measured complex index of tungsten was 2.0 to 3.0 in real part and 2.5 to 3.0 in imaginary part at 633 nm. Also, the corresponding components at 532 nm were 1.8 to 2.8 and 2.4 to 2.8. Repeatable accuracy was estimated about 1 % in measuring the complex index repeatedly at the same point on individual tungsten plates. It was, however, found that the measured complex index was linearly proportional to the reflectivity. This dependence is attributed to surface conditions such as oxidation and roughness on a microscopic scale.

Keywords : microcavity illuminant, cluster illuminant, tungsten, complex refractive index, Ellipsometry

1. Introduction

Tungsten microcavity illuminants and tungsten cluster illuminants have recently attracted interest as incandescent sources in the next generation. The tungsten microcavity illuminant has a radiator on which sub-micron cavities are perforated(1). By a quantum effect, the small cavities forbid the radiation of longer wavelength than the cutoff wavelength which is determined by the cavity size. For higher luminous efficacy, the cutoff wavelength should be set at 700 nm to reduce the infrared radiation. The luminous efficacy of the tungsten microcavity illuminant is predicted to be about 100 lm/W from a theoretical analysis when operating temperature is 2000 K and the cutoff wavelength is 700 nm(2). Regarding the tungsten cluster illuminant, the tungsten clusters, which are produced at extremely high temperature, act as a radiator(3). A sealed light tube with a tungsten compound enclosed is put into a microwave resonator. By the microwave excitation, the tungsten clusters can be yielded and heated over 4000 K. The luminous efficacy of the tungsten cluster illuminant is expected to be very high because visible light is dominant in thermal radiation at extremely high temperature of exceeding 4000 K and small clusters reduce the infrared radiation by the quantum effect. Also, the lifetime is very long because any filament is unnecessary in this illuminant.

2. Ellipsometry

Fig.1 shows a schematic diagram of ellipsometry. The ellipsometry is based on the phenomenon that the complex amplitude reflectivities of p and s polarized lights are dependent on the complex index of metal to be measured when the linearly-polarized laser beam obliquely incidents to the surface. Since the ellipsometry is a non-contact measurement technique, the complex indices of any metals can be measured even under extremely high temperature with high accuracy.

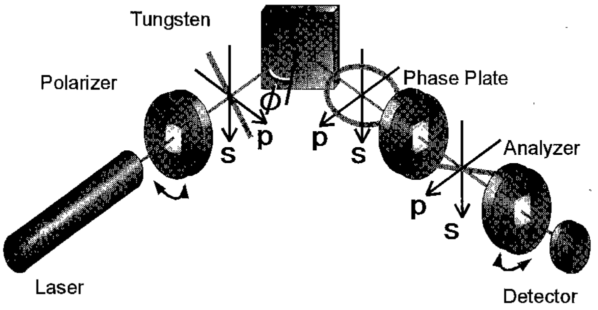

Fig. 1. Schematic diagram of Ellipsometry

The amplitude and phase of the reflected light from the metal surface are expressed by the Fresnel formulae(4). The p and s polarized amplitude components χ_{pi}, χ_{si} of the incident light and the corresponding components χ_{pr}, χ_{sr} of the reflected light are related by

$$\left.\begin{aligned} \chi_{pr} &= \frac{\tan(\phi-\gamma)}{\tan(\phi+\gamma)}\cdot\chi_{pi} \\ \chi_{sr} &= -\frac{\sin(\phi-\gamma)}{\sin(\phi+\gamma)}\cdot\chi_{si} \end{aligned}\right\} \cdots\cdots\cdots\cdots (1)$$

where ϕ is the angle of incidence and γ is the angle of refraction. γ is a complex number, depending on the complex index of the metal. Therefore, the ratios χ_{pr}/χ_{pi} and χ_{sr}/χ_{si} must be complex from Eq. (1). This means that the linearly polarized incident light in general changes into the elliptically polarized light after the reflection at the metal surface. The ratios are written as

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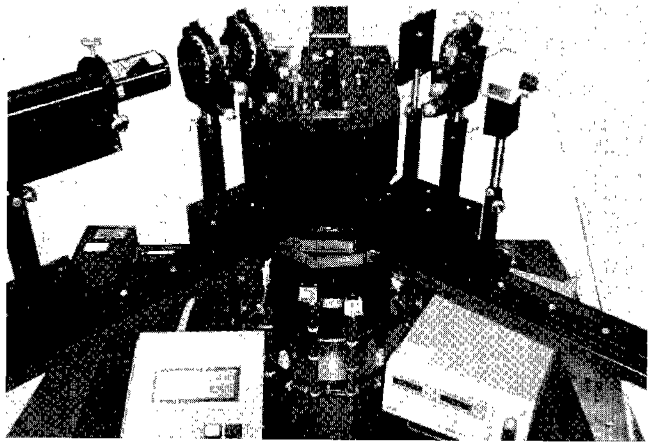

Fig. 2. Ellipsometer built for this study

$$\left.\begin{aligned} R_p &= \frac{\chi_{pr}}{\chi_{pi}} = r_p \cdot \exp(j\delta_p) \\ R_s &= \frac{\chi_{sr}}{\chi_{si}} = r_s \cdot \exp(j\delta_s) \end{aligned}\right\} \cdots\cdots\cdots (2)$$

where δ_p and δ_s denote the phase changes, and r_p and r_s the absolute values of the amplitude reflectivities. If $\chi_{si}/\chi_{pi}=1$, that is, the incident light is linearly polarized at the azimuthal angle of 45°, the polarization state of the reflected light is given as

$$\begin{aligned} \frac{\chi_{sr}}{\chi_{pr}} &= -\frac{\cos(\phi-\gamma)}{\cos(\phi+\gamma)} \\ &= \frac{r_s}{r_p} \cdot \exp\{j(\delta_s-\delta_p)\} \\ &= \tan^{-1}\Psi \cdot \exp(-j2\Delta). \cdots\cdots\cdots (3) \end{aligned}$$

The amplitude ratio r_s / r_p and the phase difference δ_s - δ_p are both a function of the complex index. Ψ and Δ are the important measures, corresponding to the amplitude ratio and the phase difference, in the ellipsometry. Using Ψ and Δ, the real part n and the imaginary part $n\kappa$ of the complex index are expressed as

$$\left.\begin{aligned} n &= \frac{n_0 \sin\phi \tan\phi \cos 2\Psi}{1+\sin 2\Psi \cos 2\Delta} \\ n\kappa &= \frac{n_0 \sin\phi \tan\phi \sin 2\Psi \sin 2\Delta}{1+\sin 2\Psi \cos 2\Delta} \end{aligned}\right\} \cdots\cdots\cdots (4)$$

where n_0 is the refractive index of air. Therefore, if the values Ψ and Δ are obtained at some angle of incidences by the ellipsometry, the complex index can be determined by Eq.(4).

Fig.2 is a photograph of the ellipsometer built for this study. A polarizer, an analyzer and a phase plate have an accuracy of 1 second to read the azimuth angle. The angle of incidence is controlled with an accuracy of 0.01 second by a servomotor.

3. Discussions

3.1 Measurment Results In this study, the Complex Reractive Index of tungsten was measured at room temperature using a He-Ne laser of 633 nm and a Nd:YAG laser of 532 nm as the light sources. The angle of incidence was determined 50 ° from the results of the pre-measurement. In the measurement, room temperature and humidity were 22 ~ 24 °C and 60 ~ 70 %, respectively. Tables 1 and 2 show the measured results for nine tungsten plates, labeled as 'Sample 1', 'Sample 2', ... , and 'Sample 9', of 99.9 % in purity. Samples 6 through 9 were polished on the surfaces by the diamond board with the particle size of 0.3 μm.

Table 1. Measurement results at 633 nm

	n	$n\kappa$	R
Sample1	2.91	2.97	0.391
Sample2	2.65	2.86	0.376
Sample3	2.48	2.55	0.343
Sample4	2.37	2.76	0.371
Sample5	2.01	2.47	0.321
Sample6	2.93	2.65	0.436
Sample7	2.88	2.73	0.418
Sample8	2.50	2.57	0.416
Sample9	2.67	2.74	0.429

Table 2. Measurement results at 532 nm

	n	$n\kappa$	R
Sample1	2.68	2.74	0.371
Sample2	2.44	2.67	0.359
Sample3	2.34	2.32	0.319
Sample4	2.08	2.51	0.351
Sample5	1.83	2.29	0.306
Sample6	2.74	2.52	0.420
Sample7	2.69	2.56	0.400
Sample8	2.30	2.41	0.399
Sample9	2.31	2.59	0.417

The gray-colored columns in the Tables 1 and 2 correspond to the polished tungsten plates. R is the reflectivity of the tungsten plate in the normal incidence. The measured Complex Reractive Index as a function of the reflectivity will be discussed in Section 3.2. Moreover, a dependence of the complex index on the wavelength will be also discussed in Section 3.3.

3.2 Reflectivity The measured complex index versus the reflectivity is shown in Figs.3 through 6. The complex index depends on sample. It is found from Figs.3 through 6 that the measured complex index is linearly proportional to the reflectivity. The relationship between the reflectivity and the complex index is generally expressed as

$$R = \frac{(n-1)^2+(n\kappa)^2}{(n+1)^2+(n\kappa)^2}. \cdots\cdots\cdots (5)$$

This equation shows that each part of the complex index increases with the reflectivity, as shown in Figs.3 through 6. Reflectivity is also known to be affected by the surface conditions. So, it is

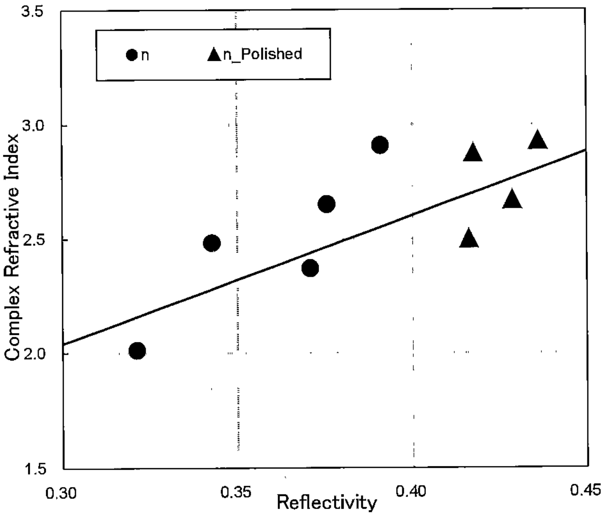

Fig. 3. $R-n$ at 633 nm

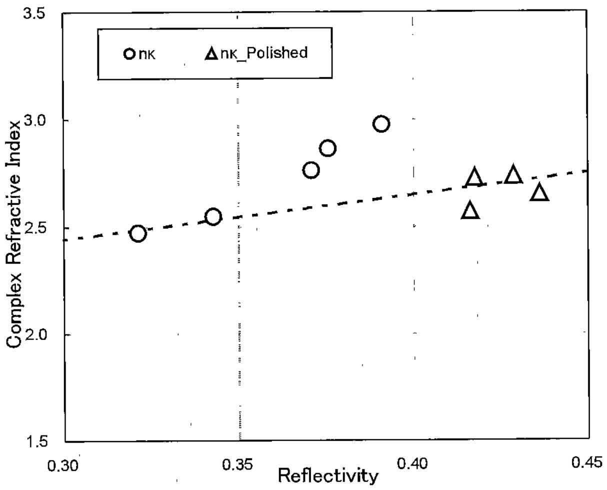

Fig. 4. R - $n\kappa$ at 633 nm

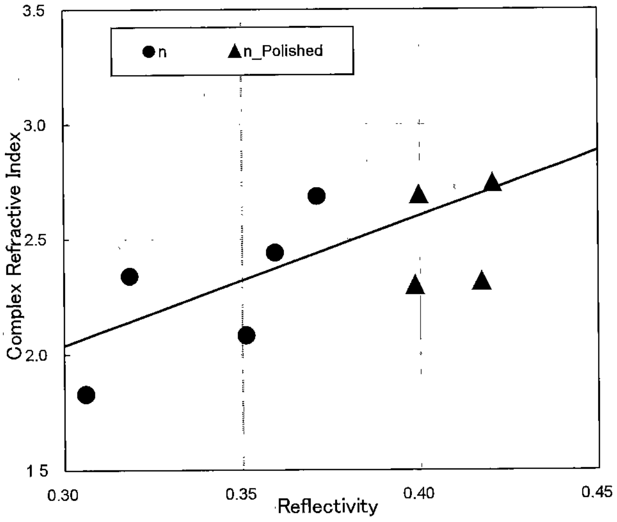

Fig. 5. R - n at 532 nm

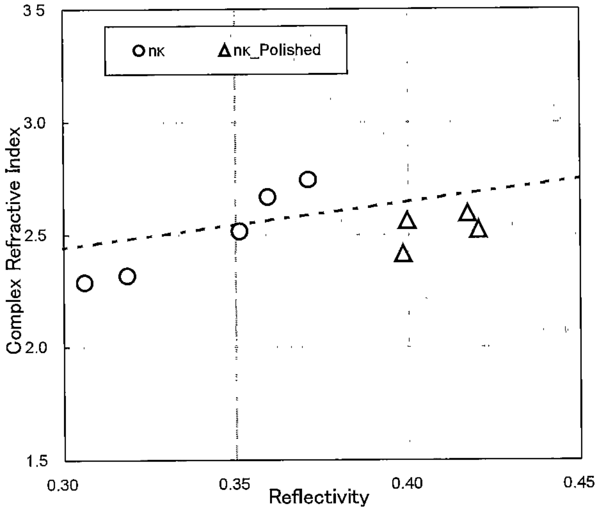

Fig. 6. $R-n$ at 532 nm

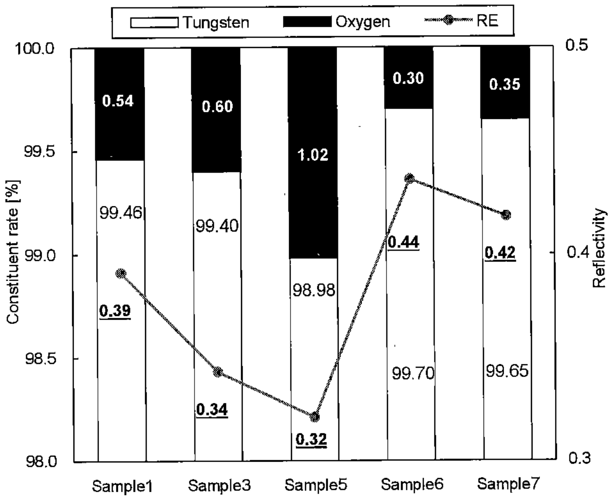

Fig. 7. Constituent rate of Tungsten

worthwhile to examine the relationship between reflectivity and surface conditions.

The constituents of each sample were measured from the surface to 2 μm deep by EPMA. The results of Samples 1, 3, 5, 6, and 7 are shown in Fig.7. In this figure, the strong correlation is seen between the amount of oxygen and the reflectivity. Samples with much amount of oxygen would be covered with the slightly oxidized layer on the surface. Since the oxidized layer has low refractive index in many cases, the decrease of the reflectivity in the oxygen-rich samples is reasonably explained by Eq.(5). For precise measurement, the oxidized layer should be removed. Moreover, the surfaces of Samples 1, 3, 5, 6, and 7 were also observed by SEM. Those photographs are shown in Figs.8 through 12. Samples with low reflectivity had rougher surfaces, on which minute particles and scratches adhered. The minute particles and scratches cause light scattering which reduces the reflectivity. The light scattering is undesirable for the ellipsometry because the effect of the scattering is not considered in Eqs.(1) through (4). Therefore, fairly smooth surface is required to realize precise measurement of the complex index.

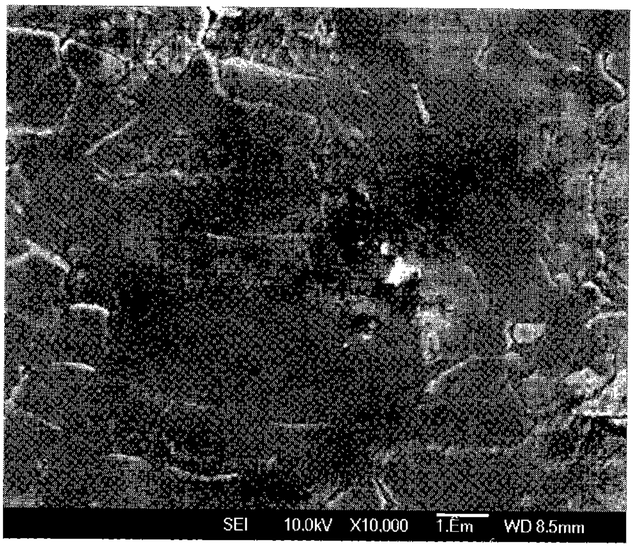

Fig. 8. Surface of sample 1

Fig. 9. Surface of sample 3

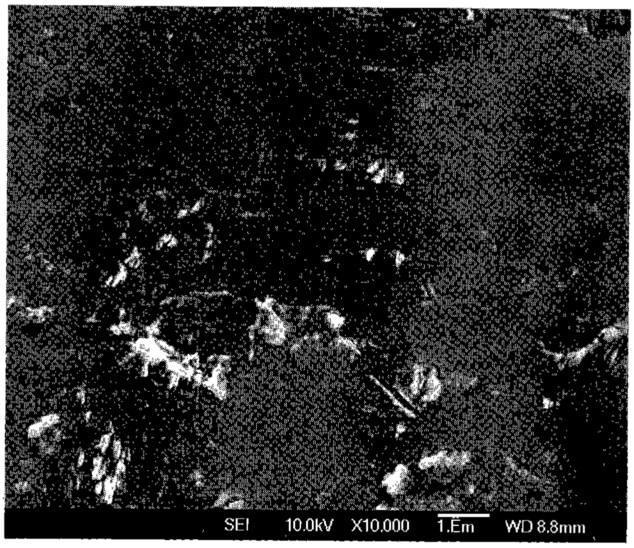

Fig. 10. Surface of sample 5

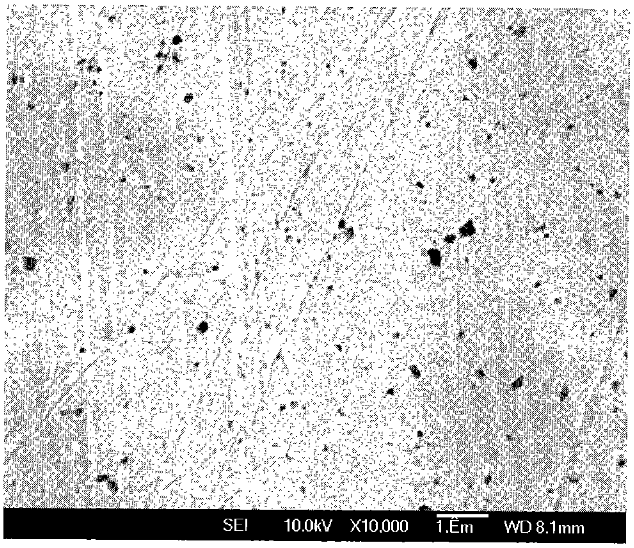

Fig. 11. Surface of sample 6

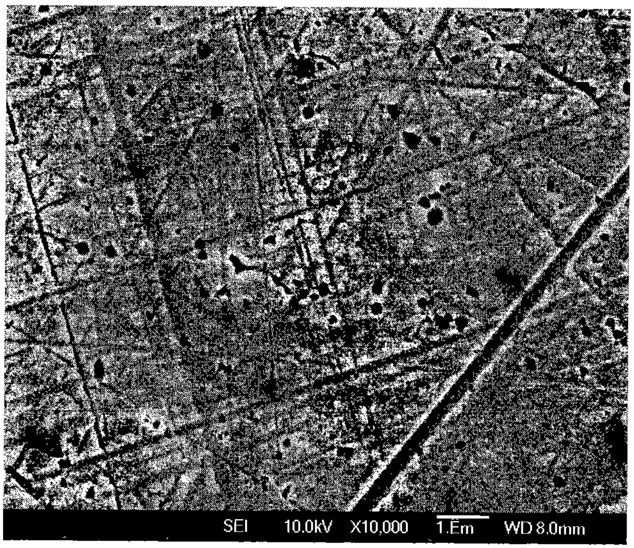

Fig. 12. Surface of sample 7

3.3 Accuracy An accuracy of the measurement considerably affects the evaluation of the luminous efficacy and lifetime of the microcavity illuminant and the cluster illuminant. According to the theoretical consideration, the evaluation is sufficiently reliable if the accuracy is within 3 %. In this section, the accuracy of the measurement is discussed. As discussed in Section 3.2, the measured complex index was dependent on the reflectivity because of the surface conditions such as oxidation and roughness. To avoid the difficulties by such undesirable dependence between the reflectivity and the surface condition, the repeatable accuracy at the same point on the sample was introduced here to evaluate the accuracy of the measurement. Tables 3 and 4 show the repeatable accuracy at 633 nm and 532 nm, respectively. The average of the accuracy is less than 1 %, not depending on the wavelength. Therefore, it would be possible to obtain the precise complex index by the ellipsometry if any tungsten plate with fairly smooth surface can be employed.

Table 3. Repeatable accuracy by the ellipsometer at 633 nm

	n	$n\kappa$	Average
Sample 1	0.38	1.63	1.01
Sample 2	0.93	1.29	1.11
Sample 3	2.26	0.16	1.21
Sample 4	0.45	0.37	0.41
Sample 5	1.80	1.29	1.55
Sample 6	0.49	0.61	0.55
Sample 7	0.26	1.26	0.76
Sample 8	1.25	0.43	0.84
Sample 9	0.43	1.07	0.75
Average	0.85	0.93	0.89

Table 4. Repeatable accuracy by the ellipsometer at 532 nm

	n	$n\kappa$	Average
Sample 1	0.02	0.70	0.36
Sample 2	0.79	0.38	0.59
Sample 3	2.54	0.15	1.35
Sample 4	2.20	1.63	1.92
Sample 5	2.16	2.08	2.12
Sample 6	0.38	1.01	0.70
Sample 7	0.04	0.18	0.11
Sample 8	1.20	0.75	0.98
Sample 9	0.83	0.15	0.49
Average	1.13	0.78	0.96

3.4 Dependence on Wavelength

Fig.13 shows the dependence of the complex index on the wavelength. Littleton measured the complex index of tungsten at 589.3 nm by the ellipsometry in 1912(4). The complex index n and $n\kappa$ by Littleton are plotted with ▲ and △ in Fig.13, respectively. J. H. Weaver also measured the complex index between 26.4 nm and 24.8 μm using a synchrotron radiation(5). The values of n and $n\kappa$ by Weaver are indicated with × and + in Fig.13, respectively. The circles ● and ○ represent n and $n\kappa$ of Sample 6, of which the reflectivity was highest in the nine samples. The values obtained in this study are smaller than those by Littleton and Weaver. It is because the reflectivity was different from that of their samples. Regarding the wavelength dependence of reflectivity, the complex index at 633 nm is larger than that at 532 nm, by 6.5 % for real part and by 4.9 % for imaginary part. This tendency is very similar to Weaver's.

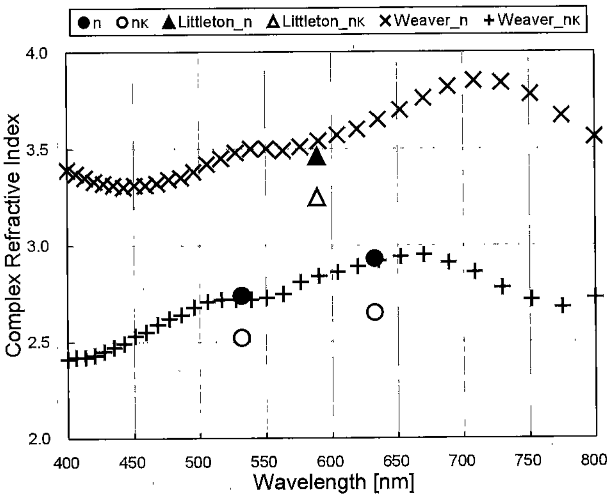

Fig. 13. Wavelength dependence of the complex refractive index of Tungsten

4. Conclusions

In this study, we measured the Complex Reractive Index of tungsten at room temperature. The measured complex index was dependent on sample. It is attributed by the surface conditions such as oxidation and roughness. So, the repeatable accuracy, independent of the surface condition, was introduced to evaluate the accuracy of the measurement. The average of the repeatable accuracy was less than 1 %. This accuracy is sufficient to determine the luminous efficacy and lifetime of the microcavity illuminant and the cluster illuminant. Moreover, the accuracy is independent of the wavelength.

The ellipsometry has a good potential for the measurement of the complex index at extremely high temperature, which makes it possible to evaluate the luminous efficacy and lifetime of the microcavity illuminants and the cluster illuminants.

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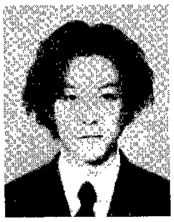

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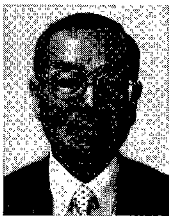

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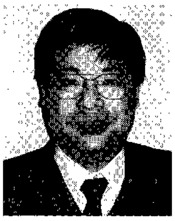

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