Ellipsometric studies on rough Zn and Cd polycrystalline samples

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Abstract

A manual null ellipsometer is constructed and used to determine the optical constants of zinc and cadmium of different surface roughness at the wavelength 632.8 nm, angle of incidence 45° and room temperature. The studied samples are polycrystalline sheets of thickness ~ 0.05 mm and surface area 30 mm x 30 mm. The samples were mechanically polished using diamond paste of different grain sizes. The final stage of polishing for the samples was with pastes of grain size 0.5 μ m, 2.5 μ m, 5 μ m, 10 μ m and 14 μ m. The ellipsometric parameters ψ and Δ were obtained and were used to calculate the optical constants *n* (refractive index) and *k* (extinction coefficient). It was found that as the roughness is increased, the refractive index *n* increases while the extinction coefficient *k* decreases. The used optical arrangement for ellipsometric measurements is presented and the procedure for extracting the results is explained. The uncertainty in the results is calculated and included in the results.

Key words: Zinc and Cadmium, Rough samples, Ellipsometry, Optical constants.

1. Introduction

Several methods are known for measuring the optical constants of metals using polarized light. These include the principal angle of incidence / principal azimuthal angle method [1, 2], reflectance of plane polarized light at non-normal incidence [3] pseudo-polarizing angle method [4] and ellipsometric measurements [5, 6]. Other techniques include interferometric, reflectance, photometric and other methods [7-9].

Ellipsometry is a very sensitive and accurate technique for measuring the optical constants of conductors provided that the surface of the studied sample is perfectly smooth. Surface roughness

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is sometimes neglected in determining the optical constants which leads to distinct results for samples measured under the same conditions [10, 11].

Plane polarized beam falling obliquely on a smooth metallic surface will be reflected in a state of elliptical polarization. Two parameters could be measured from the reflected polarization ellipse, which are the amplitude ratio $tan \psi$ and the phase difference Δ between the orthogonal parallel *p* and perpendicular *s* components of the electric vector. These two parameters are used to determine the optical constants *n* and *k*.

In this work, we studied the effect of surface roughness on the optical constants of Zn and Cd. Five samples of each metal were prepared with different roughness degrees. The parameters ψ and Δ were measured and the optical constants are derived.

2. Experimental

The studied samples are polycrystalline sheets of Zn and Cd of purity 99.999%, thickness ~ 0.05 mm and surface area 30 mm x 30 mm supplied by Zweigniederlassung der Degussa (Frankfurtam Main). Five samples of each metal (S₁, S₂, ..., S₅) were subjected to mechanical polishing using diamond pastes of different grain size. The sample S₁ was successively polished with pastes of grain size 14 µm, 10 µm, 5 µm, 2.5 µm and 0.5 µm. Samples S₂...S₅ were polished with the final step using pastes of 2.5, 5, 10 and 14 µm. Ellipsometric measurements were carried out with our constructed manual ellipsometer at angle of incidence 45° and room temperature $(20 \pm 1)^{\circ}$ C. The ellipsometer, Fig. 1, consists of: stabilized intensity He-Ne laser light source $(\lambda = 632.8 \text{ nm})$ followed by the polarizer *P* and the quarterwave plate *C* oriented with its fast axis at 45°. Light falls on the sample which is placed on a 0° - 360° horizontal rotary positioning stage of resolution 5 min. The reflected beam passes through the analyzer *A* and the detecting system (photomultiplier, DC power supply (0 – 2000 V) and digital voltmeter). The general equation of ellipsometry **[12]** is,

$$\rho = \tan \psi \, e^{i\Delta} = f(n,k) \tag{1}$$



Fig. 1 The ellipsometric system used for measuring the optical constants of Zn and Cd.

where ρ is the complex reflectance ratio, $\tan \psi$ is the ratio between the magnitudes of the parallel p and perpendicular s reflected components and $\Delta = \delta_p - \delta_s$ is the phase shift between the two components. P and A are rotated simultaneously for extinction and two extinction pairs could be obtained where

$$p_1 = p_2 \pm 90^{\circ},$$
 (2)

$$a_1 = a_2 \pm 90^{\circ}.$$
 (3)

where p and a refer to the settings of P and A at extinction. The parameters ψ and Δ are related to the extinction pairs by the relations [2]

$$\Delta = (2p_1 - 90^\circ) = (2p_2 - 270^\circ), \tag{4}$$

$$\psi = a_1 = -a_2. \tag{5}$$

For angle of incidence 45°, ψ and Δ are related to the optical constants *n* and *k* by the relations

$$n^{2}(1-k^{2}) = 0.5 \left[1 + \frac{(\cos^{2} 2\psi - \sin^{2} 2\psi \sin^{2} \Delta)}{(1+\sin 2\psi \cos \Delta)^{2}} \right],$$
(6)

$$2n^{2}k = \frac{0.5\sin 4\psi \sin \Delta}{(1+\sin 2\psi \cos \Delta)^{2}}$$
(7)



Fig. 2. Variation of the ellipsometric parameters ψ and Δ for samples of Zn of different surface roughness degrees.



Fig. 3. Variation of the ellipsometric parameters ψ and Δ for samples of Cd of different surface roughness degrees.

3. Results and Discussion

Each of the studied samples was placed on the horizontal rotary positioning stage immediately after polishing. The polarizer P and the analyzer A were simultaneously rotated until light leaving the analyzer is extinguished. The orientations of P and A at extinction determine the ellipsometric parameters ψ and Δ according to Eqs. (4) and (5). Figures (2) and (3) show the variation of ψ and Δ with the surface roughness degree for zinc and cadmium. For both metals, ψ decreases while Δ increases as the roughness increases. This behavior was observed for polycrystalline samples of gold of different roughness degrees [5, 6]. Substituting the values of ψ and Δ into Eqs. (6) and (7) and solving, we get the optical constants n and k for Zn and Cd as shown in Figures (4) and (5) and Tables 1, 2. It is clear that for both Zn and Cd, the refractive index n increases and the extinction coefficient k decreases as the degree of roughness increases. In Tables 1, 2, the calculated expanded uncertainty U_e is included in the results.

Table 1. Values of the optical constants n and k of Zn for samples $S_1 - S_5$ of different roughness degrees. U_e is the calculated expanded uncertainty.

Sample	Paste grain size;µm	ψ (deg.)	⊿ (deg.)	$n \pm U_e$	$k \pm U_e$
S_1	0.5	43.17	-195.83	1.143 ± 0.047	4.177 ± 0.205
S_2	2.5	43.08	-195.66	1.217 ± 0.053	3.941 ± 0.153
S ₃	5	43.00	-195.50	1.286 ± 0.062	3.746 ± 0.199
S_4	10	42.91	-195.17	1.390 ± 0.060	3.512 ± 0.183
S 5	14	42.75	-194.83	1.543 ± 0.056	3.194 ± 0.141

Table 2. Values of the optical constants n and k of Cd for samples $S_1 - S_5$ of different roughness degrees. U_e is the calculated expanded uncertainty.

Sample	Paste grain size;µm	ψ (deg.)	∆ (deg.)	$m{n} \pm m{U}_{m{e}}$	$m{k} \pm m{U}_{m{e}}$
S_1	0.5	37.83	-238.18	1.336 ± 0.039	3.181 ± 0.093
S_2	2.5	37.66	-237.84	1.378 ± 0.025	3.094 ± 0.072
S ₃	5	37.50	-237.50	1.419 ± 0.033	3.016 ± 0.081
S_4	10	37.25	-237.18	1.475 ± 0.028	2.904 ± 0.044
S_5	14	37.08	-236.84	1.518 ± 0.028	2.829 ± 0.065



Fig. 4. Refractive index n and extinction coefficient k dependence on the roughness degree for Zn.



Fig. 5. Refractive index n and extinction coefficient k dependence on the roughness degree for Cd.

Conclusion

We have studied the effect of surface roughness on the optical constants of polycrystalline samples of zinc and cadmium by ellipsometry. Five samples were studied of different roughness degrees were mechanically polished using diamond paste of different grain sizes (from 0.5 μ m to 14 μ m). The ellipsometric parameters ψ and Δ were calculated and used to calculate the optical constants n and k for the studied samples. It was found that as the roughness increases, n increases while kdecreases.

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الملخص باللغة العربية

دراسات إلبسومترية لمعدنى الزنك والكادميوم بدرجات خشونة مختلفة في الحالة المتعددة البلورية

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باستخدام إلبسوميتر تم تجميعه بالمعمل تم قياس الثوابت الضوئية لشرائح متعددة الحالة البلورية من معدنى الزنك والكادميوم بدرجات خشونة مختلفة لدراسة تأثير الخشونة على الثوابت الضوئية وذلك عند الطول الموجى 632.8 نانومتر وفى درجة حرارة الغرفة ، والعينات التى تمت دراستها عبارة عن شرائح ذات سمك حوالى 0.05 مم ومساحة السطح 30 مم * 30مم وقد تم الحصول على درجات الخشونة بأستخدام معجون الماس بدرجات مختلفة وقد وجد انه كلما زادت خشونة السطح زادت قيمة معامل الانكسار وصغرت قيمة معامل الخمود.