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OPTICAL STUDY OF STEEL SURFACES AFTER THERMOCHEMICAL TREATMENT

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Abstract:
In this paper we introduced the ellipsometrical measurements of steel OLC-45 after thermochemical treatment. We obtained the optical constants and the film thickness of nitrated case formed on the steel surface after thermochemical treatment. This nitrated case and the metallic substrate have different mechanical properties because of the metallic nitride formed.
The correlation between the optical properties and the mechanical properties of metallic surfaces allow to correctly decide upon the optimal thermochemical treatment settings for a certain type of steel.
Keywords:
ellipsometry, optical constants, thermochemical treatment, nitriding steel

1. INTRODUCTION

The thermochemical treatment of the metallic surface forms films with special properties, which differ from those of the metal.

Ellipsometry is an optical study of metal surfaces of a special smoothness. It allows us to obtain the optical properties of the solid surfaces and of the surface films formed on the solid surfaces from a change in the state of light polarization that is reflected on the surface [1,3,6].

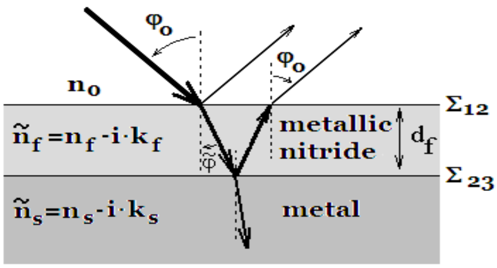

Figure 1. Light reflection on a metal covered by an optically absorbent film

Figure 1 presents the model of the metal surface coated with a film formed by the superficial thermochemical treatment, which reflects a monochromatic radiation.

The fundamental ellipsometry equation links the measurable angles Δ and Ψ to the optical constants of the substrate $\overline{n}_s, \overline{k}_s$, the superficial film thickness d_f and the optical constants of the superficial film $\overline{n}_f, \overline{k}_f$:

$$tg\Psi e^{i\Delta} = f\left(n_o, \varphi_o, \lambda, \overline{n}_f, \overline{k}_f, d_f, \overline{n}_s, \overline{k}_s\right) \quad (1)$$

In order to know the optical properties of the film formed on the surface after thermochemical treatment it is necessary to know the optical constants $\overline{n}_s$ and $\overline{k}_s$ of the substrate [1,6].

They are determined by a measurement of the angles $\overline{\Delta}_o$ and $\overline{\Psi}_o$, for the metal, before the thermochemical treatment. The optical constants of the substrate are obtained by solving the equation:

$$tg\overline{\Psi}_o e^{i\overline{\Delta}_o} = f\left(n_o, \varphi_o, \lambda, \overline{n}_s, \overline{k}_s\right) \quad (2)$$

The films formed on the surface of steel are optically absorbent so that both the thickness d_f and the optical constants $\overline{n}_f$ and $\overline{k}_f$ must be determined.

If the superficial film with complex refractive index $\tilde{n}_f = \overline{n}_f - i \cdot \overline{k}_f$ is optically absorbent, the relationship described by (1) does not allow the determination of three unknown: d_f, $\overline{n}_f$ and $\overline{k}_f$ by a single pair of Δ and Ψ parameters, measured at a single angle of incidence.

The graphical methods are commonly used to determine the three sizes d_f, $\overline{n}_f$ and $\overline{k}_f$ by means of the ellipsometric measurements:

- at two or more different incidence angles;
- at two or more wavelengths;

- for two or more incidence media with different refractive indices [1,2,6].

Reference literature presents in detail the technique for processing the data resulting from experimental measurements [1,4,6].

If the optically absorbent superficial film thickness is greater than a minimum value d_m, then radiation is absorbed in the superficial film and does not reach the Σ_{23} interface. In this case angles Δ and Ψ do not depend on the film thickness. They remain constant as can be observed in Figure 2. From Δ_f and Ψ_f we obtained the optical constants of the superficial film as bulk material. The minimum film thickness from which Ψ and Δ remain constant depends on the optical constants of the superficial film.

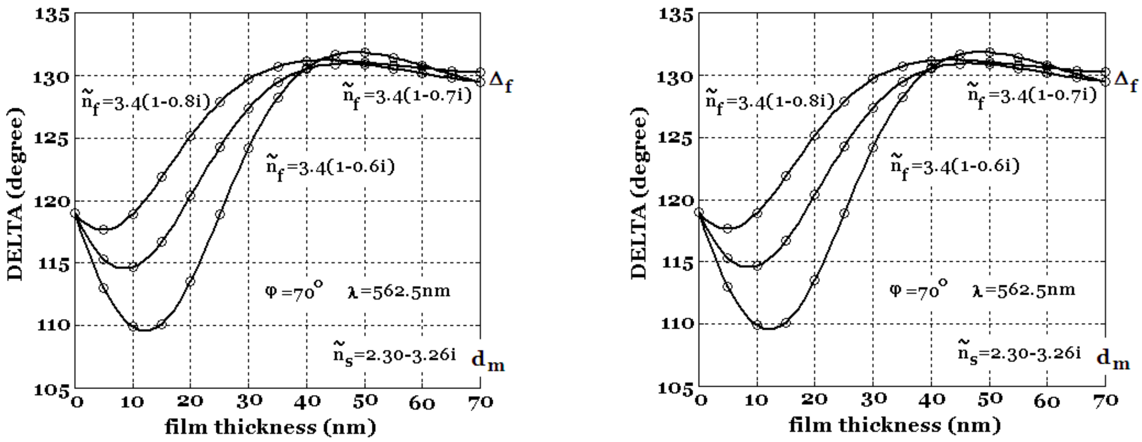

Figure 2 Δ and Ψ angles versus the thickness of an optically absorbent film formed on the metallic surface.

The ellipsometry relationship expressed by (1) no longer allows the determination of the thickness d_f of the superficial film. From the relationship:

$$tg\overline{\Psi}_f e^{i\overline{\Delta}_f} = f\left(n_o,\varphi_o,\lambda,\overline{n}_f,\overline{k}_f\right) \tag{3}$$

we can only determine the optical constants $\overline{n}_f$ and $\overline{k}_f$ based on the ellipsometrical angles at one angle of incidence. In relation (1) it is assumed that the superficial film has an arbitrarily chosen value, but greater than d_m.

2. EXPERIMENTS

The surface of steel samples OLC-45, was polished and then burnish extra-brightly with aluminum oxide burnishing powder. Before the ellipsometrical measurements for the determination of the optical constants, the metal surface of the freshly polished samples was washed in a jet of distilled water to remove impurities and then it was washed with absolute ethanol.

The thermochemical treatment of steel samples was performed in an ion-nitriding furnace for 8 hours. The samples were then washed by the method described above, before the ellipsometrical measurements of the film that was formed.

The ellipsometrical angles Δ and Ψ through which we determined the optical constants of the surfaces were measured by means of a photoelectric ellipsometer in PCSA assembly (polarized, compensatory, surface, analyzer) with an accuracy of 0.1°. The calculations were made with the help of our own computer program based on the calculation developed by McCrackin [6].

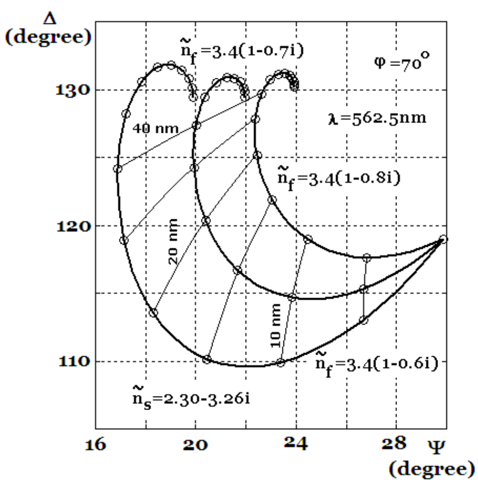

Fig. 2 The theoretical curves Δ=f(Ψ) for superficial optical absorbent film.

3. RESULTS AND DISCUSSION

There were two methods used for determining the thickness and the optical constants of the surface films.

In one of the methods, we used the optical constants of the substrate with which it can draw the theoretical curves Δ= f (Ψ) for various possible values of

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the optical constants of the film formed by thermo chemical treatment [2]. Figure 2 shows the appearance of these curves. The optical constants have values between $\overline{n}_f$ =1.5 ÷ 3.0 respectively $\overline{k}_f$ = 0.75 ÷ 1.5.

Thus, we determined the approximate values of the optical constants of the superficial film. The error of this determination depends on the accuracy of the graphical representation and of the reading of the graph.

Another method for determining the thickness and the optical constants of superficial films uses a set of two ellipsometrical measurements for metal covered with the surface film, at two different incidence angles: 60 ° and 70 ° [5].

$$tg\Psi_1 e^{i\Delta_1} = f\left(n_o, \varphi_{01}, \lambda, \overline{n}_f, \overline{k}_f, d_f, \overline{n}_s, \overline{k}_s\right) \quad (4)$$

$$tg\Psi_2 e^{i\Delta_2} = f\left(n_o, \varphi_{02}, \lambda, \overline{n}_f, \overline{k}_f, d_f, \overline{n}_s, \overline{k}_s\right) \quad (5)$$

The results of the measurements, the optical constants and the superficial film thickness are shown in Table 1.

For nitride layers whose thickness is larger than 100 nm the ellipsometrical measurement determines only the optical constants of the layer, by means of equation (3).

Table 1. The ellipsometrical angles measured and the optical characteristics of the metal and of the superficial film obtained by ion-nitriding of OLC-45 steel

Sample	Metallic substrate					Nitriding steel					
	φ_o	Δ	Ψ	$\overline{n}_s$	$\overline{k}_s$	φ_o	Δ	Ψ	$\overline{n}_f$	$\overline{k}_f$	d_f
-	deg.	deg.	deg.	-	-	deg.	deg.	deg.	-	-	nm
1	70	118,45	29,35	2,32	3,18	60	152,07	28,72	3,46	2,46	22
						70	124,92	20,35			
2	70	119,01	29,85	2,30	3,26	60	147,39	28,72	3,4	2,48	15
						70	118,42	22,06			

4. CONCLUSIONS

The determination of the optical constants and the surface film thickness require at least two ellipsometrical measurements at different incidence angles or wavelength or by using different incidence media with different refractive indices.

The optical characteristics of optical absorbent films can be obtained by rather laborious graphical methods.

If the superficial films have the thicknesses greater than 0.1 μm, it is not possible to determine the thickness by the ellipsometrical method. The optical constants determined from the Δ and Ψ ellipsometrical angles correspond to the bulk metallic nitride.

The correlation between the optical properties of films formed after thermo chemical treatment and the mechanical properties of the metals allow an optimal treatment to be applied to particular steel.

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