SECTION 5
DIAGNOSTICS AND METHODS OF RESEARCHES

MULTIPURPOSE OPTICAL SETUP FOR STUDYING RADIATION-INDUCED TRANSFORMATIONS OF METALS AND ALLOYS SURFACE

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A multipurpose optical setup for studying radiation-induced transformations of metals and alloys surface and optical properties of transparent objects is developed and created. The setup scheme is justified and the results of its verification are given.

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INTRODUCTION

Recently, branches of physics related to creation of resistant to radiation, thermal and chemical influence materials develop actively. It is linked, in particular, to creation of new nuclear power installations within alternative energy sources search, and to introduction of new radiation and electrophysical technologies.

Since materials interaction with environmental factors occurs through the surface, the latter in many cases is the first to change under these factors influence. Thus, the analysis of the surface allows to study early stages of radiation-induced processes in materials.

There are plenty of techniques to study a surface state, which may be applied depending on the purposes and objects under investigation. Among them there is a set of optical techniques (microscopy, interferometry, ellipsometry, reflectometry etc.) which allow to receive comprehensive information about the surface.

These techniques allow to study both structural changes of a surface (from units to hundreds and more nanometers) and changes in electronic structure of materials surface without any destructive effect. These makes optical techniques unique. Complex application of the listed techniques is especially effective.

Surface processes considerably influence materials optical properties. In particular, such optical characteristics as reflection and transmission coefficients substantially depend upon properties of the damaged layers, formed on the surface as a result of external influence. Parameters of light scattering from a surface allow to study the surface relief etc.

Earlier it was shown that ellipsometry is an effective method for studying phenomena that take place at initial stages of radiation impacts on materials [1, 2].

It was shown that it is effective to supplement ellipsometry by reflectometry when studying nontrivial effects, for example, blistering [3]. This is due to the fact that ellipsometry and reflectometry are based on different physical phenomena. Ellipsometry studies the change of polarization state of mirror component of the probe radiation, i.e. gets information only about those areas of the surface which reflect specularly. Reflectometry measures the total energy reflected specularly from the sample. Hence, even slight defects of the surface lead to increase of diffuse component of reflection, and, therefore, to decrease of the specular reflection. On the other hand, diffusion component contains information about the surface relief. Taking these data into account allows to make more reasonable choice of the models for ellipsometric results analysis.

Thus, combined application of several optical techniques allows better use the capabilities of each of them. The following characteristics are of interest for comprehensive optical study of radiation-induced processes surface of solids:

1. spectra of specular reflection coefficient at normal (near-normal) light incidence angle;
2. angular dependences of specular reflection coefficient at different wavelengths;
3. angular dependences of Fresnel reflection coefficients R_P and R_S at different waves lengths;
4. scattering indicatrix, as characteristic of the surface relief development extent;
5. spectra of the principal angle (Brewster's angle for transparent materials).

Measurements of the listed parameters combined with capabilities of microscopy, interferometry and ellipsometry, provide considerable amount of information about processes on a surface received in a contactless and nondestructive way.

This work is aimed at development and realization of a multipurpose setup for measurement of the characteristics listed above.

1. OPTICAL AND MECHANICAL SCHEME

The setup is based on the two-channel optical scheme described in [4] (Fig. 1).

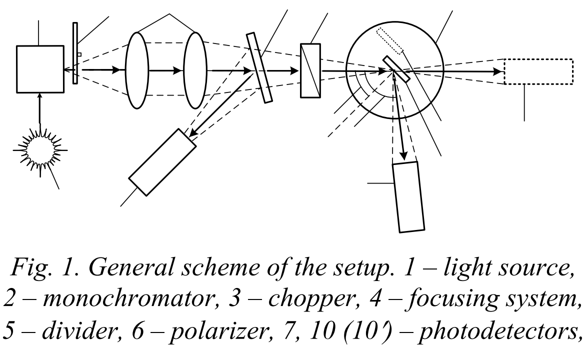

Fig. 1. General scheme of the setup. 1 – light source, 2 – monochromator, 3 – chopper, 4 – focusing system, 5 – divider, 6 – polarizer, 7, 10 (10′) – photodetectors, 8 (8′) – sample, 9 – goniometer

The beam of light from a source (1) after passing through monochromator (2) and 72 Hz chopper (3) is focused by lens system (4) on the sample (8) surface (incidence angle θ_1). Than it reflects from (or passes through) the sample and gets a photodetector (PD) PD-1 (10 or 10′). PD-1 terminates the main measuring channel. PD-1 is mounted at an angle θ_2 to the optical axis. In front of the sample there can be a polarizer (6) with arbitrary polarization azimuth.

After lens system (4) the divider (5) can be mounted. It directs a part of the beam to photodetector PD-2 (7), which terminates the additional channel (the comparison channel). This two-channel scheme advantages and application are described below.

Various photo-electric converters with proper characteristics can be used as PD-1 and PD-2. We used photomultipliers ФЭУ-100 (200...800 nm spectral range) and ФЭУ-62 (400...1200 nm range).

Monochromator of a standard spectrophotometer СФ-4 is used in the setup. Two lamps can be used: 100 W halogen lamp for 400...1200 nm spectral range and a discharge hydrogen lamp for 200...400 nm range.

An original mechanism (the goniometer henceforth) providing possibility of the automated independent coaxial rotation of the sample stage and PD-1 is the main mechanical block of the setup. The assembly drawing of the goniometer is shown in Fig. 2.

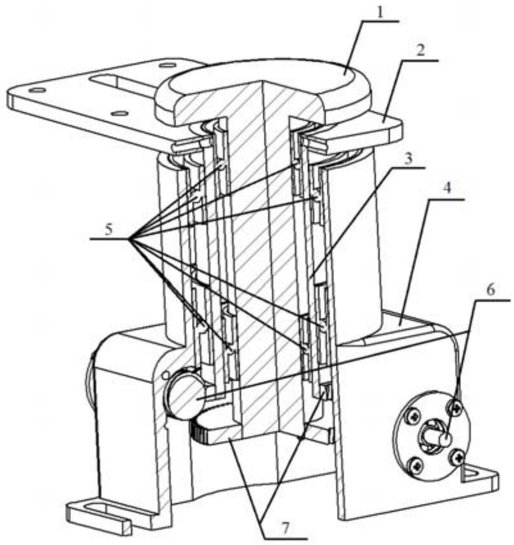

Fig. 2. Assembly drawing of the goniometer. 1 – sample stage, 2 – alidade, 3 – alidade body, 4 – the goniometer housing, 5– bearings, 6 – worms, 7– worm gears

The sample holder is mounted on the sample stage (1) in such a way that the sample surface (position (8) in fig. 1) is placed on the rotation axis of the sample stage. This axis coincides with the alidade (2) rotation axis. Photodetector PD-1 is mounted on the alidade. The sample stage and alidade can rotate independently by means of worm drivers (7). The worms (6) are protrude outside the goniometer housing (4) for connection to the stepper motors (SM).

The sample stage has a mechanism for translational motion of the sample holder parallel to the stage surface. This allows to input/output the sample into/out the beam (positions (8) and (8′) of the sample on Fig. 1).

The design of the goniometer allows PD-1 and the sample independent rotation. It makes possible to measure all the parameters listed in the introduction. The actual smallest value of θ_2 at which the detector doesn't close the beam is less than 10°.

Besides, the setup makes it possible to measure a transmission spectra at any incidence angle and any polarization azimuth (i.e., actually, angular dependences of Fresnel transmission coefficients T_P and T_S).

The measurement type can be chosen programmatically without changes of the optomechanical scheme of the setup.

2. MEASUREMENT TECHNIQUE

Spectral measurements on the setup are carried out in two passes over the spectral range. At first pass intensity of the initial signal without sample at the main channel (dependence $I_0(\lambda)$) is measured. At this pass PD-1 should be set to $\theta_2 = 180^\circ$ position (“peek-a-boo” position) that corresponds to a position (10′) in Fig. 1. At second pass the measurements are made with the sample in the measuring channel (dependence $I_1(\lambda)$).

For specular reflectance coefficient $R(\lambda)$ measurements at θ_1 incidence angle PD-1 should be set to $\theta_2 = 2\theta_1$ position. The polarizer should be removed or its polarization plane should be set to 45°. The required value R (with a fixed wavelength λ) is calculated according to expression:

$$R = \frac{I_1}{I_0} \cdot 100\% \cdot$$

Fresnel coefficients R_P and R_S may be measured in the same way, but with the polarizer set properly.

To measure scattering indicatrix one should fix incidence angle θ_1 and wavelength λ and rotate PD-1 within admissible range of θ_2.

To measure transmission coefficient T one should place the sample into position (8) (fig. 1) and remain PD-1 in the “peek-a-boo” position ((10′) in fig. 1). Than T may be calculated similarly to R:

$$T = \frac{I_1}{I_0} \cdot 100\% \cdot$$

When applying such measuring technique two types of distortions (fast and slow) always take place. The noises of PDs, PD power supplies and light source may be referred to as fast distortions. Their influence is almost completely removed via synchronous detecting and temporal averaging.

Slow distortions can be caused by ripple light source that is especially actual when using discharge sources at UV-range (which is most important for metals). Besides, high-voltage power of a photodetector may have some variable component which considerably will influence the outcome at small signals. Also there can be other sources of slow distortions.

The comparison channel is aimed to remove slow distortions. PD-2 should be powered from the same supply as PD-1. Then all distortions are registered by the PDs in phase. Inphase components may be removed by dividing the main channel signal by the comparison channel signal. The detailed description of the two-channel technique and its analysis is given in [4].

3. SYSTEM OF AUTOMATION AND SIGNAL PROCESSING

The setup as an automation object consist of three controls (the monochromator wavelength selector, the object stage and the alidade) and two PDs (Fig. 3).

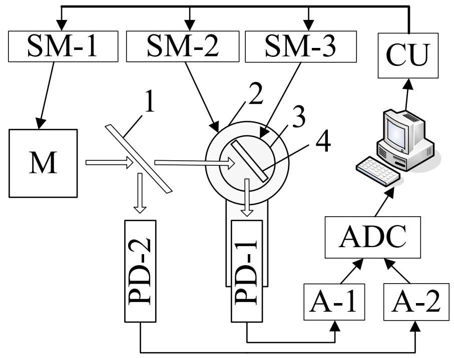

Fig. 3. Scheme of the setup as an automation object. 1 – divider, 2 – alidade, 3 – sample stage, 4 – sample, M – monochromator, PD-1(2) – photodetectors, A-1(2) – amplifiers, CU – control unit, SM-1(2,3) – stepper motors, ADC – analog-digital converter

Automation of the setup is carried out as follows. Stepper motors SM-1 and SM-2 are mounted to the goniometer worms and provide not worse then 0.02° accuracy of the stage and the alidade positioning. SM-3 rotates the wavelength selector of the monochromator and provides minimal change of λ not large than 0.5 nm. ДШИ-200-1 stepping motors were used.

All SM are connected to specially developed control unit (CU) which is connected to a computer (PC).

Signals from both PDs are directed to current-voltage converters combined with measuring amplifiers (U-1(2)) [5], and further to the two-channel analog-digital converter (ADC). The digitized data is transmitted to the PC for processing. Since all measured values are relative, absolute calibration of ADC is not required and a PC sound card can be used as ADC.

Special software providing all types of measurements was developed to control the experiment. The created automation system is based on the universal hardware-software platform [6].

The first stage of data processing consists in distortions (ambient lighting, PDs dark current etc) removing. For this the signal is subject to software synchronous detecting which is a function of the platform used. Quality of the synchronous detector is high enough to carry out measurements at ambient light without any light-blocking. Also the system allows to register signals comparable with PhEM dark current.

4. APPROBATION AND TESTING

Detailed testing of the device in various modes was carried out. Its characteristics are close to the expected.

Unfortunately, metal samples reflection spectra can't be used to estimate the measurements accuracy quantitatively by comparing with reference data since it significantly depend upon a number of uncontrollable factors [7]. Nevertheless, experimental reflection spectrum of molybdenum and the reference one [8], qualitatively coincide (Fig. 4).

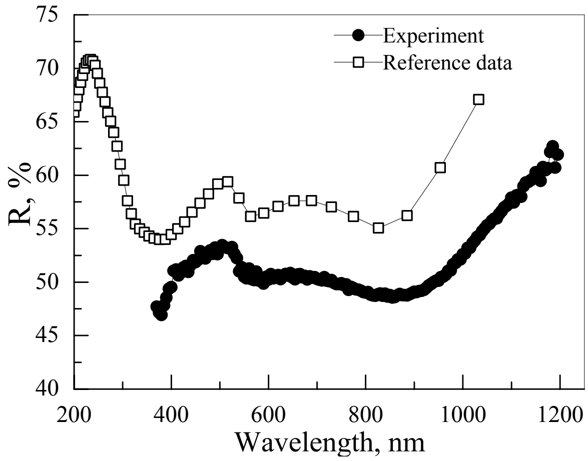

Fig. 4. Mo reflection coefficient spectra

The plate of optical glass ЖЗС-18 was studied to estimate the accuracy quantitatively. This very brand of glass was chosen since it has complex transmission spectrum: there are ranges of both high and low transmission and absorption edges are rather steep. These allowed to test the setup in wide signal range.

Fig. 5 presents experimental reflection $R_{\exp}(\lambda)$ and transmission $T_{\exp}(\lambda)$ coefficients spectra of ЖЗС-18 glass and reference data [9] (for convenience $R_{\exp}(\lambda)$ is plotted multiplied by 10). One can see that all features of the measured and reference spectra coincide, but the measured transmission values are slightly lower than the reference ones. This is due to the reflection from the back surface which isn't considered in reference data. To compensate this effect the sum $R_{\exp}(\lambda)+T_{\exp}(\lambda)$ was found and it coincided with the reference transmission values with accuracy better than 2% over the whole spectral range (Fig. 5).

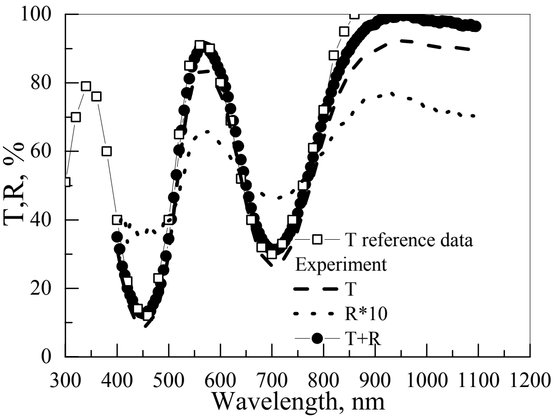

Fig. 5. Spectral dependences of transmission (T) and reflection (R) coefficients for ЖЗС-18 glass

Angular dependences of Fresnel coefficients $R_P(\theta)$ and $R_S(\theta)$ for БС-12 glass at $\lambda = 550$ nm (Fig. 6) were measured to test the possibility of angular and polarizing studies. Qualitatively these dependences completely correspond to theoretical representations: at small angles $R_S(\theta)$ and $R_P(\theta)$ coincide, $R_S(\theta)$ grows monotonously, $R_P(\theta)$ has one inflection point and one minimum. The minimum angle corresponds to transparent materials characteristic called Brewster angle (θ_{Br}). It's important since $tg\theta_{Br} = n$. To find θ_{Br} precisely the $R_P(\theta)$ curve was approximated with a 3rd degree polynomial (insert in Fig. 6) near the minimum, and θ_{Br} value was determined as the polynomial minimum. The found value for БС-12 glass is $\theta_{Br} = 56.42^\circ$ that corresponds to refraction index $n_{\exp} = 1.506$. The reference БС-12 refraction index value is $n_{ref} = 1.508$. Thus, the mistake is ~0.1%. Measurement of θ_{Br} (or the principle angle for absorbing materials) and, respectively definition of refraction index of transparent materials, is one of the algorithms of the setup software.

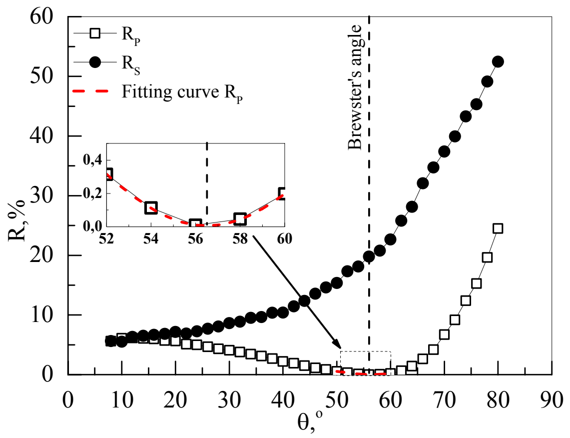

Fig. 6. Fresnel coefficients R_p and R_s angular dependences for БС-12 glass

To test the setup in scattering indicatrix measurement mode samples of stainless steel with different roughness were studied (Fig. 7). The roughness was formed by surface sputtering by Ar ions.

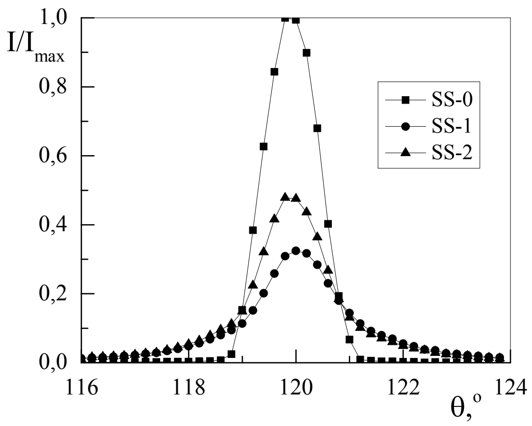

Fig. 7. The dispersion indecatrix of stainless steel samples: SS-0 an initial sample, SS-1 and SS-2 the samples sputtered by Ar plasma with various fluence

The initial sample (SS-0) and two irradiated to different fluence ones (SS-1 and SS-2) were studied. Mass loss for SS-1 and SS-2 was Δm_1=14.1 µg and Δm_2=14.4 µg, respectively. The sample with bigger Δm is the roughest, and the initial one is the smoothest.

One can see (see Fig. 7) that the scattering indicatrix of rougher surfaces are wider and lower. This is fully consistent with theoretical concepts. The theory of light scattering from rough surfaces can be applied to obtain quantitative information about characteristics of a roughness from such experimental data [10].

CONCLUSIONS

A multipurpose setup for studying various optical characteristics of materials in wide spectral range (from middle UF to near IR) is created and tested. The set of experimental data which may be obtained using the created setup, may be used to study early stages of radiation-induced transformations in metals and alloys. Its application together with an ellipsometry technique may be especially effective.

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МНОГОФУНКЦИОНАЛЬНЫЙ ОПТИЧЕСКИЙ КОМПЛЕКС ДЛЯ ИССЛЕДОВАНИЯ РАДИАЦИОННЫХ ПРЕВРАЩЕНИЙ НА ПОВЕРХНОСТИ МЕТАЛЛОВ И СПЛАВОВ

А.И. Беляева, А.А. Галуза, И.В. Коленов, А.А. Савченко

Разработан и реализован многофункциональный оптический комплекс для исследования радиационных превращений на поверхности металлов и сплавов, а также оптических характеристик прозрачных тел. Обоснована схема комплекса и приведены результаты его тестирования.

БАГАТОФУНКЦІОНАЛЬНИЙ ОПТИЧНИЙ КОМПЛЕКС ДЛЯ ДОСЛІДЖЕННЯ РАДІАЦІЙНИХ ПЕРЕТВОРЕНЬ НА ПОВЕРХНІ МЕТАЛІВ І СПЛАВІВ

А.І. Беляєва, О.А. Галуза, І.В. Коленов, А.О. Савченко

Розроблено та реалізовано багатофункціональний оптичний комплекс для дослідження радіаційних перетворень на поверхні металів і сплавів, а також оптичних характеристик прозорих тіл. Обґрунтовано схему комплексу та наведено результати його тестування.