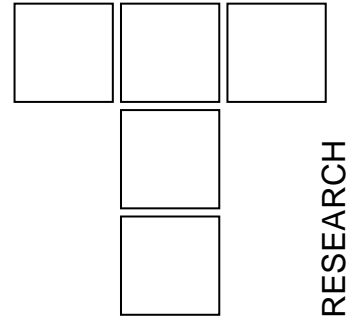


# Optical Coatings and Their Quality



*The development of optical coatings occurred relatively late, but today their number and characteristics cannot be comprised by one brief survey. In this paper, a short survey of basic types of thin optical coatings and their characteristics is presented. Special emphasis is given to definition of the quality of optical coatings.*

**Keywords:** *Optical coatings, quality.*

## 1. INTRODUCTION

Together with rapid development of optical electronics and quantum electronics, the forms of optical elements are enriched and the criterion for the quality of making is considerably intensified. The quality of elaborated areas of optical elements is expressed in parts of wave lengths of light radiation that passes or is reflected from them. The dimensions of optical elements and angles between some surfaces are tolerated in the limits of microns, that is angle seconds. The possibilities for choice of optimal solution for realization of corresponding optical function are big, concerning very large number of different optical elements. In order for them to correspond to their purpose in subconcatenations and concatenations, beside the tolerances of making which are strictly defined, special attention is paid to the choice of thin coatings which are, by definite procedures, applied to optical element.

## 2. OPTICAL COATINGS

The basic function of thin optical coatings is to improve characteristics of optical elements and to enable correct function of optical instrument and system. Depending of, above all, the reasons for applying to optical surface, layers are divided into the following types:

- antireflexive coatings,
- coatings – filters,
- mirrors,

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- dividers of light (semimirrors),
- protective transparent coatings and
- electric-implementing coatings (warmed coatings).
- Basic characteristic of mentioned coatings are:
- chemical stability,
- resistance to corrosion,
- mechanical and thermal resistance and
- optical qualities.

The choice of the type of coating comes from technical demands for given optical position, that is element, conditions of exploiting of the instrument, temperature area in which the instrument operates, position of the given element in the instrument and its shape, and also from the way of cleaning of optical instruments.

### 2.1 Antireflexive coatings

Smoothed surface of an optical element, in relation to the intensity of falling light, reflects part of that light depending on the index of breaking of material. If the index of breaking of material is bigger, the reflection of smoothed surface grows. If the system consists of large number of elements, the losses owing to reflection are extremely big, so it is necessary to reduce reflection on the surfaces of optical elements. For this purpose, antireflexive dielectric coatings are applied to smoothed surfaces. Thin layer is a system of three optical areas separated by two marginal surfaces whose interspace is negligibly small in relation to the travel of light in optical areas outside marginal surfaces. The simplest case of thin optical coating is got when the falling area of light is air, and

the ground tiles are made of optically smoothed glass. In that case, the layer is defined by breaking index of air ( $n_0=1$ ), breaking index of materials of thin layer  $n_f$  and breaking index of ground  $n_s$ .

Table 1. View of signs and symbols of thin coatings drawings of optical elements

	Data for surface according to DIN ISO 1302
	Reflexive layer on the front surface
	Reflexive layer on the back surface
	Dividing layer
	Layer with reduced reflection
	Special layer
	Polished layer

Thin coating is characterised by thickness which represents optical thickness of thin layer material. The optical thickness of layer is product of breaking index of layer material  $n_f$  and its geometrical thickness  $d$ . The falling light ray is partly reflected on marginal surfaces, and partly it passes into the other are (Figure 1). If the breaking index of thin layer material and ground is  $n_f < n_s^{1/2}$ . Namely, it can be concluded that, in given area of spectrum, the values of reflection on polished optical layer will lessen by applying of some non-absorptional material with optical thickness  $\lambda/4$  if the breaking index of that material is less than the breaking index of the material of ground. In the Table 2, the view of possible thin-layer materials for realization of antireflexive coatings is given.

Table 2. The view of antireflexive layer materials

Material	MgF <sub>2</sub>	SiO <sub>2</sub>	Na <sub>3</sub> AlF <sub>6</sub>	LiF	CaF <sub>2</sub>
Breaking index	1.38	1.44	1.36	1.37	1.43
Ground index	1.904	2.074	1.849	1.883	2.045

Considering the fact that the zero value of reflection is achieved only theoretically when there is  $n_f = n_s^{1/2}$ , for improvement of porosity of optical element antireflexive coatings with two and more materials are used. Thus, we differ one-fold, two-fold, three-fold and manyfold antireflexive coatings.

In the Figure 2 there is a spectral curve of one-fold antireflexive layer which is realized by applying of MgF<sub>2</sub> with thickness  $\lambda/4$  to polished surface of optical glass BK7. Reflection of layer in minimum is 1.5 % and the layer can be realized in spectral area from 400-2500 nm with the thickness of low reflexive zone from 200-300 nm.

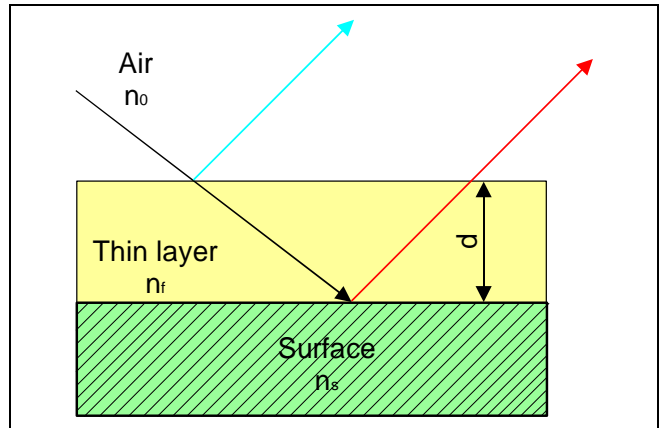


Figure 1. The scheme of a simple thin layer

The usage of two sublayers in realization of layer structure enables fulfillment of condition  $R < 0.5\%$  in wider spectral area.

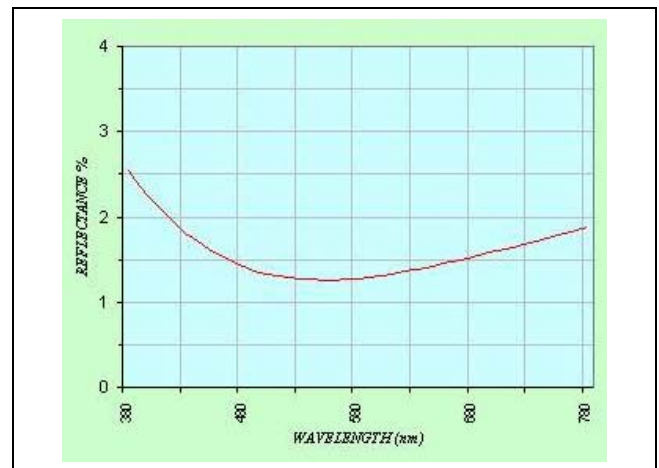


Figure 2. Spectral curve of one-fold antireflexive layer

Two-fold antireflexive layer is realized by combination of high – index material (CeO<sub>2</sub>, ZnS, TiO<sub>2</sub>) and low-index material (MgF<sub>2</sub>, Na<sub>3</sub>AlF<sub>6</sub>, SiO<sub>2</sub>).

The principle is based on the fact that in the first phase the reflection of the surface grows, by applying of high-index material to the amount in which the applying of low-index material will give zero reflection.

Wide-zonal antireflexive layers are, in practice, realized as combinations of three or four sublayers with full amount of  $\lambda/4$  thickness.

In the case of the layer with three materials, the combination of optical thickness  $\lambda/4 + \lambda/2 + \lambda/4$  from materials with middle+high+low value of breaking index is used. As low-index material,  $\text{CeF}_3$ ,  $\text{LaF}_3$ ,  $\text{Al}_2\text{O}_3$  are most commonly used. Which of possible combinations will be used depends on, above all, the type of the surface but also on the type of optical element. Surfaces with high breaking index have unfavourable qualities as an optical material, because they are characterized by high reflection on the marginal area surface-air.

In the recent years, the interest for realization of optical instruments in two spectral areas: 2 – 5  $\mu\text{m}$  and 8 – 14  $\mu\text{m}$ , which makes the realization of quality layers of low reflection difficult.

If the optical instrumen is made of several elements with high-index material, its transparency is negligible and the practical usage of these apparatuses has no sense. That is why it is necessary to do applying of antireflexive layers to the surface in order to increase porosity. For the analysis of antireflexive layers the surfaces from  $\text{As}_2\text{S}_3$ , Si and Ge are mainly used. These materials are transparent in two zones of infrared spectrum area, which are, in the recent years, the subject of special research. Namely, spectrum areas from 2 – 5  $\mu\text{m}$ , that is 8 – 14  $\mu\text{m}$  are highly transparent areas of atmosphere, and the realization of optical instruments in these areas of wave lengths has manifold usage.

Layers can also be classified in relation to the scope of wave lengths when the reflection of optical element is less. Thus, we differ antireflexive coatings for ultraviolet (UV), visible (V) and infrared (IC) spectral area.

Modern optical electronic apparatuses also demand antireflexive layers for two areas of spectrum. These layers are called double-antireflexive layers. Their realization is complex and the mathematical analysis with using of accounting technique is necessary for optimizing the number of sublayers, breaking index of material and thickness which will be realized.

## 2.2 Mirrors

Thin optical layers with marked reflection ( $R > 80\%$ ) are mirrors. According to their nature the mirrors can be realized on the base of:

- metals,
- metals with dielectrical coatings and
- on the base of manifold dielectrical thin coating structure.

According to the position on optical element, mirrors can be applied as back side and front side.

Back side mirrors are mainly realized by applying of Ag and by its protection with the layer of Cu and lacquer. Front side mirrors are mainly combination of metals (Ag or Al) and are protected by layer of  $\text{SiO}$ ,  $\text{SiO}_2$  or  $\text{MgG}_2$ . If the layer is Ag, the mirror has reflection above 90 % in visible spectral area, and Al-mirror gives reflection above 87 %. These values of reflection do not satisfy the modern optical system, that is why their reflection is increased by applying dielectrical layers. The advantage of metal-dielectrical interferential layers of the mirror is especially expressed in systems that function in wider spectral area or give passage to light in two spectral areas.

Since the construction of metal dielectrical mirrors needs less number of sublayers, their technological realization is much simpler than the realization of just dielectrical mirrors.

In the Figure 3, the characteristics of protective mirror coating on the base of silver is showed. Spectral characteristics of the layer are:

- reflection  $R_{\text{Ag}} > 97\%$  in the spectrum area 450 – 2500 nm with high stability.

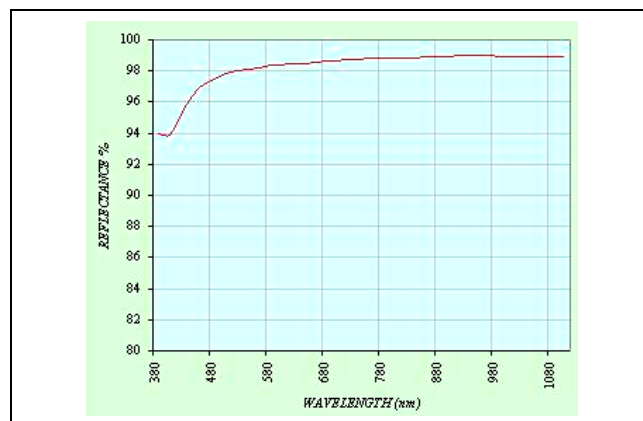


Figure 3. Spectral characteristic of division of mirror reflection on the base of silver (Ag) in combination with dielectrical materials

In the Figure 4 there is the view of front side (a) and back side (b) mirror.

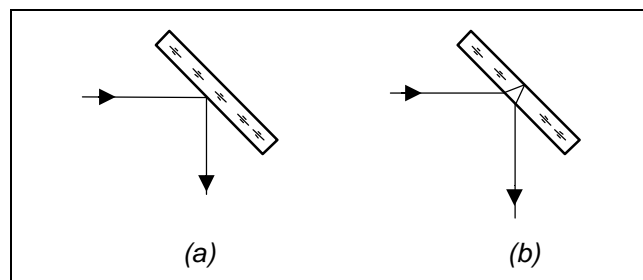


Figure 4. Front side (a) and back side (b) mirror

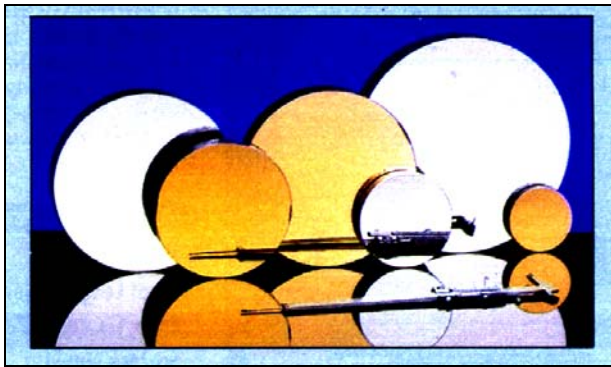


Figure 5. The view of metal mirror coatings of Al and Ag

### 2.3 Dividers of light (Semimirris)

Thin optical layers with reflection above low and high values are called semimirris. They can be realized on the base of metal (Cu, Al) or combination of dielectrical materials. Namely, the divider of light is the layer which we apply onto optical surface in case we want that surface to divide light bundle in two directions (Figure 6).

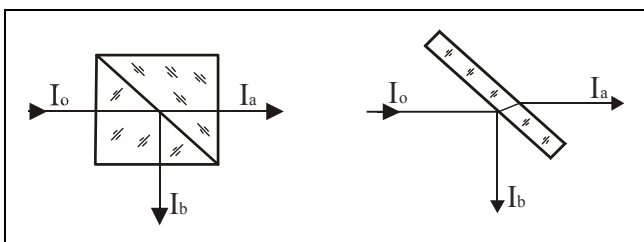


Figure 6. Dividers of light: a) Cube prism as divider of light, b) planparallel plate as divider of light

The falling bundle of light of intensity  $I_0$  is, on the divider layer, divided so that the part of the light  $I_B$  is reflected and, the part  $I_A$  passes. Considering the fact that this type of layers equally reflect wider spectral area, we call them intensity dividers of light.

Semimirris on the base of metals are harder realized considering the fact that these coatings are very thin (25 – 30 nm) and that with the little change of thickness the spectral characteristics are essentially changed.

## 3. THE CONTROL OF QUALITY OF OPTICAL COATINGS

Special care in the process of production of optical elements is given to control of thin optical coatings. Thin coatings always has two function. It, on one hand protects the polished surface, and on the other hand it gives the definite spectral function to optical element. In order for layer to do both function it is necessary to check cleanness of layer, colour, hardness, adhesion, moisture persistence, temperature

changes and to check its optical characteristics after its applying.

Before applying the optical coating special care is given to the control of quality of making the optical elements.

Considering the fact that every optical element in a system brings corresponding number and size of errors, when accounting the optical function it is required to even the total error according to statistical analysis of single errors. The errors of processing are different, and the most common even with standard elements are the errors of flatness that is spherical (errors of leaning), errors of centrality with lenses, pyramidal errors and angle errors with prisms and errors of voltage caused by processing. In this work there is a view of mentioned errors caused in processing of optical elements.

### 3.1 Cleannes of the coating

Cleannes of the coating is controlled in the same way as surface errors on polished optical area. In the process of preparing the elements for the layer, the errors which manifest either as uncovered area, stains, carvings or as damages caused by breaking of material in the course of applying are possible. Allowed errors are regulated by constructive documentation or special directions for controlling of ready optical elements.

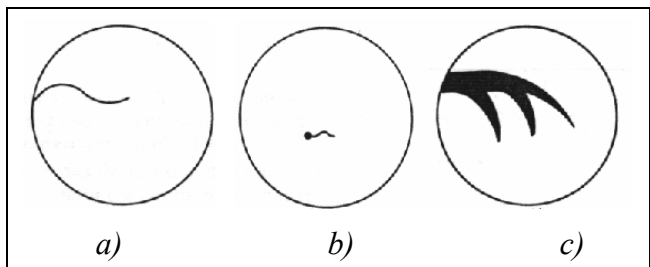


Figure 7. Basic types of carvings: a) yarn carvings, b) yarn carvings with connection in the shape of the spot (until recently marked as spot carvings), c) Ribbon carvings

Active surface of optical element is always polished. The degree of polishing is very high and is expressed through microunevenness of the surface. In comparison to other types of material processing polishing gives minimal value of microunevenness of the surface.

Technological process of processing the optical elements enables that in the final phase, that is in processing by polishing, the level of microunevenness does not overcome the value of 20

– 30 nm. Surface elaborated like this, on the other hand, doesn't have any influence on the light rays that passes or are reflected from it. However, in processing there are errors which condition light losses, dissipation or reflection on the surface. These disturbances of fine structure are called surface errors and they can be divided into: lynxes, scratches, spots, stains and hairs.

Reasons for occurring of surface errors may occur because of errors of optical materials or because of insufficiently precise process of processing the optical elements.

### 3.2 Colour errors

Thin layer is characterized by definite shade of colours. By observing it, it can be roughly evaluated whether the thin layer correspond to spectral demands. Namely, due to different geometry which is present in applying of coatings especially with elements of larger dimensions or sharper radius of the sphere, the division of the layer is not equal on all surface, which can be observed in the change of colour of layer in different zones of optical area.

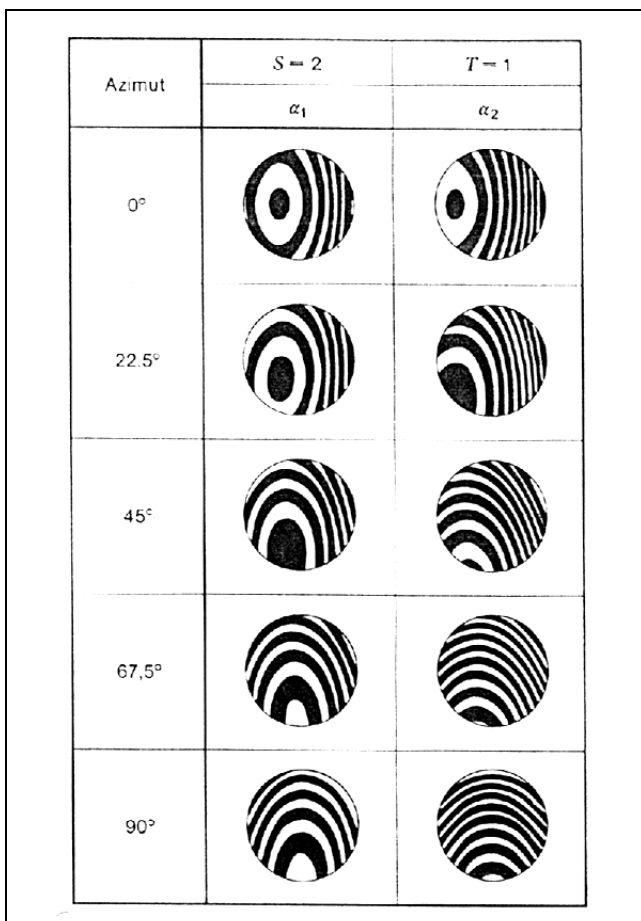


Figure 8. The oval error of leaning

The error of planation or sphericity is commonly called the error of leaning because it is identified as the

consequence of finishing the contact - leaning surface of the controller and the surface of the optical element. Considering the fact that then there are interferential stripes or rings, the error of planation or sphericity is also called the error of the colour.

According to the shape the error of the colour can be circular, oval or saddle - shaped.

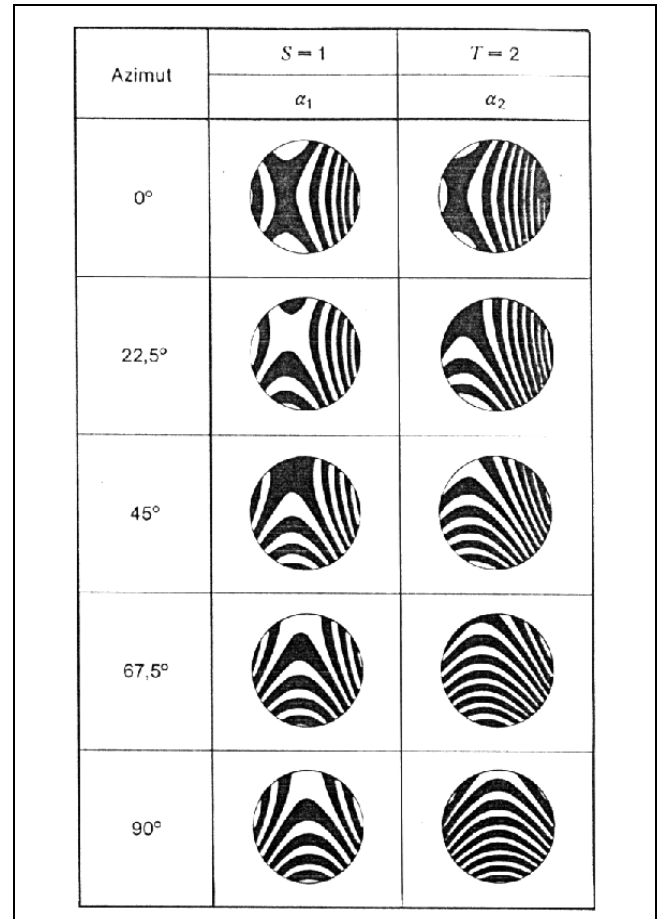


Figure 9. Saddle - shaped error of leaning

The checking of the colour with controller can be manifested as leaning from the centre of leaning from the back. If the controller which is brought to contact with the surface of the element is lightly pressed on front side the stripes will move. If the stripes move from the centre to the back of the observed surface, then the leaning is from the centre and reversly, when stripes move towards the centre. Then the leaning is from the back. If the observing is done in day light stripes are coloured.

In colour arrangement blue-yellow-red leaning is from the back, and if the arrangement of colours is red-yellow-blue, the leaning is from the centre. If the stripes are regular, aberration from the centre of the area is even in all directions. If the stripes are irregular, irregularity of the surface of the processed optical element is evaluated on the basis of their shape.

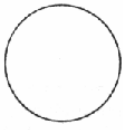











	S = 0	S = 1	S = 2	S = 3
T = 0	 3/0(0) -	 3/1(0) -	 3/2(0) -	 3/3(0) -
T = 1	 3/1(2) -	 3/2(2) -	 3/3(2) -	 3/4(2) -
T = 2	 3/2(4) -	 3/3(4) -	 3/4(4) -	 3/5(4) -

Figure 10. Types of legal errors of the colour

Controllers are made with absolute correctness in relation to given radius or flat surface. In optical production, many types of controllers are produced. As their surfaces are exposed to damage in constant usage, it is necessary to make workshop controllers for serial production. Controllers are always made in pairs of + and - surfaces and with the same radius. For workshop usage only the controller that is necessary is made that is, the convex one for the concave surfaces and the concave one for the convex surfaces of the lens.

Apart from the mentioned division according to the shape, the errors of the colour can be divided into rough, legal that is into equally arranged errors of leaning and fine errors of leaning. With rough errors there is no regular shape of interferential stripes, but only their appearance is registered in a high degree. If all interferential stripes are in the shape of a curve of the second rank, the error is legal. In the Figure 10, there are several types of legal errors of the colour.

### 3.2 The hardness of the coating

The hardness of the layer is examined by scrubbing the surface on which the coating is applied. For different layers different test for examining the hardness are examined. Basically, thin layers are divided into hard and soft, and criteria for examining the hardness are given for both groups of the coating. Soft coatings must mainly be resistant to cleansing with cotton - wool tampons

without putting them into alcohol, optical mixture or acetone, whereas the hard layers are examined according to special proceedings. The basic thing of the hardness examination consists in that that the surface with applied layer is wiped definite number of times with the rubber of definite hardness under given pressure. After the cleansing of the surface no changes are allowed on the coating.

### 3.2 Adhesion of the coating

Adhesion of the coating is very important and its checking enables reliability of building optical elements into mechanical strata. Adhesion is examined with sticky stripe of definite degree of binding in this way: the stripe is stuck to the surface of the optical element and lifted by a rapid move. If the layer is not left on the stripe, its adhesion is satisfying. The examination of resistance of the coatings to the impact of moisture of salt solution has aim to determine in what measure the layers are resistant to the impact of climate conditions.

The usual moisture test is done in duration of 12 hours at the chamber moisture of 98 % and the temperature of 40 °C. However, for special cases the test is done in cycles and lasts longer.

### 3.2 Spectral characteristics of the layer

Spectral characteristics of thin layers are examined spectrophotometrically. Spectrophotometers are apparatuses which measure the division of reflection or transparency, and the range of work is from UV area (220 nm) to far infra-red spectral area.

Today, for the control of thin layers, the spectrophotometers of famous world firms Perkin Elmer, Beckman, Zeiss are used.

The production of spectrophotometers can be in performance of optical diffractive grating with big power of exposition, prisms with high degree of dispersion, and modern systems make mainly combinations of prism and grating systems.

The large progress of electronics has enabled the production of almost automatized spectrophotometers. Every modern spectrophotometer disposes of the writer of spectral curve and numerical pointer of measure value.

The principle of measuring the reflection is based on the relative comparing of spectral division of specimen and etalon.

Dispersive system enables the exposition of spectral lines in the area from ultraviolet to infrared area of the spectrum (1  $\mu\text{m}$ ). Errors, ( $\pm 1\%$ ) that occur, are mainly the consequence of nonparallelism of the surface of the specimen, disposition of light from the surface of the specimen if it is not flat, double breaking and errors in material.

The most convenient system for measuring the reflection is Ulbricht's ball which is covered by the layer of MgO. The measure of reflection is done on the basis of comparing it to the etalon which can be a tile with applied MgO or BaCO<sub>3</sub>. These two diffuse coatings have the reflection of 96 % in the spectral area of  $600 \pm 50 \mu\text{m}$ . Etalon tile can be contaminated or cuneiform, thus, the errors are even possible while determining the range of the spectrophotometer's scale. Therefore, the calibrating of the instrument is done with larger number of etalon tiles.

Their quality satisfies if the spectral curves do not resign from the middle value more than 0.5 %.

The check of the etalon can be done by means of the mirror with approved arrangement of spectral reflection. For this purpose, Al-mirrors, which are applied vacuumly onto the polished tile made from optical glass, are most commonly used. The mirror, which is used for testing, must be freshly applied, considering the fact that Al oxidize into atmosphere with time, which creates the layer of Al<sub>2</sub>O<sub>3</sub> on its surface.

Spectrophotometers dispose of, mainly, three ranges of the scale 1:1, 1:3.3 and 1:10, which enables changing of reflection with the absolute error 0.5 % in scale range 0 - 100 %, 0.15 % for

the scale range 0 - 30 % and 0.05 % for the total scale range 0 - 10 %.

The errors which come from specimen are mainly the consequence of non-parallelism of surfaces of the specimen, defocusing of the light bundle, scattering of light from the surface of the specimen if it is not flat, double breaking and material errors.

While measuring the spectral characteristics it is necessary at the change of spectral area from visible to infrared, to redo calibrating because the reflection of the etalon is not the same if it is measured with two different detectors. For measuring the reflection in the area under 10 % of the reflection value of the antireflexive layers, planparallel tile from low-index glass (BK-7) can be used as etalon.

Measuring the high reflection of light becomes more and more current with rapid development of laser technique and technology. Classic spectrophotometers in the field of reflection measuring has very limited possibilities and maximal correctness of measuring is, in the range of reflection value 90 - 0.5 %.

With aim of improving the correctness of measuring, we usually to spectrometers, which have abilities to measure reflection, add special supplements. The supplements enable measuring the reflection beneath the values from  $\pm 0.1\%$  of correctness. Reflexive supplements are done usually in the principle of manifold reflection where the number of the mirrors that are used is determined by request for measure correctness.

Determining the breaking index of the material of the thin layer has special meaning for quality production of optical coatings.

Spectrophotometric method for determining the breaking index of thin-layer materials is based on measuring the extreme value of reflection of simple coatings and on reading the position of wave lengths, on which the extreme values of reflection are. With change of thickness of applied thin optical coating, the extreme value of reflection changes along the spectrum. By applying the layer of different wave lengths can be measured, so in that way, with several experiments it can be done the dispersion of breaking index of thin layer material on the basis of measuring the reflection of applied layers.

On the basis of calculated errors of breaking depending on given values of breaking index of material ( $n$ ) and breaking index of optically polished grounds ( $n_p$ ), it can be concluded that by spectrophotometric determination of maximal and minimal reflection value of thin layer, the values of thin coating material are determined for all materials with the error limit  $5 \times 10^{-3}$  with favourable choice of ground to which the layer is applied.

That means that the calculated values of breaking index of thin-layer material on the basis of measuring the extreme value of reflection of thin layer material are reliable on the second decimal, and for the certain materials, with favourable choice of the ground, error is not bigger than 0.002.

The advantage of spectrophotometric method is in possibility of its usage in determining the dispersion of breaking index of thin layer material, by applying several different thickness of the layer.

#### 4. CONCLUSION

Optical layers has developed relatively late, but their development has been dynamic and caused the appearance of several kinds of thin-layer elements. Apart from complex theoretical analysis, optical layers demand modern apparatus and the knowledge of technology of applying material onto polished surfaces. The quality level of optically polished surface is 20-30 nm, which points to the fact that optically polished surface is top degree of processing. In comparison to mechanical processing of materials, optically polished surface is for factor  $10^2$ - $10^3$  processed more quality and the surface errors are in molecular dimensions. Thus, the constructor has a great problem to achieve demanding quality of processing with given correctness.

The quality of optical elements is defined by technical documentation in the shape of constructive drawing or special technical directions for accepting the optical elements by production control. In principle, it is possible to estimate the optical element or system so that it does not practically have any mistakes and thus to make it closer to ideal. However, this system is not possible in practice considering the fact that in processing the errors cannot be wholly eliminated. Apart from processing errors, the errors of materials from which optical elements are made are always present in bigger or less measure.

Ideal optical system causes disproportional increase of production expenses. Thus, it is necessary for optical constructors to be familiar with basic things of technology for producing optical elements and on the basis of it to bring in system calculation a certain range of tolerances for errors of material and processing, but not to disturb functionality tolerances of optical elements can be observed from more aspects. The basic parameters which the constructor must bear in mind are the breaking index, Abbe's number of dispersion of the breaking index and material homogeneity, correctness of the processed surfaces, thickness and size of active surfaces, the amount of reflection that is transparency are brought to minimum. On enables making of very precise measure instruments for controlling of optical parameters in the process of element production.

Considering the fact that every optical element in the system brings certain number and size of errors, while calculating the corresponding optical function it is necessary to coordinate total error on the basis of statistic analysis of single errors.

Great efforts are made in the direction of constant improvement and finding new possibilities for realization of optical layers in our country.

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