Measurement of low reflectance coatings on Glass

Using

The Model 191 Series SPECULAR GONIOREFLECTOMETER HEAD

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1.0 INTRODUCTION

Gamma Scientific Model 191 Series Specular Gonioreflectometer Head is an optical head designed to measure the reflectance of specular optical surfaces. When used in conjunction with one of the Gamma Scientific spectroradiometer systems, it will measure the spectral specular reflectance of optical surfaces, both flat and curved, whose thickness is greater than 1.5 mm. Special configurations are available and have been tested in high volume coating production monitoring applications that can measure the first surface coating on curved and flat samples as thin as 0.5 mm. The head may be placed on the surface to be measured or it may be used with an adjustable stand (Option 2), allowing non-contact of the surface to be measured, or inverted stand (Option 3), which allows the head to be inverted and the sample placed on the contact feet.

The Model 191 Series is an optical system with the availability of different configurations that allow optimized performance for different measurement requirements. Each configuration has some type of illuminant, either a tungsten halogen lamp, LED, deuterium or xenon illuminants. Projection optics provides the illumination to the sample. Receiving optics, available with and without collection integrating cavity and polarization analyzers, collects the radiant flux from the projection source after reflectance from the sample. This reflected radiant flux is transferred by means of a fiber optics probe to the D-Series Detectors, either directly or through a spectral selector such as the NM-Series Monochromators, GS-1250, or GS-1290 Series RadOMA™ spectroradiometers, 2020-19B Filter Accessory or a filter either photometric or narrow band.

The illuminating projector microscope, in the most common configuration of the 191 Series, contains the tungsten halogen lamp, a condensing system, and a small slit aperture which is imaged onto the surface of the sample. Other configurations allow measurements in the ultraviolet region by coupling light from deuterium or xenon lamps into fused silica projection optics. Radiant flux reflected from the surface is collected by means of the objective lens of the receptor microscope whose size is such that all the reflected rays of light from a perfect specular surface are intercepted. Elements with positive curvature greater than 4 inches or 102 mm may be accurately measured without adjustment. Coated parts with almost any radius of curvature may be measured even down to less than 0.5 inch or 12.6 mm radius with precise spectral reflectance results when standardized to uncoated parts. The size of the measured sample can be down to less than 0.25 inch, 6.1mm with the adjustable mounting stand, but must be larger than 1.9 inches or 48 mm in diameter to use the measurement plane established by three stainless steel acorn nut feet that can be installed in the metal base of the 191 Series Head. There is an outer circle of alternative mounting points for the three acorn feet which can be used if the sample is larger than four inches in diameter and increased stability is desired. Also available are optional 191C-02 mounting stand for measuring smaller measurement samples or the 191C03 that mounts the Gonioreflectometer inverted allowing measurement samples to be placed on the three measurement plane reference points.

Several configurations for production environments are available that provide an extremely robust optical head machined from a single block of aluminum that provide the same basic measurement capabilities in terms of sample curvature thickness and even smaller sizes. A
popular example that has been used in production line applications is the model 191FW. This model has a main housing that is machined out of a single block of aircraft grade aluminum.

2.0 DESCRIPTION

Figure 1 shows a typical configuration of a 191 Series Gonioreflectometer. Shown in this figure is a Model 191P attached to a Model 191-02 Adjustable mounting stand. The small box just to the left of the mounting stand contains the lamp current monitoring shunt which allows the tungsten halogen lamp regulation to be precisely stabilized when attached to the Model RS-3 power supply (Option 1). The illuminating microscope, on the right side of the figure, is mounted on an arm which moves in a semicircular path in a track with graduations extending from 0 to 60 degrees on either of the surface normal. The range is limited mechanically to 10 to 46 degrees range in incidence and reflectance angles. The illuminating microscope may be locked into position by tightening the thumb screws protruding axially from the center of curvature next to the microscope tube, and may be moved along the semicircular rack by turning the knobs protruding perpendicular to the plane of incidence established by the projection and collection microscope axes. On top of the microscope barrel is an assembly consisting of a condensing lens, a tungsten halogen lamp, a small slit, 0.010"x0.030" (0.25x0.76mm) and within
the barrel, a projection lens. The projection lens images the slit at unit magnification onto the surface of the sample located in the plane of contact of the three stainless steel acorn feet.

The receptor microscope is positioned and moved in the same manner as the illuminating microscope. The upper end for the configuration shown in this figure has polarization analyzer slide and also the eyepiece included with each 191 series head which is used to align the head to the measurement sample when using 191-02 optional adjustable mounting stand.

Figure 2 illustrates the integrating cavity assembly with the rectangular aperture opening on the planar side of the cavity in a 191 configured without the polarization analyzer. The same focal length objective lends as the illuminating side, is used in the receptor microscope, however, its' entrance pupil diameter is larger than that of the exit pupil from the illuminating microscope in order to accept all of the radiant flux reflected from flat or curved surfaces. With a properly aligned 191 Series, the receptor objective lens re-images the projected slit image (on the surface),
into the rectangular aperture field stop in the integrating cavity, such that the second surface reflection is blocked by the remainder of the aperture disc.

The integrating cavity is packed with a polytetrafluoroethylene (PTFE) material for maximum reflectance over a wide spectral region. The radiant flux collected is then transmitted through a hold in the side of the hemispherical integrating cavity into a fiber optics probe, and then transferred to the spectral selector's entrance port. A thumb screw is tightened to secure on end of the fiber optics probe into the cavity housing.

In addition to the hemispherical integrating cavity assembly, a separate adapter is provided with the 191D and 191P that couples the fiber optic probe directly into the end of the collection microscope. As with a properly aligned 191 Series using the integrating cavity, the receptor objective lens re-images the projected slit image (on the surface), through the rectangular aperture field stop. The direct coupling adapter allows more light to enter the Monochromator thus allowing the use of semiconductor detector to extend the wavelength measurement capability further into the near infrared than photomultipliers can measure. There is, however, a disadvantage since the use of this adapter is more sensitive that the integrating cavity to changes in the radius of curvature the surfaces being calibrated against and measured. When measuring antireflection coatings using this adapter, it may be necessary to calibrate the spectral measurement system with an uncoated sample with the same radius of curvature as the coated pieces.

![Figure 3](image.png)

**Figure 3** The 191P configured with collection integrating cavity coupled to a fiber optic light guide installed in the calibration and measurement position.

**Figure 3** shows the 191 configured with collection integrating cavity coupled to a fiber optic light guide installed in place of the eyepiece. The 700-3 Series fiber optic light guide would normally couple to one of the NM series monochromator or the FP-X-X.X Series couples into the GS1250 and GS-1290 series of spectroradiometers.
**Figure 4** shows a close-up of the 191P, previously shown in Figure 1, which includes the added ability of measuring the energy in either the S or P plane of polarization (the electric field of vector perpendicular (Senkrecht in German) or Parallel to the plane of incidence). The receptor microscope is positioned and moved in the same manner as the 191 Series described above. Either of the two polarizers can be inserted into the optical path by sliding the dented holder either direction. This exposes either a "S" or "P" indicating which polarization orientation is transmitted. The center position is a clear aperture which gives measurement results identical to the standard configuration of the 191.

Separate calibration files must be created for the S and P polarization measurement modes based on the Fresnel first surface reflectance laws. This can easily be done with any of the Gamma Scientific spectral analysis software packages with the calibration files on the disk supplied with this manual.

For verification of alignment, the upper portion of the integrating cavity can be removed by pulling on the knurled screw allowing access to the rectangular aperture. An alignment eyepiece, as show in Figure 1, and a neutral density filter is included with the 191 Series/P and may be inserted in the same cylindrical housing vacated by the hemispherical cavity, and this alignment eyepiece may be focused sharply on the integrating cavity's aperture by rotating the upper portion of the eyepiece. When the lamp is on, the bright image of the slit is now seen within the area of the rectangular aperture and the second surface reflection, which is on the side closest to the surface normal, is blocked by the upper portion of the aperture disc. If the second surface is visible in the rectangular aperture then both surfaces will be included in the measurement.
The 191 Series when placed on the surface should have the angle of incidence for the illuminating microscope set equal to the angle of reflection for proper use. The 191 Series is adjusted at the factory for proper alignment on a flat surface, however, it can be verified using the above technique.

The overall system configuration is shown in the illustration below. This configuration shows the scanning grating family of products, NM-3 or NM-5 single grating spectral selectors or the NM-7, NM-9 or NM-10 double grating spectral selectors. The GS-1250, GS-1290 family of array detector based spectroradiometer can be used here also and are the normal choice for production monitoring applications.

Figure 5 System configurations of the variable angle 191C gonioreflectometer optical head configured with a NM series scanning spectroradiometer which is equivalent to the GS-1250, and GS-1290 Array detector based spectroradiometers. The fixed angle Model 191FW provides the same geometry as the 191C set to a 10 degree angle but in a more robust package for production applications.
3.0 MEASUREMENT PROCESS AND RESULTS

Typical results from the measurement of antireflection coating are shown in this section and are compared with measurements made with second surface having an index matched liquid between the back surface and a piece of black absorption filter material.

3.1 Measurement process

The first step in measurement process is to calibrate the reflectance measurement scale of the spectroradiometer connected to the gonioreflectometer optical head for the chosen angle of incidence. This is accomplished by using the known physical properties of glass materials and the known physical properties of light interaction with materials. The glass material used as the standard reference to establish the scale of reflectance is Schott BK7 optical glass. The spectral index of refraction of this glass from this manufacturer is known to a high precision and is held to a tight tolerance; BK-7 was chosen because of the negligible change in the index of refraction from melt to melt, +/-0.001 for 546.1 and 587.6 nanometers and the index within a single melt varies less than +/-0.0001 for 587.6 nanometers. Using the known spectral index of refraction and the known characteristics of how light behaves at the intersection of two planar materials, each with a different index of refraction described by Snell’s Law and the Fresnel formulae, the spectral reflectance of BK7 can be determined to a low uncertainty.

The reflectance of BK7 at a 30 degree incidence angle over a 360 to 940 nanometer range is shown below.

The scale for this plot is 0.042 to 0.0465reflectance equals 4.2% to 4.65 % reflection.
Looking at the plot relative to a 1.0 reflectance or 100% reflection shows the magnitude of the BK7 reflectance relative to what a perfect mirror reflecting surface would give.

Now the BK7 is placed in the measurement plane of the Model 191 Gonioreflectometer optical head and used to standardize the measurement instrument.
3.2 Measurement Results
The data below is the same BK7 sample used to calibrate, measured as an unknown. This step is taken to illustrate that a valid calibration has occurred. The difference between the calibration standard data and the measured data shown in this plot is 0.14% different in the visible portion of the spectrum.

Now the first surface of the coated sample, in this case an antireflection (AR) coating, can be place in the measurement plane and the spectral data acquired as shown on the next page.
This plot shows the Anti-reflection coating with the uncoated BK7 data and the plot scale set to cover 0 to 0.15 reflectance or 0% to 15% reflection.
The Logarithmic scale plot shown below gives a more defined image of the low reflectance regions of the Anti-reflection coating.
Some Anti-reflection coated products can have coating on both sides of the glass article and this second side coating can be easily measured by flipping over the sample and measuring the back side first surface. For this sample, the back side spectral reflectance is shown below and on the next page on a logarithmic scale plot.
Logarithmic scale plot of BK7, an Anti-reflection coated glass plate and the coated back side of the glass plate reflectances.

Now with this data the spectral reflectance of both surfaces can be predicted to a first order by simply adding these two spectral reflectance data files of the front and back surfaces. The only difference between the sum and the measured reflectance of both surfaces is the spectral transmittance losses or fluorescence gains from light passing through the glass substrate material.
The resulting spectral reflectance obtained by adding the front and backside reflectances is shown below.

From this plot you see that there is only a small region of wavelengths between 640 and 700 nanometers that has a lower reflectance than the uncoated BK7 single first surface reflectance.
The difference between the both surfaces measured and the sum reflectance of the two individual surface measurements is shown in next plot and shows the measured both surface reflectances lower than the sum of each surface reflectance; this difference is a result of the attenuation of the glass material.
If the both surface measured reflectance function is divided by the summed each surface reflectance function the result is the spectral transmittance of the glass substrate which is shown below.
The next step involves the temporary contact of a black absorption filter glass such as Schott RG1000 to the back side of the coated substrate using an index of refraction matching liquid. This will simulate the use of the coated substrate in the final application. In this condition the first surface alone was once again measure and both surfaces measured with the bonded black absorption filter material. The results of these measurements are shown in the plot below showing the advantage of the antireflection coating.
4.0 CONCLUSION

In this brief overview of the Gamma Scientific Model 191 Gonioreflectometer Spectroradiometer systems only a portion of all of the capabilities for material and process control and analysis of presented. In the over 50 years of Gamma Scientific providing comprehensive optical radiation measurement systems and instruments, the spectral gonioreflectometer systems have been used to measure not only antireflection coatings for spectral efficiency but also other optical performance metrics such as:

- color reproducibility
- coating uniformity when coupled to an automated motion system; gantry or robotic arm
- coating thickness
- high reflectance coating characterization
- Solar panel coatings

The customer set for these myriad of measurements include the world’s best companies whose focus on quality sets them apart from others. They include:

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Boeing
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Lockheed Martin
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