

Study of the performance of optical-grade germanium infrared materials using various optical characterization techniques

Steven Brantegem, Heidi Ottevaere, Virginia Gomez, Erik Stijns, Hugo Thienpont, Igor Romandic*, Thierry Vandeveldel*, Dirk Vyncke*

Vrije Universiteit Brussel, Dept. of Applied Physics and Photonics (TW-TONA),
Pleinlaan 2, B-1050 Brussels, Belgium
tel.: ++32 2 629 18 13, fax: ++32 2 629 34 50, e-mail: sbranteg@vub.ac.be

* Umicore Electro-Optic Materials, Watertorenstraat 33, B-2250 Olen, Belgium
tel.: ++32 14 24 57 00, fax.: ++32 14 24 55 34, e-mail: optics@umicore.com

Abstract

Optimization of the optical quality of optical-grade germanium components requires an in-depth investigation of the different contributions to the optical loss in germanium. In this paper we therefore focus on this optical characterization. We give an overview of possible characterization techniques to determine surface roughness and surface/bulk absorption and we highlight the obtained optical characteristics. To conclude we select the most appropriate non-destructive characterization tool for each optical parameter.

Introduction

In a lot of applications, optical resolution demands for infrared (IR) imaging systems are steadily increasing. Therefore the performance of optical elements must also improve. Unarguably, germanium (Ge) remains the material of choice for high performance thermal imaging in the 8–12 μm spectral range. The IR-grade Ge single crystals, from which the optical elements are made, are usually produced by the Czochralski crystal-pulling process. This process has become the dominant technique for the production of Ge blanks [1]. But, because Ge single crystals intended for IR optical applications are grown along the [111] crystal direction, these crystals are not dislocation-free. We present in this paper a way to measure the two main optical loss mechanisms. Knowledge of these loss contributions will give us useful information on both the effect of surface finishing (grinding, polishing and coating) and the absorption losses. Even more important, it will give us an indication of how to improve these processes. The separate contributions of the surface and bulk absorption in Ge have not been characterized to date. To characterize these loss contributions, we will select the most appropriate measurement techniques. We prefer to use only non-contact optical methods, because of their non-destructive character. Roughness and absorption will be briefly discussed and an overview of the results will be presented as well.

Determination of surface roughness

Surface roughness is generally known as a measure of the topographic relief of the surface and as a consequence an extremely determinative parameter for the quality of optical elements. In this section we will mainly deal with two methods to measure this surface profile, namely “optical non-contact surface profilometry” and “angle resolved scattering” (ARS). The first technique makes use of a commercial WYKO NT2000

(Fig. 1a) and is based on a Mirau interference microscope. For the second technique, ARS, we built a dedicated lab setup (Fig. 2a) and this technique is used as a supplementary alternative. All samples, used in this characterization study, are polished, have a diameter of 40 mm and a thickness of 5 mm. The study of rough samples is refrained from this paper because of less importance for high-quality optical elements.

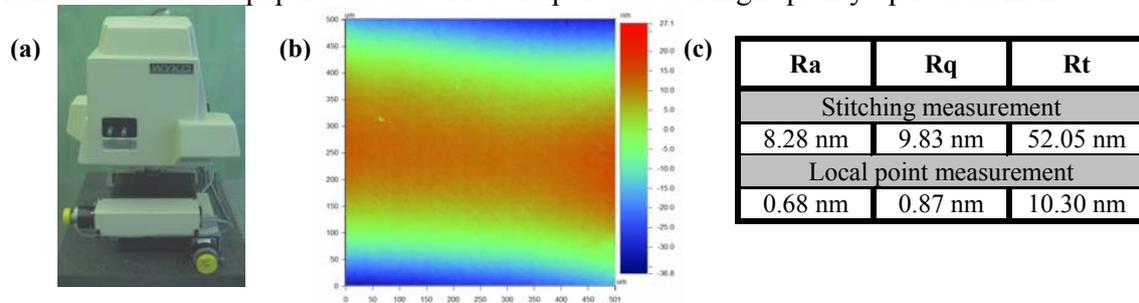


Figure 1: (a) WYKO NT2000 (b) 2D surface roughness map (c) Resulting roughness values with Ra = Average Roughness, Rq = RMS Roughness, Rt = Peak-to-Valley Roughness

The surface profiler system uses two approaches to measure a wide range of surface heights. We will restrict ourselves to phase-shifting interferometry (PSI) only, because this approach allows measuring nearly smooth surfaces ($0.1 \text{ nm} < \text{heights} < 160 \text{ nm}$). The applied 50x microscope objective offers a total effective magnification of 102.4x, if we take the field of view (FOV) multiplier to be 2. This is the maximum magnification that we can obtain with the profiler installed in our lab. The larger the magnification (102.4x) the better the lateral resolution (X: $0.20 \mu\text{m}$, Y: $0.17 \mu\text{m}$), but this will impose restrictions on the size of the measured area. Therefore a stitching function makes it possible to characterize a larger field of view without switching to a lower magnifying objective. This preserves the high lateral resolution required to image small features within a large area. However, to reduce the computation time we select an area of $500 \times 500 \mu\text{m}^2$ to be measured with the stitching technique. An example of a contour plot obtained on a $500 \times 500 \mu\text{m}^2$ area is shown in figure 1b. The results are given in figure 1c. Compared to a local point measurement ($46 \times 60 \mu\text{m}^2$) the values obtained with stitching ($500 \times 500 \mu\text{m}^2$) are higher. This is because large areas also show considerable waviness.

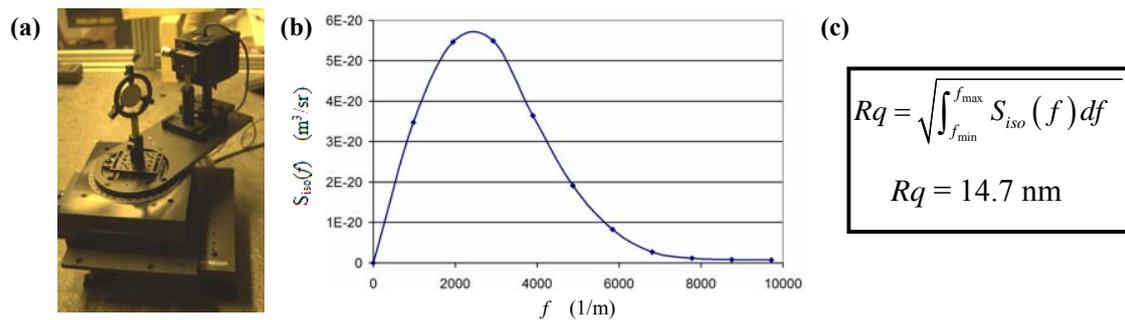


Figure 2: (a) Scatterometry lab setup (b) Isotropic PSD (c) RMS Roughness resulting from (b)

As an alternative technique we have also investigated ARS. The terms most useful in defining and measuring scattering are collectively called BSDF (Bidirectional Scatter Distribution Function). We will concentrate on the more common BRDF, which is a full description of the angular distribution of the reflected scattered light. It assumes an incident light beam at a fixed angle of incidence with respect to the Ge crystal. The BRDF value at each scatter angle is the scattered light power at that angle, divided by the

incident light power and includes various geometric factors. Here, the BRDF is measured by sweeping a single detector along an arc (Fig. 2a). The BRDF is essentially proportional to a roughness characterization function known as the power spectral density (PSD) function (Fig 2b). Except for a change in units and the fact that it is plotted against spatial frequency f instead of scattering angle, a PSD plot looks like the corresponding BRDF plot. Because germanium is an isotropic material, we can use the isotropic PSD function to calculate the roughness. The isotropic PSD has the feature that the surface RMS roughness can easily be found by integration from f_{min} to f_{max} . The RMS roughness is equal to the square root of the calculated area under the curve (Fig. 2c) [2].

We see that the measured RMS surface roughness obtained on the same sample using the WYKO (9.83 nm) and ARS (14.7 nm) are in good agreement, but in order to make an extensive comparison between both measurement techniques, the spatial frequency bandwidth limits of both methods have to be taken into account.

Characterization of absorption

In this section we will present two methods that allow us to measure the absorption of Ge. We will compare them and select the most appropriate technique that fulfils our demands. The most widely adopted method for determining optical absorption losses in the infrared region is laser calorimetry. Important to know is that, for calorimetry purposes, the Ge samples need all to be two-sided AR coated. Thermocouples are attached to the sample edge and, for a given incident laser power coming from a CO₂ laser, the thermal rise (laser ON) and fall (laser OFF) as a function of time is registered. The magnitude of thermal rise in the sample is then proportional to the absorption coefficient α of the sample [3].

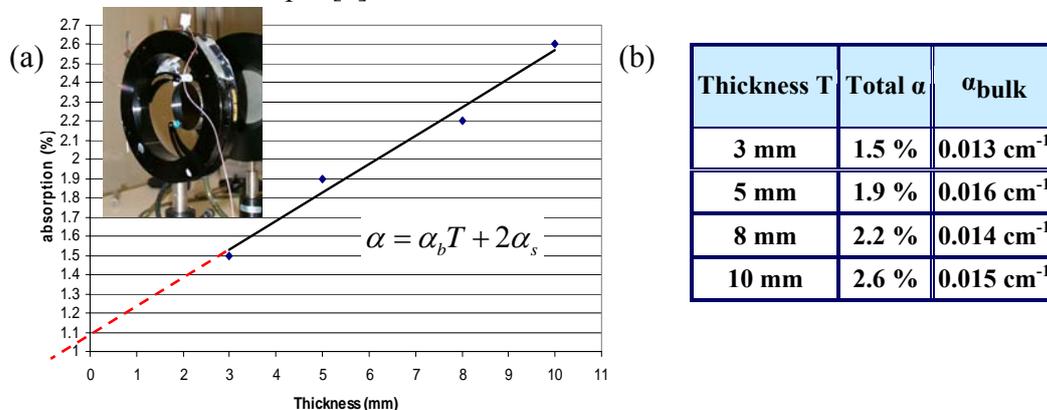


Figure 3: (a) Separate contribution of bulk and surface to the total absorption value
(b) Measured absorption values ($2\alpha_s = 1.1\%$)

To separate the contribution of the bulk and the surface, the total absorption coefficient is measured as a function of the sample thickness T . Plotting α vs. T for four different thicknesses results in a linear behavior where the slope is equal to the theoretical bulk absorption and the intercept is equal to twice the surface absorption (Fig. 3a). This method is only valid if we assume that all the sample surfaces are identical and have a superior optical finish. This is accomplished by polishing and coating all the samples at the same time under exactly the same conditions. The main advantage of calorimetry is the ease and the speed with which we can obtain absolute absorption values (Fig. 3b). Also the possibility to separate the contribution of the bulk and the surface makes

calorimetry a first choice measurement technique. However information about local absorption points can not be obtained. Therefore we have studied photothermal deflection spectroscopy (PDS), a relative technique, where the absolute absorption value can not be derived directly.

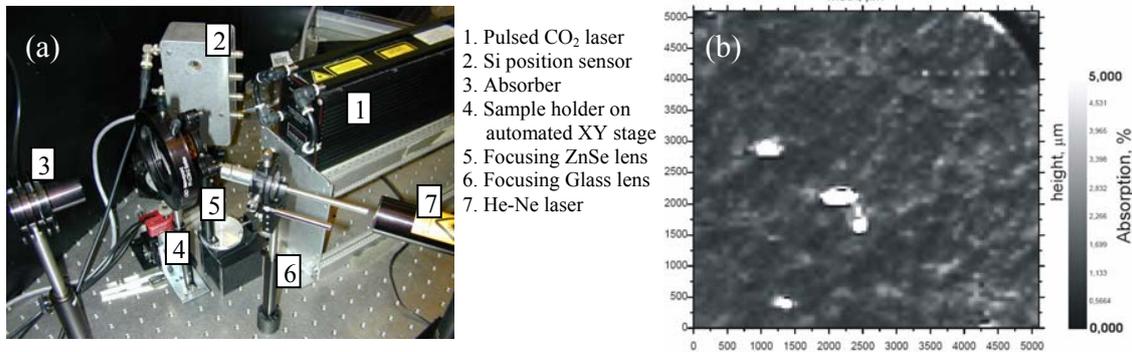


Figure 4: (a) PDS lab setup (b) Result of an absorption measurement with PDS

Let us first take a closer look to the lab setup (Fig. 4a). A modulated CO₂ pump laser beam creates a thermal diffusion wave inside the sample. This thermal oscillation penetrates the sample and causes a modulated change in its refractive index right above the irradiated region. A He-Ne probe laser beam traveling parallel to the surface will be subsequently deflected. This deflection is measured by using a Si position sensing detector. The amplitude and phase of the deflected probe beam carry information about the absorption of the material [3]. The pulse rate of the CO₂ laser will affect the thermal diffusion length. Low frequencies will make absorption spots, which are deeper into the bulk, more visible. High frequencies dim faster and give only information about absorption spots near the surface. As mentioned above, PDS can only compare the absorption of local areas with each other on the same sample surface (Fig. 4b). Another drawback is the amount of time (17 h) needed to scan an area of 5 x 5 mm², which is intolerable from an industrial point of view. PDS is a good complement to calorimetry if information is needed about the absorption distribution along the sample surface.

Conclusion

We have demonstrated several techniques to determine two decisive parameters for the evaluation of the optical quality of germanium blanks: roughness and absorption. Advantages and disadvantages of all methods have been discussed and as a result we have selected the most appropriate measurement technique. This pointed out that optical non-contact surface profilometry and calorimetry are the two methods of preference for determining surface roughness and absorption respectively.

Acknowledgements

This work was supported by IWT in the context of a cooperation between the VUB and Umicore NV.

References

- [1] I. De Ruijter, "Germanium enters a new era", *Photonics Spectra*, pp. 55-59, July 1987.
- [2] J. Bennett, L. Mattson, "Introduction to surface roughness and scattering", OSA, pp. 20-35, 1999.
- [3] U. Willamowski, "Measuring the absolute absorptance of optical laser components", *Applied Optics*, pp. 8362-8370, 37(36), 1998.