



<b>Publication Year</b>	2018
<b>Acceptance in OA</b>	2020-10-26T15:30:49Z
<b>Title</b>	Si/SiO <sub>2</sub> -based filter coatings for astronomical applications in the IR spectral range
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<b>Publisher's version (DOI)</b>	10.1117/12.2311310
<b>Handle</b>	<a href="http://hdl.handle.net/20.500.12386/27993">http://hdl.handle.net/20.500.12386/27993</a>
<b>Serie</b>	PROCEEDINGS OF SPIE
<b>Volume</b>	10691

# PROCEEDINGS OF SPIE

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**SPIE.**

Event: SPIE Optical Systems Design, 2018, Frankfurt, Germany

# Si/SiO<sub>2</sub>-based filter coatings for astronomical applications in the IR spectral range.

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## ABSTRACT

Order sorting filters had to be coated for the Cryogenic Infrared Echelle Spectrograph upgrade (CRIRES<sup>+</sup>)-instrument, a high-resolution IR spectrograph to be set up at ESO's Very Large Telescope in Chile. Therefore SiO<sub>2</sub> was chosen as material with low refractive index. Si and Ge have been investigated as materials with high refractive index, whereby Si has been chosen for the application of the coating. Three types of high-pass filters were deposited with transmission bands starting at 0.96 μm, 1.47 μm and 2.9 μm. These filters need to block effectively all wavelengths between 0.5 μm and the respective band. Therefore, in the blocking range, an optical density above four, or above three for the filter starting at 2.9 μm respectively, had to be achieved. The filter-coatings also needed to survive thermal cycling down to 65K while only introducing a small wave front error. The lower total thickness, compared to coatings consisting of other materials, and the low film-stress are favorable properties for coatings deposited onto prisms and other more complex optical components.

**Keywords:** SiO<sub>2</sub>, Si, silicon, high-pass filters, magnetron sputtering, CRIRES<sup>+</sup>

## 1. INTRODUCTION

The search for exoplanets is a current focus of astronomical research and brings with it a need for precise and high resolving measuring instruments. CRIRES<sup>+</sup> will be the improved version of an already existing device, which is going to be commissioned at the Very Large Telescope (VLT) in 2018. This scientific instrument will allow the confirmation of transit exoplanet candidates orbiting cool stars. The atmospheric characterization of transiting planets and the study of the origin and evolution of stellar magnetic fields are further research foci. After the upgrade, this instrument is a cross-dispersed spectrograph increasing the observing efficiency by a factor of 10. Therefore, three filter substrates and several mirrors have been coated at Fraunhofer IOF by means of reactive magnetron sputtering. The filters, which are the subject of this paper, are covering a wavelength range from 960nm to 5300nm, covering the optical Y+J-, H+K- and L+M-bands. The blocking of transmission had to be realized from a wavelength of 500 nm upward to the edge of the particular filter, starting at 0.96 μm, 1.47 μm and 2.9 μm, respectively. The filters and hence the optical coatings will be cooled down to approximately 65K in operating conditions and have to resist this temperature change and the thermal cycling multiple times without fatigue or a change of the optical properties. The wave front error of the filters had to be below 50nm rms. The optical density (OD) in the blocking range of the YJ and HK filters has to be above four and above three for the LM-filter, for a beneficial signal to noise ratio [1], [2].

The coatings have been applied by magnetron sputtering with IOF's PreciCoat, which is an inline sputtering device with eight cathode stations, three for DC sputtering, four stations that are operated as dual magnetrons and one with an ion source. The whole system is cryogenic pumped, with an additional turbomolecular pump for reactive processes. Substrates with dimensions up to 500 mm x 500 mm and 100 mm in height can be processed. The device is built in a top down sputtering setup and without the possibility for optical or quartz crystal monitoring.



Figure 1. PreciCoat magnetron sputter device at IOF

## 2. PRELIMINARY TESTING

### Selection of coating materials

Choosing the right material for a coating is an important issue. In general, to utilize the interference effect one material with a refractive index as low and one with a refractive index as high as possible is needed [3], [4]. For this application,  $\text{SiO}_2$  has been chosen as low index material, because it is a well-known coating material with a low extinction coefficient in a wide range of the desired transmission section. Si and Ge have been evaluated as candidates for the high index material. Theoretical values for the bulk materials show, that both have a high refractive index but unfortunately the deposited amorphous layers have a higher than desirable extinction coefficient [5]. On one side, Si is the material with a lower extinction coefficient, but a slightly lower refractive index. On the other side, Ge has the higher refractive index, which would be desirable to achieve a higher contrast in refractive index and therefore a lower total physical thickness of the coating. Yet, the extinction coefficient nearby the cut on wavelength of the YJ1-filter is already high. Hence, samples with single layers of all three materials have been prepared for a detailed investigation.

### Determination of optical constants

All samples have been measured under  $6^\circ$  and  $60^\circ$  AOI in a range from 200nm to 2500nm by means of a Perkin Elmer Lambda UV/VIS-spectrophotometer [6], as well as in a range from 1300nm to 25000nm using a Perkin Elmer Fourier Transform IR spectrometer [7]. These experimental data were used to calculate the optical constants for the materials with the software "LCalc" [8]. After that first coating designs, which would meet the specifications for the filters, were created. Afterwards it became clear, that the absorption of the high index materials would be a challenge. The comparison of literature values [5] of both materials to the deposited materials can be seen in figure 2. Especially the Ge samples exhibited a higher than expected extinction coefficient, which made it impossible to meet the specifications for the coatings, even with theoretical designs.

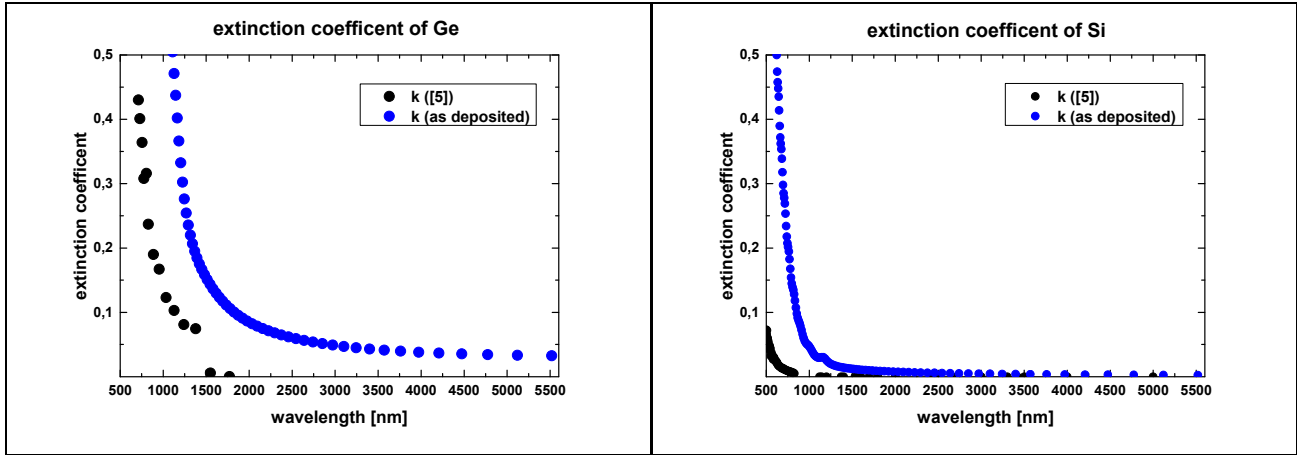


Figure 2. Extinction coefficient of Ge and Si as deposited compared to values found in literature

### Modifying the optical properties

In order to reduce the extinction coefficient of the high refractive index coating materials a pretreatment of the materials by annealing was investigated. Two temperatures  $t_1$  and  $t_2$  have been tested. The temperature cycles consisted of a heat up, hold and cool down period in a timeframe of several hours. Thereby, the annealing temperature should have been kept as low as possible, to prevent the substrates from being damaged. Afterwards the samples have been examined as described above. Due to the annealing the extinction coefficient and film stress of the Si- and Ge-layers were reduced, but also the refractive index [9]. The effect of the annealing process on the optical constants of the high index materials is shown in figures 3 and 4.

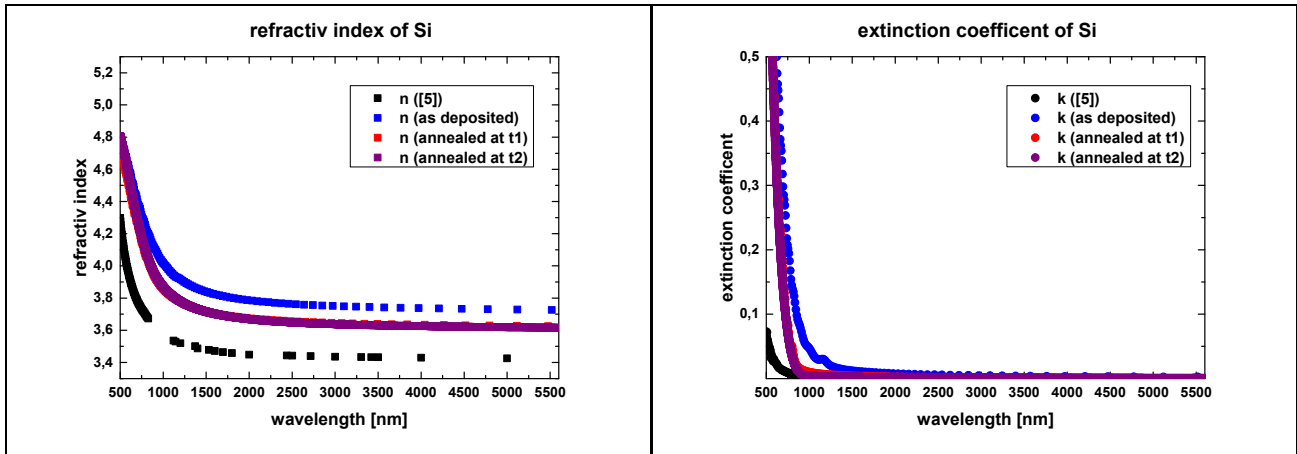


Figure 3. Comparison of the optical constants of Si before and after annealing

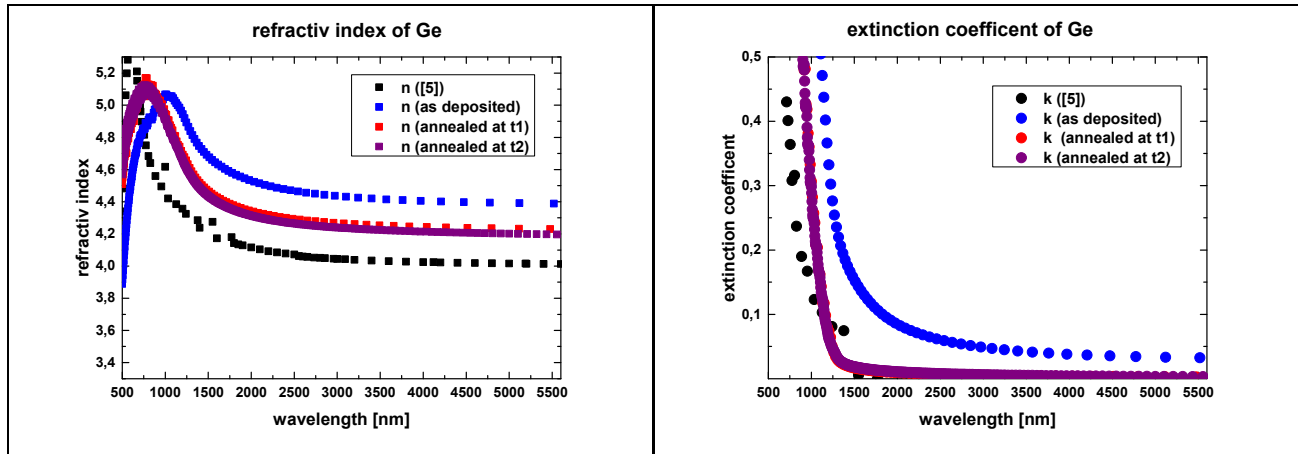


Figure 4. Comparison of the optical constants of Ge before and after annealing

Contrary to Ge and Si, the influence of the annealing process on the optical constants of the SiO<sub>2</sub>-layer was below the measurement accuracy. Only a decreased film stress could be measured.

### 3. COATING OF THE CRIRES<sup>+</sup>-FILTERS

#### Coating design

The obtained optical constants have been used to create theoretical coating designs, and it became clear, that Si would be the high index material of choice. Even with the reduced extinction coefficient, it has not been possible to find a theoretical design, which meets the specified transmission criteria, when using Ge as high index material. Overall, the main design objectives were to keep the total physical thickness as low as possible, to reduce the risk of particle generation due to ablation of material coming from the wall of the recipient, and to avoid extremely thin layers in the design. Regarding the optical performance, an OD as high as possible was a main goal, in order to achieve a signal to noise ratio as good as possible. Simultaneously first deposition tests with thick coatings were carried out and sent to an ESO facility, for a cool down test to operating temperature. After the tests, the samples were sent back to IOF for microscope analysis. A noticeable ablation of the coating at the edge of the substrate (see figure 5) could be attributed to water contained in the coating. The SiO<sub>2</sub>-layers have been identified as the source of the problem, resulting in an adjustment of the respective coating parameters. With the modified process, these defects were not found any more after cool down tests.

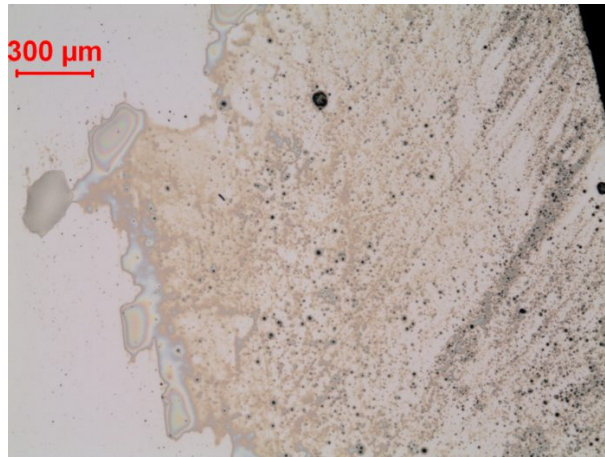


Figure 5. Ablation of the coating near the edge of the sample after cool down to cryogenic temperature

### Coating process

The filters themselves have been coated in two tranches, for reasons of risk management. Due to the length of the process, there was an increased risk of particle generation in the coating device. Therefore, the Si- and SiO<sub>2</sub>-layers have been deposited from different target positions. After the successful delivery of the first tranche, slight changes in the transmission spectrum for the second LM-filter were implemented, to fulfill requests for minor adjustments by ESO. In addition, IOF had an improved design for the coating of the second YJ-filter. Hence, five different filter designs were realized. Especially the optical density in the blocking range is noticeable, considering the width of the covered spectral range. Figure 6 shows as an example a measurement of the transmission and the optical density of a witness sample for the first HK-filter.

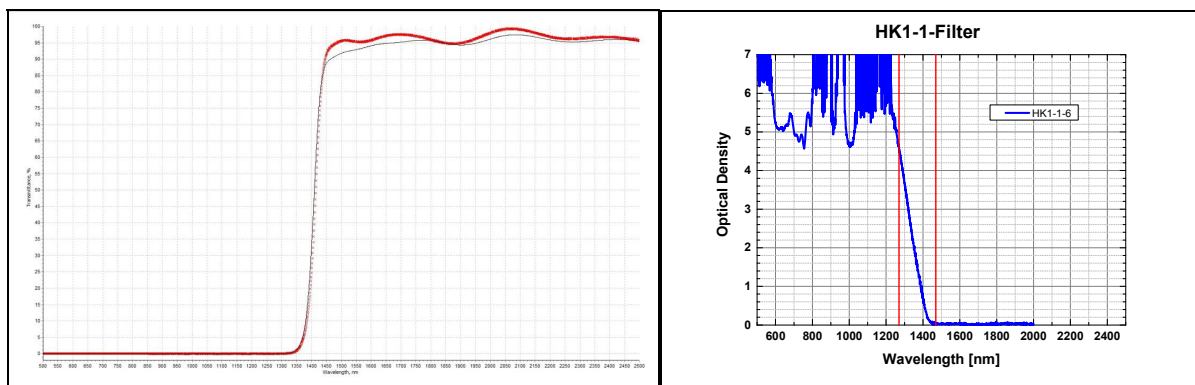


Figure 6. Transmission measurement (red dots) compared to design curve from the first HK-filter at 6° AOI (left); measurement of optical density (right)

## 4. CONCLUSION

Six filters have successfully been realized, for the CRIRES<sup>+</sup>-project. Therefore, Si and Ge have been examined as high index materials, resulting in the choice of Si for the final designs. The optical constants of the materials have been modified by annealing. Thereby the extinction coefficient and film stress of the Si- and Ge-layers could be reduced, but also the refractive index. Regarding the optical constants of the SiO<sub>2</sub>-layers, no changes were detected due to annealing,

yet the film stress of the SiO<sub>2</sub>-layers also has been reduced. After the first cool down test, deposition parameters for the SiO<sub>2</sub>-layers have been adjusted to prevent the ablation of the coating due to the small amounts of water in the coatings. Nearly in the whole transmission range, a transmission higher than 95% could be achieved. The target values of the optical density of four respectively three have been achieved and surpassed in the whole blocking range for nearly all filters (see figure 6). Realizing an OD as high as possible was more important than increasing the transmission by 1-2%. Because the filters had to be annealed to achieve the specified values, it is clear that they can withstand a temperature range of several hundred degrees.

## ACKNOWLEDGEMENTS

The authors are thankful to Dr. Olaf Stenzel, Lisa Akhmadeeva and Vera Forwerk, for the measurements and the calculations to determine the optical constants of the coating materials.

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