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Title: **Optical properties of hot-pressed B_4C in the EUV**

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Abstract

Hot-pressed B_4C is found to have a high normal reflectance in the extreme UV spectral region above 49 nm. This reflectance is comparable to or higher than chemical vapor deposited SiC in the spectral region from 49 to 92 nm. Reflectance measurements as a function of the angle of incidence were used to obtain the optical constants of B_4C in the spectral range 49-121.6 nm.

OCIS Codes: 120.4530 (Optical constants), 120.5700 (Reflection), 230.4040 (Mirrors), 260.7210 (Ultraviolet, far), 260.7200 (Ultraviolet, extreme)

1. Introduction

Optical coatings and materials in the spectral region below the MgF_2 and LiF absorption cutoff wavelengths of 115 nm and 102.5 nm, respectively, that will be referred to as extreme ultraviolet (EUV), have relatively low normal reflectance. Mirrors with high reflectance in the EUV are necessary for both earth and space science applications because important spectral lines lie in this spectral region. Observation of the universe in these spectral lines is usually complicated by the low intensity of the sources and by the fact that observations have to be made outside the terrestrial atmosphere.

Different reflective materials are used for astronomy or atmospheric physics observations in the EUV below the MgF_2 and LiF cutoff wavelengths. Polished chemically vapor deposited (CVD) SiC is the material with the highest normal reflectance in the EUV down to 60 nm[1], with values around or above 40%. Thin films of different materials, like SiC prepared by magnetron sputtering[2] and by ion-beam-deposition (IBD) [3], and IBD B_4C [4] (with somewhat lower reflectance), and Ir [5] (with a significantly lower reflectance) are often advantageous because they involve low substrate temperature processes and are cost-effective. The development of new reflecting materials in the EUV is highly desirable not only for astronomy applications, but also for synchrotron radiation devices, plasma physics, free electron lasers and spectroscopy. In this paper we show the reflectance and optical constants of a new EUV material: hot-pressed B_4C.

2. Experiment

Samples of hot-pressed B_4C were provided by Pure Tech Inc. They were made following the same process used to prepare hot-pressed sputtering targets: 99.5% purity B_4C powder is compressed under a force of 3000 psi at a temperature exceeding 2270 K. Hot-pressed B_4C samples were 25 mm in diameter. Samples were polished by General Optics. Roughness measurements were made using a WYKO TOPO 3D. Rms surface roughness was 0.37 nm averaged over three locations.

The reflectance measurements in the EUV were made with a reflectometer-monochromator system described elsewhere[6]. Samples were cleaned before reflectance measurements with Alconox and water solution and rinsed with distilled water and doubly distilled acetone.

3. Optical constant calculation

Radiation is generated in the lamp supposedly with no preferential polarization axis, and is therefore unpolarized at this stage. However, partial polarization is introduced in the monochromator as an unwanted effect originated in the beam reflection at the diffraction grating. This results in a partially polarized radiation beam impinging onto the sample. The influence of polarization on the reflectance can be described by a single parameter that will be referred to as the degree of polarization *p*, which was a known parameter of the reflectometer, as will be described below. *p* is defined as:

$$p = \frac{I_p - I_s}{I_p + I_s} \qquad (1)$$

where I_p and I_s indicate the fraction of the incident intensity with the electric vector parallel and perpendicular, respectively, to the plane of incidence. In our reflectometer we arbitrarily defined p referred to the horizontal plane of incidence (actually, all planes of incidence can be covered by rotation of the sample holder-detector chamber with respect to the incident beam). Therefore, for reflectance measurements performed in the vertical plane of incidence of our reflectometer, I_p and I_s are interchanged, and p turns into $-p$.

The reflectance at the boundary between vacuum and the medium to be investigated is given by:

$$R = \frac{1+p}{2} R_p + \frac{1-p}{2} R_s \qquad (2)$$

where R_p and R_s are the reflectance for p and s polarization, i.e., they equal the square modulus of the amplitude reflectance for parallel and perpendicular incidence, respectively, given by the well-known Fresnel equations. Since no efficient polarization devices are available in the 50-120 nm spectral range, we performed reflectance measurements with the partially polarized beam, and accounted for the degree of polarization of the beam in the interpretation of the experimental data.

The effect of roughness onto the reflectance was taken into account through the Debye-Waller factors:

$$R_{s,p}{}^{*} = R_{s,p} \ \exp\left[-\left(\frac{4\pi\sigma \cos\theta}{\lambda} \right)^2 \right] \qquad (3)$$

where $R_{s,\ p}$ gives the reflectance of a smooth boundary as calculated with the Fresnel equations, and $R_{s,\ p}^{*}$ describes the reflectance of the real non-smooth boundary, parameterized through the rms roughness of the boundary, σ. θ and λ stand for the angle of incidence referred to the boundary normal, and the radiation wavelength, respectively.

The complex refractive index $\boldsymbol{n}=n+ik$ of hot-pressed B_4C was determined by means of a random search of the pair (n, k) using a least square algorithm that minimized the sum of the squared residuals between the reflectance measured at every incidence angle and the calculated fit:

$$s_j^{\ 2} = \sum_{i=1,\ldots,m} \left\{ R^{\exp}{}_{\theta(i)} - R\left[\theta(i), n_j, k_j, p\right] \right\}^2 \qquad (4)$$

where $R^{exp}{}_{\theta(i)}$ is the reflectance measured at the angle of incidence $\theta(i)$, and $R[\theta(i),n_j,k_j,p]$ is the reflectance calculated with Eq. 2 for the trial set (n_j, k_j) of optical constants in the iteration j, and for the partial polarization of the beam p as defined in Eq. 1. The summation in Eq. 4 was extended to the 5 angles of incidence at which reflectance was measured. They were 15º, 30º, 45º, 60º and 75º, all of them in the horizontal plane of incidence of the reflectometer. Measurements at 15º were also performed in the vertical plane of incidence, in order to show an average reflectance that is independent of the beam polarization, as will be described in Section 4. Minimization in Eq. 4 gave us the pair (n, k) that better fitted the reflectance measurements and this pair was taken as the optical constants of B_4C. The advantage of optical constant determination using a multi-angle reflectance fitting over the Kramers-Kronig method is that the former does not need any data or model on the reflectance or optical constants of the material outside the spectral region investigated. This advantage is definitive for new materials, like hot-pressed B_4C, which have not yet been fully investigated.

The degree of polarization *p* of the monochromator was a known wavelength-dependent parameter in the minimization. It had been previously determined using reflectance measurements versus the angle of incidence taken on float glass samples. Reflectance measurements were performed in two perpendicular planes of incidence: the horizontal and the vertical plane of incidence. A least square algorithm similar to the one described in Eq. 4 was used to obtain *p* and, in addition, the complex refractive index of glass. Measurements in 2 perpendicular planes of incidence were necessary in order to obtain the partial polarization of the beam; however, measurements in only one plane were sufficient for optical constant calculation when *p* is known. Once *p* was obtained for every wavelength, it was considered a constant parameter of the monochromator.

4. Results

Reflectance measurements at 15° from normal were measured in 2 perpendicular planes of incidence, as explained in Section 3. The average of both is represented in Fig. 1 in the spectral region 49-200 nm. This average equals the reflectance that would be obtained with unpolarized radiation (p=0), i.e. $(R_s+R_p)/2$, and is therefore independent of the partial polarization of the beam. This result is straightforward from Eq. 2 by averaging the reflectance obtained in one plane of incidence with that obtained in the perpendicular plane of incidence, where *p* changes to –*p*, as stated in Section 3.

Reflectance measurements at 5 incidence angles ranging from 15° to 75° were used to obtain the optical constants of hot-pressed B_4C, as was described in Section 3. The optical constants of hot-pressed B_4C so obtained are shown in Table 1 and in Fig. 2.

5. Discussion

Fig. 1 compares the near normal reflectance of CVD SiC[3] (at 15°), aged IBD B_4C films[7] (at 15°), and Ir films[5] (at an unstated angle) to that of hot-pressed B_4C. Reflectance of hot-pressed B_4C is considerably higher than that of IBD B_4C in the spectral region between about 54 and 160 nm, and reflectance of both materials take similar values both at 49 nm and above 170 nm. Reflectance of hot-pressed B_4C is higher than that of Ir films for wavelengths ranging from 54 to 180 nm. Hot-pressed B_4C shows a higher reflectance than CVD SiC in the spectral region below about 67 nm. For instance, at 49.0 nm hot-pressed B_4C reflectance is 0.173 compared to 0.077 for CVD SiC. Both materials have a similar reflectance in the spectral region from 60 to 92 nm; at wavelengths above 92 nm CVD SiC shows a higher reflectance than hot-pressed B_4C. Hot-pressed B_4C reflects over 40% in the interval 80-121.6 nm. All the above makes hot-pressed B_4C one of the materials with the highest EUV reflectance. Hot-pressed B_4C might then be a suitable mirror material for astronomy observations in the EUV, where important spectral lines like those of HI, DI, H_2, HeI, CIII, NV, OV, OVI, NeVIII, MgX, SiXII, etc., are lying.

Fig. 2 compares the optical constants obtained for hot-pressed B_4C to those of IBD B_4C from Blumenstock *et al*. [7]. The higher reflectance of hot-pressed B_4C longward of 54 nm relies on its lower refractive index *n* (except at 121.6 nm) and its higher extinction coefficient *k*. The optical constant difference between both forms of B_4C may be due to a likely lower density of the amorphous IBD B_4C film compared to the crystalline hot-pressed B_4C.

Mirrors for EUV observations with application to earth and space sciences are usually placed on satellites orbiting in a low earth orbit, which is an extremely harsh environment due to the presence of energetic atomic oxygen. Candidate materials for mirrors have to be able to withstand this environment for a time long enough to carry out the mission. EUV reflectance of both IBD SiC films[8] and CVD SiC mirrors [9, 10] is known to strongly degrade under exposure of these materials to the low-earth-orbit environment (refs. 8 and 9), and to 5 eV oxygen atoms (ref. 10). However, IBD B_4C films were able to withstand short-term exposure to atomic oxygen in the low-earth-orbit environment [8]. This relative inertness of B_4C compared to SiC in the low-earth-orbit environment, along with its high EUV reflectance, make hot-pressed B_4C an interesting reflective material for both earth and space science applications.

6. Summary

A new material, hot-pressed B_4C, has been shown to have an important normal incidence reflectance in the EUV, which exceeds that of CVD SiC for wavelengths shorter than about 67 nm, and is similar to this material between 67 and 92 nm. Hot-pressed B_4C has also higher reflectance than IBD B_4C in the spectral region 54-160 nm. The optical constants of hot-pressed B_4C were calculated from reflectance measurements versus the angle of incidence. Better stability of IBD B_4C compared to SiC in the atomic oxygen atmosphere of a low earth orbit makes hot-pressed B_4C an interesting material to be considered for an EUV mirror for space applications.

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Figure Captions

Fig. 1. Near normal reflectance of hot-pressed B_4C compared to CVD SiC, ion-beam-deposited B_4C films, and Ir films. Straight lines connecting symbols are drawn to guide the eye.

Fig. 2. Optical constants of hot-pressed B_4C compared to those of ion beam deposited B_4C. Straight lines connecting symbols are drawn to guide the eye.

Table 1. Optical constants of hot-pressed B_4C		
Wavelength (nm)	n	k
49.0	0.50	0.41
54.3	0.45	0.63
58.0	0.45	0.74
67.2	0.53	1.02
74.0	0.60	1.15
83.5	0.77	1.45
92.0	0.86	1.61
104.8	1.11	1.81
121.6	1.77	2.05

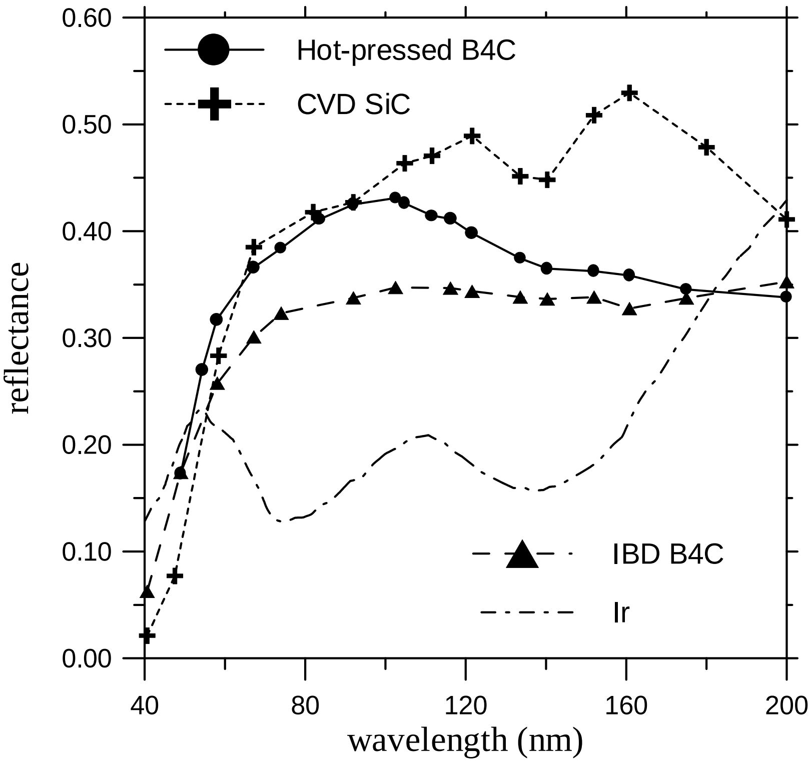

Fig. 1
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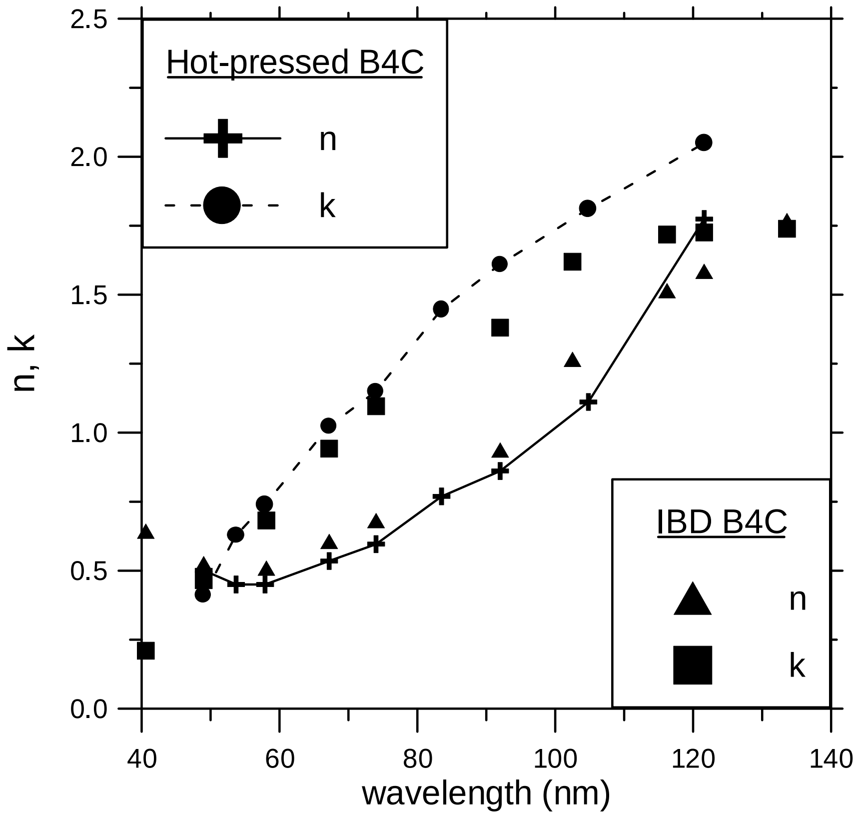

Figure 2
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