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ION BEAM FIGURING OF CVD SILICON CARBIDE MIRRORS

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ABSTRACT

Optical and structural elements made of silicon carbide are increasingly found in space instruments. Chemical vapor deposited silicon carbide (CVD-SiC) is used as a reflective coating on SiC optics in reason of its good behavior under polishing. The advantage of applying ion beam figuring (IBF) to CVD-SiC over other surface figure-improving techniques is discussed herein. The results of an IBF sequence performed at the Centre Spatial de Liège on a 100 mm CVD-SiC mirror are reported. The process allowed to reduce the mirror surface errors from 243 nm to 13 nm rms. Beside the surface figure, roughness is another critical feature to consider in order to preserve the optical quality of CVD-SiC. Thus, experiments focusing on the evolution of roughness were performed in various ion beam etching conditions. The roughness of samples etched at different depths down to 3 μm was determined with an optical profilometer. These measurements emphasize the importance of selecting the right combination of gas and beam energy to keep roughness at a low level. Kaufman-type ion sources are generally used to perform IBF but the performance of an end-Hall ion source in figuring CVD-SiC mirrors was also evaluated in this study. In order to do so, ion beam etching profiles obtained with the end-Hall source on CVD-SiC were measured and used as a basis for IBF simulations.

1. INTRODUCTION

Silicon carbide is an excellent candidate for lightweight space system due to its high specific stiffness and low thermal susceptibility. These last years, the trend was to propose space optical instruments built completely in SiC (mirrors and structures) to obtain an athermal behavior. Four basic types of SiC are commonly available: hot pressed or sintered, siliconized or reaction bonded, single crystal and chemical vapor deposited (CVD). Among them, chemical vapor deposited silicon carbide (CVD-SiC) is a superior material for optical applications thanks to the high quality polish (<0.3 nm rms) that can be achieved. CVD-SiC can be used for the whole mirror or alternatively as a reflective coating to

reach high optical performance, while sintered or reaction bonded SiC is used for the substrate and mechanical structures. Pure CVD-SiC mirrors can be produced either by conventional fabrication, near-net-shape fabrication or precision replication [1]. In conventional fabrication, a solid piece of CVD-SiC is machined, lapped and polished. However, CVD-SiC is still a hard material for these conventional means of fabrication and requires diamond machining with relatively slow shallow cuts. This is also true for the fabrication of SiC mirrors with CVD-SiC coating. In this case, CVD-SiC is coated on the SiC surface after optical machining and before optical polishing. At this level, high accuracy figuring is hardly achievable by classical polishing means, particularly for aspherical mirrors which are more and more required for space applications. This can be improved by introducing ion beam figuring as an additional step.

2. ION BEAM FIGURING OF CVD-SiC

Ion beam figuring (IBF) is an advanced technique that has been used since more than fifteen years to improve the figure of optical elements. This technique was first demonstrated by Wilson et al. [2] and afterwards started to be used on a larger scale mainly on glass materials [3]. The technique consists essentially in rastering an ion beam across the mirror surface with a variable velocity in order to remove the desired shape and thickness of material from a substrate; it makes use of the ion sputtering process. This deterministic technique allows to correct very thin optics in reason of the near-zero pressure applied by the beam, and can be used nearly on all kind of optical shape (aspherical, ...). Ion beam figuring is usually performed as a final step in optical manufacturing because the etch rate it provides is particularly low (tens of nm/min only at the beam center). Note, however, that the ion beam etch rate of CVD-SiC is not very different from those of other optical materials which is not the case for conventional machining.

The two possible disadvantages of IBF are the risks of heating the material and the potential increase of the

surface roughness. Heating has to be taken into account notably for glass where thermal gradients can lead to structural damage [4] but it is irrelevant for CVD-SiC which displays a low thermal sensitivity. Concerning the evolution of CVD-SiC roughness under ion beam milling, previously published results showed different increases of roughness related to different sample fabrication [5-6]. As a matter of fact, CVD-SiC prepared by ductile grinding presented the lowest roughness evolution ($< 0.5 \text{ nm}/\mu\text{m}$) presumably of absence of subsurface defects; it is therefore a good candidate to ion beam processing. A study on the evolution of roughness upon ion mill was recently led at CSL [7]; relevant results will be summarized hereafter.

Over the last years, some works have dwelled on the ion beam figuring of optics made of SiC as it has become a strategic material notably for space applications [8-9]. The interest in such a process has also been shared by CSL and has justified different experiments. Some of these are presented in this paper : a sequence of IBF performed on a CVD-SiC flat mirror, preliminary tests for the use of an end-Hall ion source and measurements of the evolution of roughness upon ion milling. CVD-SiC samples manufactured by Rohm and Haas (sic-001) were used in these experiments. It is a polycrystalline cubic (β -phase) SiC with randomly oriented crystallites.

3. CSL ION BEAM FIGURING FACILITY

The Centre Spatial de Liège (CSL) has developed a laboratory IBF facility which has been operational since 1996. The ion beam figuring facility was described thoroughly elsewhere [10] but its main characteristics are summarized hereafter. The cylindrical vacuum chamber is 1.4 m high and has a diameter of 1.2 m, giving an internal volume of about 1.5 m^3 . The overall facility is located in 10 000-grade clean room. Substrates up to 200 mm diameter can be processed . A hollow cathode ion source of 3 cm in diameter is fixed on a 4-axis motion system. A fifth axis is set on the substrate mount. The five axes are software controlled in such a way that the ion beam scans always the workpiece at normal incidence, constant distance and appropriate velocity. Fig. 1 shows the vacuum chamber of the IBF facility during the correction of a mirror.

In fact, this facility is the prototype of an industrial facility installed at AMOS sa, which can process mirrors up to 1 meter.



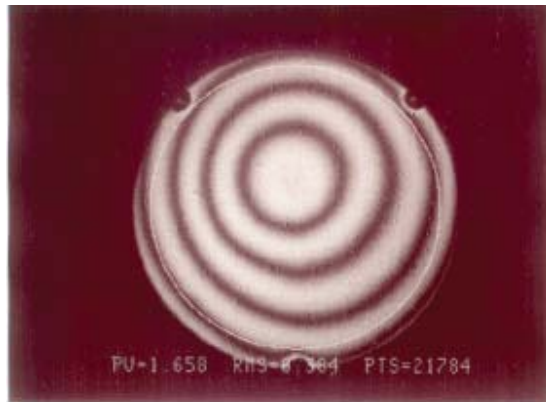
Fig. 1. The vacuum chamber of the IBF facility during the correction of a mirror. The ion beam can be seen through the window.

4. IBF OF A CVD-SiC MIRROR

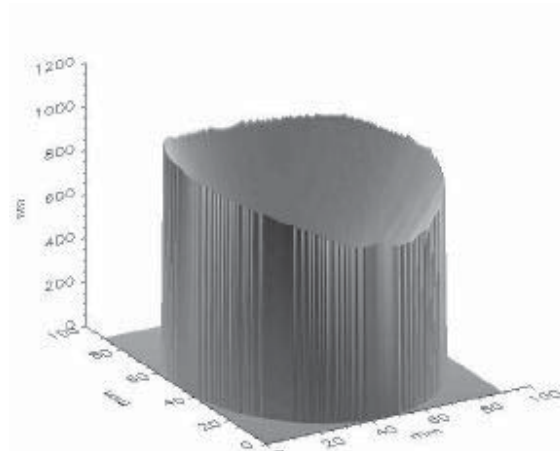
A 100 mm diameter flat mirror in CVD-SiC has been corrected by IBF in two sweeps in the CSL facility. The initial surface error of 243 nm rms has been decreased to 13 nm rms on the 88-mm diameter useful optical aperture. Surface statistical results are summarized in Table 1. The root-mean-square (RMS), peak-to-valley (PV) values and the convergence factor, defined as the ratio of the surface statistics before and after the process, are reported. The interferograms measured with a Zygo Mark IV and the 3D-plot of the mirror surface at the different steps of IBF are shown in Fig. 1 and Fig. 2 respectively. On the full reflective surface (99 mm), the error figure was 294 nm rms initially and 20 nm rms only after the 2nd IBF run.

	RMS [nm]	PV [nm]	Convergence factor RMS - PV
Initial	243	1049	
After 1 st run (6h 25')	28	205	9 - 5
After 2 nd run (8h 29')	13	86	19 - 12

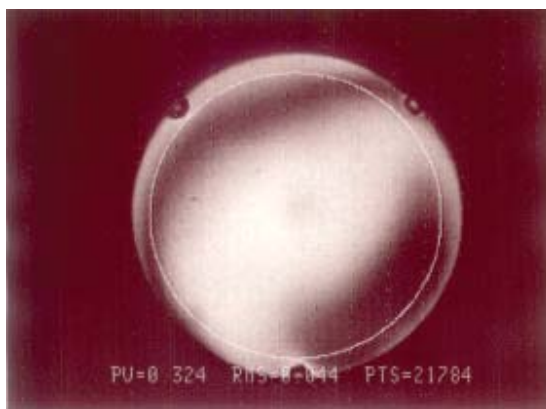
Table 1. Surface statistical figures of the CVD-SiC mirror corrected by IBF (on a 88 mm dia. aperture).



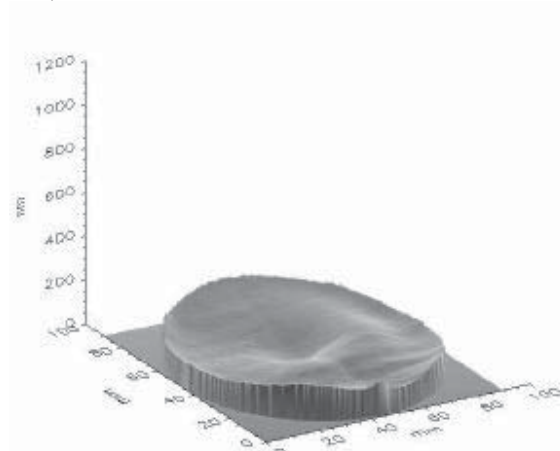
a) Initial surface errors



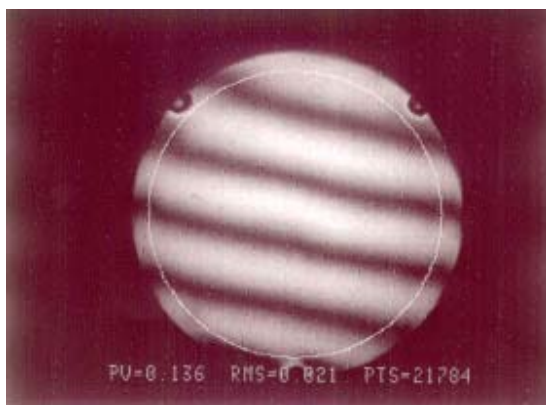
a) Initial surface errors (RMS=243 nm, PV=1049 nm).



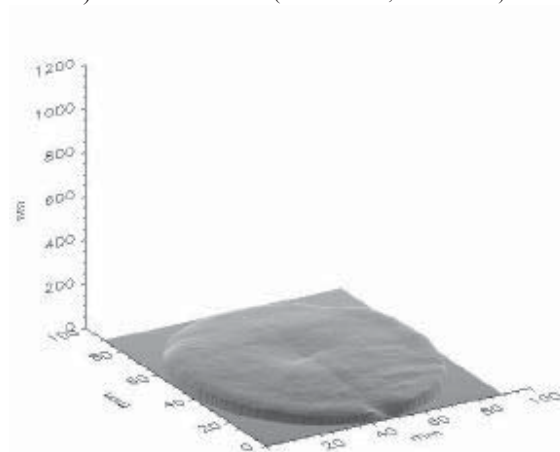
b) After 1st IBF run.



b) After 1st IBF run (RMS=28 nm, PV=205 nm).



c) After 2nd IBF run.



c) After 2nd IBF run (RMS=13 nm, PV=86 nm).

Fig 1. Interferograms ($\lambda=633$ nm) of the surface of the mirror at the different IBF steps. Statistical values are computed over the 88 mm dia. useful aperture represented by the white circle.

Fig 2. 3D plot of the surface errors on the 88 mm dia. useful aperture

For the first run the full beam exiting the 3-cm ion source has been used. For the second run a narrow beam get with the help of a beam mask has been used. The radius of this narrow beam was four times smaller than the full beam. Typical etching profiles for both beams on CVD-SiC are shown in Fig. 3. Fig. 2 shows that the low frequency errors are removed in a very efficient way. The correction of shape by IBF is obviously limited by the size and shape of the ion beam (the tool). Initial high frequency errors like the cross at the center are beyond the ion tool resolution used here. The lowest surface errors that could be reached in one run as computed by our IBF software for this mirror and the narrow beam profile used was 3 nm rms and 41 nm pv on the 88 mm aperture. Beam masks providing ion beams 2-3 times narrower than this one had already been used for other applications.

5. IBF OF CVD-SiC USING AN END-HALL ION SOURCE

End-Hall ion sources are currently used for space propulsion. We had the opportunity to test for IBF such a source specially adapted to industrial applications. More precisely, a 7-cm end-Hall SI70 ion source from Snecma motors. In Table 2 the main characteristics of the SI70 are compared to those of the normally source used at CSL for IBF: i.e. a 3-cm Kaufman hollow cathode source with a 2-grid acceleration system. End-Hall ion sources work at lower beam energy than Kaufman sources, but can provide a significantly higher beam current density. Thus, one can expect high removal rates which makes these sources particularly attractive.

For glass materials, higher current densities normally means higher risks of structural damage through heating but whereas the low thermal sensitivity of CVD-SiC makes it essentially impervious to such hazards. This hints to that the SI70 would be particularly suited to the ion beam figuring of SiC. Fig. 3 shows typical etching profiles on CVD-SiC flat mirrors obtained with the end-Hall and Kaufman sources and measured by optical profilometry. For the latter, the etching profile of the full 3-cm ion beam and of a masked beam are shown. The relatively high removal rate of the end-Hall ion source on CVD-SiC can be appreciated in Fig 3. In fact, the removal rate computed (by integration of profiles represented in Fig 3. on beam radius) was 7.5 times higher with the end-Hall ion source than with the Kaufman source, although the former was not operated at maximal power, namely 400 W instead of 1200 W.

IBF simulations were performed on the basis of the etching profile provided by the SI70 on medium to large size optics from 200 mm in diameter, whereas it is less significant for smaller size mirrors in reason of the

relatively large beam size. Obviously, masking the beam should allow to circumvent the problem. The main advantage that is emphasized by the simulations is the reduction of the process duration when using the SI70. For instance, a 400 mm dia. mirror with a figure error of ~ 200 nm rms could be corrected in 6 hours while it would take 18 h approximately with the 3-cm Kaufman ion source. Taking only the removal rate into account, a better performance would have been expected from the SI70 but one needs also to consider the size and shape of the beam with respect to the size of the defects to remove and the over all surface to correct. The SI70 is definitively a larger tool than the Kaufman source. Moreover, the shape of the SI70 etching profile is partly lorentzian, with extended edges which are not as suited to IBF computation as a pure gaussian etching profile commonly provided by Kaufman sources is. This results in a greater thickness of material removed with the SI70 which obviously increases the time process. This should be improved by masking of the beam to reduce the edges of the etching profile.

	SI70	Hollow cathode ion source with grids
Type	End-Hall	Kaufman
Gas	Xe, Kr, Ar, O ₂	Ar, Xe (...)
Gas flow	20-80 sccm	1-4 sccm
Exit beam diameter	7 cm	3 cm
Beam energy	50 - 450 eV	50-1200 eV
Max. beam current	> 2 A	100 mA
Max. current density	> 30 mA/cm ²	10 mA/cm ²

Table 2. Comparison of the main characteristics of the SI70 (end-Hall type ion source) with the Kaufman ion source commonly used for IBF at CSL.

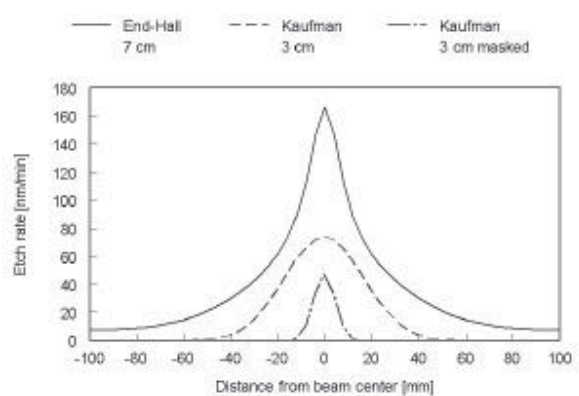


Fig 3. Comparison of typical etching profiles on CVD-SiC for a 7-cm end-Hall ion source (SI70) and a 3-cm Kaufman ion source.

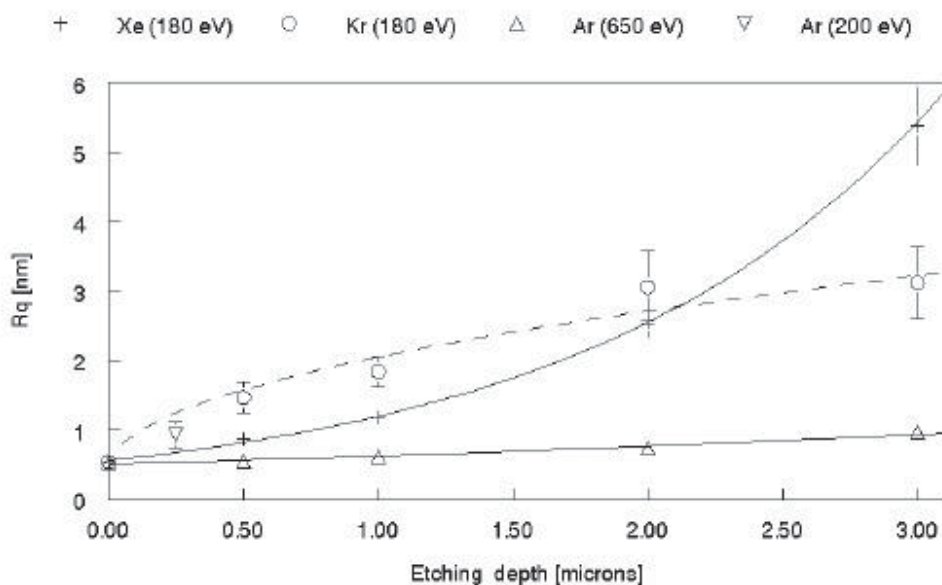


Fig. 4. Evolution of the RMS roughness, R_q , of CVD-SiC etched with Xe^+ , Kr^+ and Ar^+ (optical profilometer, x10).

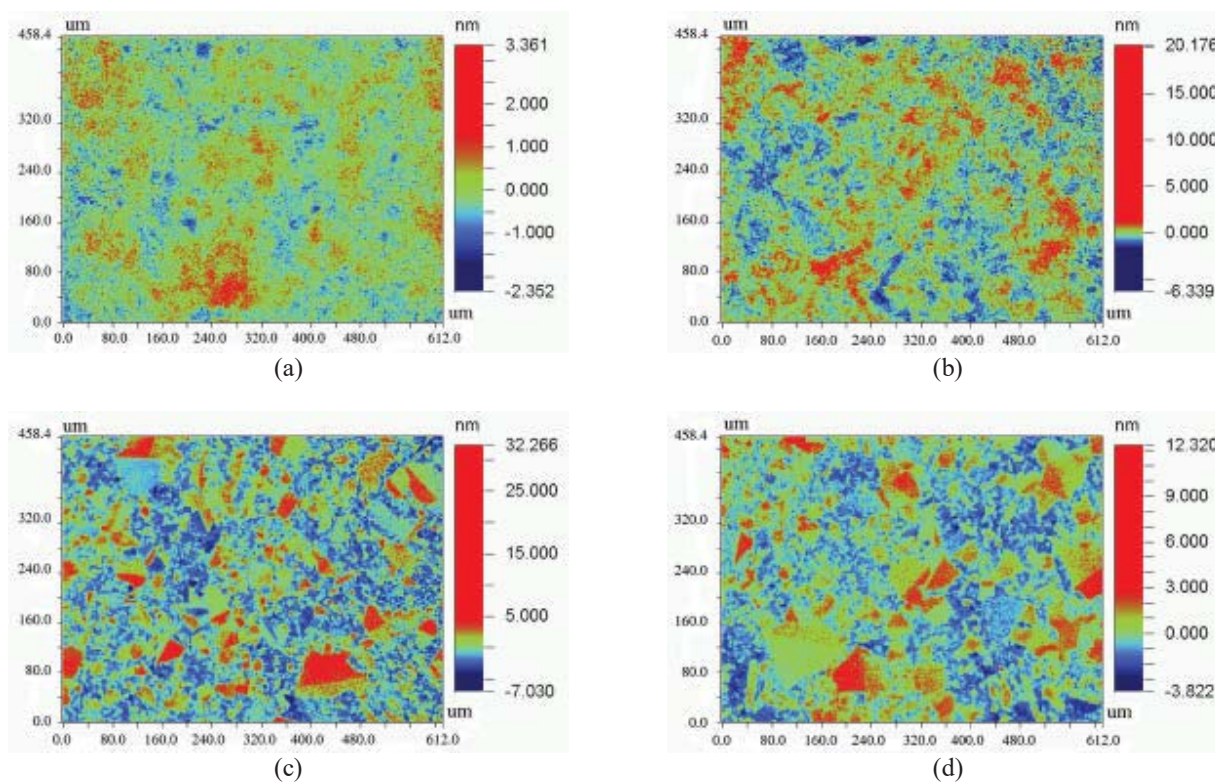


Fig 5. Micrographs of the CVD-SiC surface obtained by optical profilometry: (a) before ion etching , after 1 μm etched (b) with 650 eV Ar^+ , (c) with 180 eV Kr^+ , (d) with 180 eV Xe^+ .

Eventually, during the time we used the end-Hall ion source (>100 hours), its main advantages at the point of view of IBF were: its sturdiness, the stability of the beam, the possibility to tune the shape of the beam with the magnetic field and its high removal rate.

6. EVOLUTION OF CVD-SiC ROUGHNESS UNDER IBF

Roughness was measured as function of etching depth in CVD-SiC for the both kind of ion source presented in the last section : the 3 cm Kaufman-type ion source and the 7 cm end-Hall source (except for etching with 200 eV Ar⁺ where a 10 cm Kaufman-type ion source was used). The procedure remained the same whichever source was used for the etching experiments. Each sample was exposed to several successive ion doses with the same beam parameters and etching conditions. Following each etching session, the surface roughness was measured by using a WYKO RST Plus optical profilometer with a x10 magnification in the PSI mode (Phase-Shifting Interferometry). Ten measurements were made randomly on the sample surface near the center of the ion beam impact area to compute the average rms roughness value and the standard deviation. To each investigated set of beam parameters corresponds a new sample.

Etching experiments were performed on pure CVD silicon carbide samples having a roughness of 0.5 nm rms. First of all, an end-Hall ion source was used to etch these samples with Xe⁺ and Kr⁺ down to a depth of 3 μm. A discharge voltage of 200 V was applied, which leads to an ion beam energy distribution centered on ~180 eV. Rms roughness measurements are plotted in Fig. 4. The roughness increases for both gases but in a different way (up to 3 nm rms for Kr⁺ and 5 nm rms for Xe⁺ after 3 μm etching depth). Other silicon carbide samples were etched with the 3-cm Kaufman source with 650 eV Ar⁺ down to a depth of 3 μm. In this case the roughness evolution was low as it can be seen in Fig.4 and remained under 1 nm rms after 3 μm etched. This very moderate increase compared to the etching with Xe⁺ and Kr⁺ seems to be linked to the beam energy. Indeed, a sample etched with 200 eV Ar⁺, generated by the 10-cm Kaufman source, reveals an important increase of the roughness: 0.9 nm rms after only 0.25 μm etched and a rapid evolution of the surface topography similar to 180 eV Xe⁺ or Kr⁺ etching. Fig. 5 shows pictures get with the optical profilometer of the CVD-SiC surface before and after 1 μm etched with 650 eV Ar⁺, 180 eV Xe⁺ and 180 eV Kr⁺.

The evolution of CVD-SiC roughness under ion beam milling depends strongly at least on the beam energy and the nature of the ions used for the IBF process. Depending on these parameters, the CVD-SiC structure is more or less highlighted by the ion beam milling. The stronger increases of the CVD-SiC roughness with low energy ion beam (~ 200 eV) gives the preference to use higher energy ion beam for IBF applications requiring low roughness. It is easily reachable by Kaufman ion sources with grids, while beam energy above 200 eV should be investigated more in details to see if the maximum energy reachable by the end-Hall ion source (~ 500 eV) is enough to maintain the roughness increase to minimum.

7. CONCLUSIONS

The ion beam figuring of CVD-SiC has been demonstrated at the Centre Spatial de Liège. An example of a CVD-SiC mirror correction where the rms value was decreased by near a factor 20 was presented. An end-Hall ion source has also been tested for ion beam figuring of CVD-SiC. It represents an interesting alternative to the ion sources commonly used in IBF. Eventually, the evolution of roughness was identified as a critical aspect of the IBF process applied to CVD-SiC.

8. ACKNOWLEDGEMENTS

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