Fixed abrasive grinding of CVD SiC mirrors

Thomas Bifano,* Yuan Yi,* and W. Keith Kahl†
*Aerospace and Mechanical Engineering Department, Boston University,
Boston, MA, and †Oak Ridge National Laboratory, Oak Ridge, TN, USA

A strategy for machining ceramic (silicon carbide) mirrors is described. By taking advantage of the relative ductility of ceramics compared with that of glass, it is shown that fixed-abrasive, ultraprecision grinding is a viable fabrication process for high-performance aspheric mirrors. Also, it is shown that ductile grinding of ceramic substrates can produce optical quality surfaces, particularly in infrared applications. Laboratory-scale ductile-regime grinding of chemically vapor deposited silicon carbide is described, as is a scale-up of the technology to a commercially available ultraprecision machine tool that has been retrofitted for grinding aspheric ceramic components.

Keywords: *ceramic machining; ductile-regime grinding; optical fabrication; ultraprecision grinding*

Introduction

Chemically vapor-deposited silicon carbide (CVD SiC) is a leading candidate material for large, space-based, high-energy mirror applications. Because of its large specific stiffness, purity, and structural stability, CVD SiC is also being used in less exotic applications, such as in the production of lens molds and replicated optics. When finished to subnanometer roughness, SiC is an excellent reflector, particularly for 10 to 12.5-μm wavelengths, where its reflectance is greater than 90%.[1,2] However, it is a difficult material to polish because of its low polishing rate. For identical conditions of grit size and polishing pad pressure, the Preston coefficient (removal rate per unit time) for SiC has been found to be less than 1/20 that of optical glasses (BK-7 and fused silica).[3] These polishing experiments were conducted with diamond abrasive on a Kemet lap and with B_4C abrasive on a cast iron lap. Although identical polishing conditions can be used to contrast the polishing process for the three materials, different polishing conditions (e.g., greater pad pressure for SiC workpieces than for glass workpieces) might result in improved process economy for SiC polishing without significant reduction in the surface quality of the finished part. An alternative to polishing that could also produce optical quality surfaces is ultraprecision, fixed-abrasive grinding. This article describes such a diamond grinding process for the production of ceramic mirrors.

Despite its relatively low Preston coefficient and relatively high cost, SiC could replace glass in many advanced optical applications because of its toughness, stiffness, hardness, and large critical depth of cut (d_c) for damage-free machining. *Table 1* compares some of the relevant material properties of SiC with those of a fused silica glass. An important point of this comparison is the critical depth of cut, with values calculated using the critical depth of cut model and corroborated by experimental grinding tests, described in detail in reference 4. The critical depth of cut is a parameter linking the properties of the workpiece material with the abrasive grain cutting depth (e.g., machining chip thickness) that is required to avoid subsurface fracture in machining. Material removal processes in which the dominant mechanism of irreversible deformation is plastic flow (as opposed to fracture) can be achieved on many brittle materials in a technology known as ductile-regime machining[4–12] (also known as shear-mode, viscoplastic, or damage-free machining). Surfaces that are machined in a ductile regime have less subsurface damage and better surface finish than those machined in a brittle regime, with comparable contour accuracy. For this reason, ductile-regime machining is a promising technology for high-precision grinding of brittle glasses and ceramics for optics, mechanical bearings, semiconductors, and other components requiring advanced material properties in combination with tolerances measured in nanometers. There are certain models, now well established, describing the relationship between machine tool operational parameters,

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Address reprint requests to Thomas Bifano, Aerospace and Mechanical Engineering Department, Boston University, 110 Cummington Street, Boston, MA 02215, USA.

Table 1 Optical material comparison: SiC versus glass

Property	CVD SiC ceramic	Fused silica glass
Coefficient of thermal expansion	2.5 μm/K	0.03 μm/K
Fracture toughness	3.3 MPa $m^{0.5}$	0.74 MPa $m^{0.5}$
Specific stiffness	150 MNm/kg	30 MNm/kg
Hardness	25 GPa	5.1 GPa
Critical depth of cut (d_c)	180 nm	6 nm

workpiece material properties, and the critical depth of cut for ductile-regime machining. The most basic requirement for maintaining a ductile material removal regime is to ensure that the depth of cut made by an individual abrasive cutting grain does not exceed some threshold value (i.e., the critical depth of cut). If the actual grain depth of cut exceeds the critical depth of cut, fracture occurs beneath the abrasive grain, and the material-removal mechanism changes from ductile plowing to brittle crack propagation. Because typical ultraprecision machine tools permit minimum actuation steps of approximately 5 nm (laboratory scale) to 10 nm (commercially available), the distinction between SiC (d_c = 180 nm) and fused silica glass (d_c = 6 nm) is that it is much easier to grind SiC in an entirely ductile regime, both in the laboratory and in production.

Silicon carbide is generally fabricated in one of three processes: reaction bonding (RB), hot pressing (HP), or chemical vapor deposition (CVD). The former two processes lead to a two-phase material (silicon carbide plus silicon for RB, silicon carbide plus a binder for HP) with the silicon carbide solidifying in its α phase (i.e., hexagonal crystal structure). The CVD process produces a single phase material: silicon carbide crystals in the β phase (cubic crystal structure). The α silicon carbides are considerably less expensive to fabricate than are CVD SiC, but there is a penalty to pay for the introduction of a second phase when considering ultraprecision machining processes: the two phases are likely to machine at different rates, and perhaps with different material removal mechanisms (i.e., brittle fracture versus plastic flow). In recently reported ultraprecision machining tests on reaction-bonded SiC, it was demonstrated that subnanometer roughness is more difficult to achieve on this two-phase material.[13,14] For this reason, we will limit our discussion of ultraprecision grinding to results obtained on the more ideal CVD SiC material, although other forms of SiC may be more economical for production.

This article describes high-precision diamond grinding of as-deposited polycrystalline CVD SiC to optical quality. Because CVD SiC is a single-phase material with a relatively large critical depth of cut, it ought to be machinable in the ductile regime. It will be demonstrated that CVD SiC can be ground to surface finishes of 5 to 10 Å RMS, with no subsurface damage. Also the scale-up of this concept to a commercially available ultraprecision machine tool retrofitted for ductile grinding will be described. A problem encountered in fixed-abrasive grinding of large CVD SiC components is the eventual dulling of the grinding wheel diamonds. This phenomenon and some possible solutions, will be described for SiC microgrinding.

Laboratory-scale fixed abrasive microgrinding of CVD SiC

Experiments were performed on an air-bearing-based ultraprecision grinding apparatus in a Blanchard configuration. The apparatus has been described elsewhere.[14,15] The infeed axis was controlled in real time with a bandwidth of 300 Hz and a resolution of 5 nm, using capacitance gauge feedback and piezoelectric actuation in a microcomputer based control algorithm. The coolant was distilled water. The CVD SiC test samples were ground under the conditions described in *Table 2*. Machining conditions and wheel selection were based on prior experience and do not reflect an optimization process, although they prove sufficient to achieve a ductile grinding regime for this material. On the issue of wheel selection, there are two bond types that are generally available for ultraprecision diamond grinding applications: resin or bronze. Resin is more compliant, leading to a more even distribution of cutting forces over the diamonds in the wheel and consequently lower grinding forces on individual grains. Because of this compliance, using a resin bond wheel makes it easier to maintain subcritical grinding forces (i.e., easier to maintain a ductile grinding regime), but at a cost of reduced contour accuracy.[5] All of the experiments reported here were conducted with resin bond grinding wheels.

The ground CVD SiC surfaces were examined using a scanning electron microscope (SEM). SEM images of the ground CVD SiC show that the prevailing mechanism of material removal was plastic flow (*Figure 1*). In this photomicrograph, the only dis-

Table 2 Grinding conditions for microgrinding CVD SiC

Wheel geometry	100 mm diameter, 6 mm wide cup wheel (M4D, Norton)
Wheel type	4- to 8-μm natural diamond, resin bond, concentration 75
Wheel preparation	Pyrex-based lapping with 5-μm diamond paste
Workpiece	$5 \times 5 \times 25$ mm
Crossfeed axis	Air bearing, piezoelectric inchworm actuator (5 nm resolution)
Infeed axis	Flexure, piezoelectric stack actuator
Infeed axis control	5 nm resolution, closed-loop (PID), 300-Hz bandwidth (3 dB)
Machining conditions	Fixed infeed step, constant velocity crossfeed
Infeed and crossfeed	1 μm, 36 mm/min (20 passes) 0.2 μm, 18 mm/min (50 passes) 0.2 μm, 9 mm/min (50 passes)
Wheel speed	5,400 RPM
Grinding coolant	Distilled water
Measured stiffness	50 MN/m (normal to grinding contact, 1 N Static load)

cernible features are cutting marks spaced about 1.5 μm apart, corresponding to the cross-feed of the workpiece per revolution of the grinding wheel. In the brittle-regime surface grinding operation used to prepare these samples, grinding damage extended less than 5 μm deep, as measured by optical microscopy of visible fracture pits. It has been reported that ductile-regime grinding of glass does not significantly propagate existing fracture,[16] and it would appear from these surfaces that that is also the case in ductile-regime grinding of SiC. The existence of residual subsurface damage is more difficult to detect in SiC. The common etching technique, involving exposure of the SiC to molten KOH at a temperature of about 725°K, preferentially etches both subsurface cracks and grain boundaries, making it difficult to distinguish between the two. As an alternative technique, it is possible to infer the presence or absence of subsurface damage by ion milling the finished surface.[17,18] It was found that ion milling to a depth of 3 μm caused a significant (500%) increase in the RMS roughness of a polished sample, whereas no significant increase was observed in the roughness of ductile-ground samples that were ion milled to the same depth. Although both the polished sample and the ductile ground sample were finished to a roughness of approximately 10 Å RMS before ion milling and both appeared damage-free in SEM images, the ductile-ground sample had been machined to a depth exceeding the preexisting damage depth (5 μm), whereas the polished sample had been machined only enough to achieve subnanometer RMS roughness, presumably leaving some subsurface damage beneath the specular surface. It is important to note that the polished sample could have been machined to a depth in excess of the damage depth, in which case we would expect no remaining subsurface damage. In this case, however, it was not.

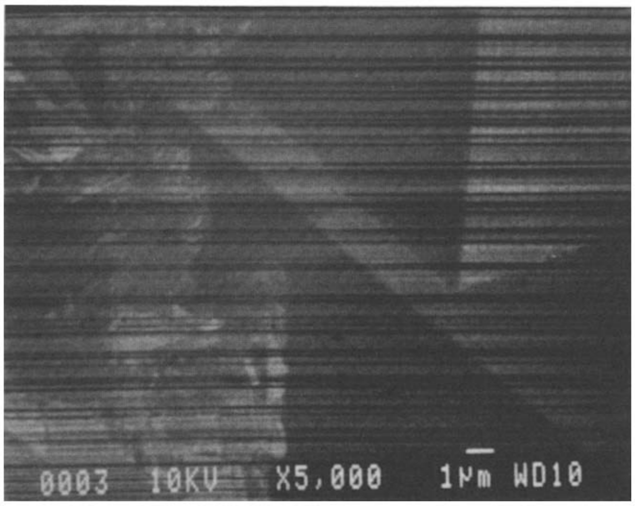

Figure 1 SEM photograph of the CVD SiC ground on the laboratory-scale ultra-precision grinding apparatus

The preliminary results of these ion milling tests, which were performed independently at both Oak Ridge National Laboratory and NASA-Marshall Space Flight Center, have been reported previously.[17,18] The conclusion drawn by both studies was that ion milling acts to expose and enlarge subsurface damage in ceramics and glasses, roughening the surface. This increased roughness is visible qualitatively in SEM photographs and quantitatively in contact profilometry (roughness) results. We can conclude that these results, in which the ductile ground samples did not significantly roughen after several micrometers of ion milling, indicate that the ductile-ground samples are largely free of subsurface damage.

To measure the surface roughness, the ground CVD SiC surfaces were examined using a Talystep profilometer. The results of a random series of 16

Table 3 Talystep profile data for CVD SiC ground

Measuring conditions for Talystep	
Instrument scan speed	Talystep
Filter	2.57 µm/sec
Stylus	Waviness, 25 Hz
	0.1 × 2.5 µm (red)
Talystep results (RMS profiles)	
50 µm scan	5.5 Å
150 µm scan	13 Å
Flatness (25 mm scan)	67 nm Ra, 430 nm P-V

profile measurements are summarized in *Table 3*, and a representative Talystep trace is shown in *Figure 2*. Over a 50-µm scan, the average roughness was 5.5 Å RMS. Notably, the average RMS roughness values measured perpendicular and parallel to the grinding direction were the same.

Wear of the diamond grinding wheel in microgrinding of CVD SiC

For the grinding geometry used in these experiments (wheel speed much greater than cross-feed speed), the specific grinding energy, *U*, can be approximated as follows:[10]

$$U = \frac{F_T V_W}{MRR} \quad (1)$$

where U is the specific grinding energy in J/m^3, F_T is the tangential grinding force in Newtons (N), V_w is the grinding wheel peripheral speed in m/sec, and *MRR* is the material removal rate in m^3/sec. Specific grinding energy was determined for a set of grinding experiments using measured values of F_T, V_w, and *MRR* as described in reference 10. U was essentially steady for the first 3 mm^3 of material removal and increased dramatically after 3 mm^3 of material removal. These results are plotted in *Figure 3*. Eventually, increased grinding energy will result in a more damaged surface as forces between the grinding wheel and the workpiece increase.[10]

There are at least three plausible causes for the observed increase in grinding energy: dulling of the abrasive grains, "loading" of the wheel with grinding debris, and sloughing of active diamonds from the wheel surface. All three of these phenomena have been observed in ultraprecision grinding wheels.[5] Under some circumstances, the grinding wheel will perform adequately in a single pass across the workpiece and can be "dressed" by conventional means (e.g., by contacting the wheel surface with a porous ceramic stick) between passes. In ultraprecision grinding, this intermittent dressing can be used along with a tool set station to achieve satisfactory results in terms of figure accuracy and surface roughness. If the grinding wheel wear is excessive in a single pass across the workpiece,

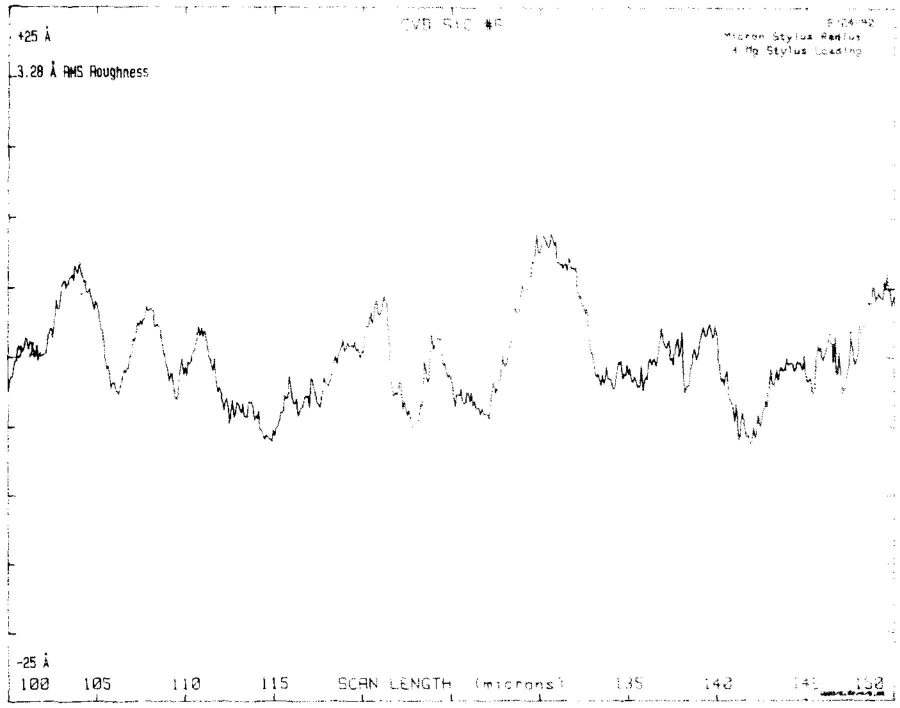

Figure 2 Talystep profile of the as-ground CVD SiC surface

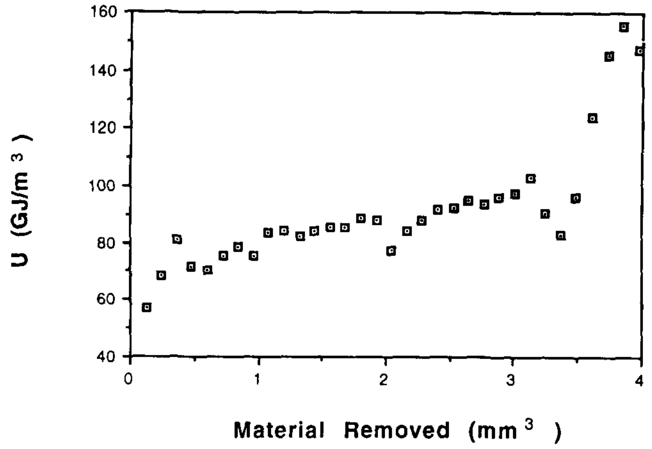

Figure 3 Specific grinding energy for microgrinding of CVD SiC

however, then in-process dressing may provide an alternative to intermittent dressing. Currently under study in the United States and abroad are various techniques for continuous redressing of metal bond diamond grinding wheels using electrolytic or electrochemical processes to continually expose new diamonds at a controlled rate during ultraprecision grinding.[19–21] For resin-bonded diamond grinding wheels, continuous in-process dressing using bound glass fiber dressing media is also being explored.[22]

CVD SiC grinding on a commercially available ultraprecision machine tool

Experiments were also performed on a commercially available ultraprecision grinding machine in a series of experiments conducted at the Oak Ridge National Laboratory Productivity Validation Test Bed (ORNL-PVTB). The high stiffness grinding platform used in these experiments was a retrofitted RTH/Rank-Pneumo Nanoform 600. The grinding head used on this machine was a motorized 100 mm diameter air bearing spindle fitted with resin bond wheels with 4–8 or 30-μm size diamond grit. A CVD SiC flat mirror and two concave spherical mirrors (f/400 and f/14) were ground using this machine. The machine is capable of producing axisymmetric aspheres as easily as it can produce spheres, but spherical contours were chosen to aid in post-process metrology. The flat was used as a baseline for comparison with the previously described laboratory experiments. The different f numbers for the spheres were chosen to fulfill actual needs for finished components by research collaborators with ORNL. All workpieces were 75 mm in diameter. The grinding configuration consisted of a vertical grinding wheel axis, peripheral grinding wheels, and a horizontal-axis rotating workpiece. The workhead spindle was traversed for crossfeed, with the periphery of the grinding wheel contacting the workpiece first at its outer edge and then traversing toward the workpiece center. The grinding spindle was mounted on a linear horizontal axis for infeed motion. Grinding parameters are shown in *Table 4*. Distinct from the previously discussed work on a laboratory-scale grinder, the Nanoform 600 manufacturing tests were conducted with a greater depth of cut and a slower crossfeed rate. The flat mirror cutting conditions result in a (geometrically) calculated grain depth of cut of approximately 100 nm. (In this grinding configuration, the grain depth of cut can be approximated by geometrically reconstructing the cross-sectional profile cut by an individual high spot on the grinding wheel.[23]) The f/400 mirror was first rough ground and then received a finishing cut, also with a calculated grain depth of cut of approximately 100 nm. The f/14 mirror was rough ground and then received a series of finishing cuts with a calculated grain depth of cut of approximately 500 nm, somewhat in excess of the 180-nm critical depth of cut for the ductile-brittle transition on this material. As the results will show, the difference between these finishing operations has significant impact on the quality of the ground surface.

The CVD specimens ground at the PVTB were evaluated for surface roughness, figure accuracy, and light-scattering behavior (*Table 5*). The surface finish of the PVTB ground samples was measured with a laser optical profiler and a Talystep profilometer. Roughness and figure accuracy were measured on a BRO interferometer and with mechanical probing. A scatterometer was used to measure the bidirectional reflectance distribution function (BRDF).

The roughnesses of the 75-mm flat and the f/400 sphere were approximately an order of magnitude larger than that obtained on the laboratory-scale grinding apparatus. For the f/14 sphere, which was ground with a grain depth of cut that exceeded the critical depth of cut, the roughness increased by another factor of five. Nevertheless, the figure error measured for the f/14 sphere was better than one wave over 95% of clear aperture, and the surface appeared specular. All of these ground specimens still show some surface damage under scanning microscopy (*Figure 4*).

It is appropriate to compare results from this grinding process with some polished CVD results (*Table 6*) reported in the literature.[1,2]

Of particular note is the similar performance of the ground material as a reflector in the long wavelength infrared (10.6 μm) when compared with polishing. Conversely, the roughness must be reduced to about 1 Å RMS to achieve low scatter in the visible (0.633 μm) wavelengths. For applications involving infrared wavelengths, there appears to be only marginal improvement with polishing over the as-ground CVD SiC for off-peak values of BRDF. No data are available for time spent performing the super-polishing operation shown in this table.

Table 4 Grinding conditions on PVTB machine

	Flat mirror	f/400 SiC mirror	f/14 SiC mirror
Grinding geometry	Contour grinding, peripheral wheel. Fixed infeed step, constant velocity crossfeed	Contour grinding, peripheral wheel. Fixed infeed step, constant velocity crossfeed	Contour grinding, peripheral wheel. Fixed infeed step, constant velocity crossfeed
Roughing wheel geometry	None	150-mm diameter 1.6-mm edge radius	150-mm diameter 1.6-mm edge radius
Roughing wheel		Resin bond, 30 μm	Resin bond, 30 μm
Finishing wheel geometry	150-mm diameter 1.6-mm edge radius	150-mm diameter 1.6-mm edge radius	150-mm diameter 1.6-mm edge radius
Finishing wheel	Resin bond, 4–8 μm	Resin bond, 4–8 μm	Resin bond, 4–8 μm
Part speed	50 RPM	50 RPM	50 RPM
Wheel speed	6,000 RPM	4,000 RPM (roughing) 5,000 RPM (finishing)	3,500 RPM (roughing) 5,000 RPM (finishing)
Roughing infeed and crossfeed	None	2 μm, 25 mm/min (19 passes)	2 μm, 25 mm/min (70 passes*) 0.5 μm, 5 mm/min (three passes) 0.5 μm, 2.5 mm/min (one pass)
Finishing infeed and crossfeed	1.25 μm 0.75 mm/min	2 μm, 20 mm/min (two passes) 1 μm, 5 mm/min (three passes) 0.25 μm, 0.5 mm/min (one pass)	0.5 μm, 2.5 mm/min (three passes)
Coolant	Mineral spirits	Water and rust inhibitor	Water and rust inhibitor

* Seventy roughing passes were necessary for the f/14 mirror to achieve the required nominal shape. Because the f/400 sphere was shallower, fewer roughing passes were required.

Table 5 Average grinding performance of CVD SiC on PVTB machines

	Nanoform 600 grinding results		
	75 mm flat	f/400 sphere	f/14 sphere
Talystep roughness (RMS)	55 Å	72 Å	317 Å
Figure accuracy	0.82 μm P-V	Not measured	0.96 μm P-V

Table 6 Comparison of BRDF: ground CVD SiC with polished CVD SiC

BRDF @ 10° (sr^{-1})	Nanoform 600 75 mm flat	Nanoform 600 f/400 sphere	Nanoform 600 f/14 sphere	Superpolished (1 Å RMS)
0.633 μm	Not measured	Not measured	3.9×10^{-1}	3.0×10^{-5}
10.6 μm	3.7×10^{-4}	4.5×10^{-4}	9.2×10^{-4}	2.5×10^{-4}

Figure 4 SEM of CVD SiC ground on the Nanoform 600, showing some residual grinding damage in a largely ductile-ground surface

Conclusions

From these preliminary grinding experiments, it has been determined that for CVD SiC, mirror finishes can be achieved by ductile regime grinding. Moreover, SiC has an advantage over glass in the production of large optics because of its greater ductility and because the surface is reflective in the infrared wavelengths (e.g., 98% reflectance at 12 μm wavelength[1]) without postmachining coating or polishing. In terms of optical quality, the precision-ground CVD SiC exhibits comparable performance at 10.6 μm wavelength to reported data on polished CVD SiC.[1,2] The relatively large critical depth of cut of SiC, coupled with its relatively small Preston coefficient, may make ductile-regime grinding an economically viable process for producing ceramic optics when compared with polishing processes. Because of its larger stiffness, smaller workspace, and reduced number of degrees of freedom, the laboratory benchtop grinding apparatus uniformly provides superior results in grinding of silicon carbide. Nevertheless, the scale-up of this process to a Rank-Pneumo Nanoform 600 platform has provided the basis for production-level machining of ceramic mirror components.

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