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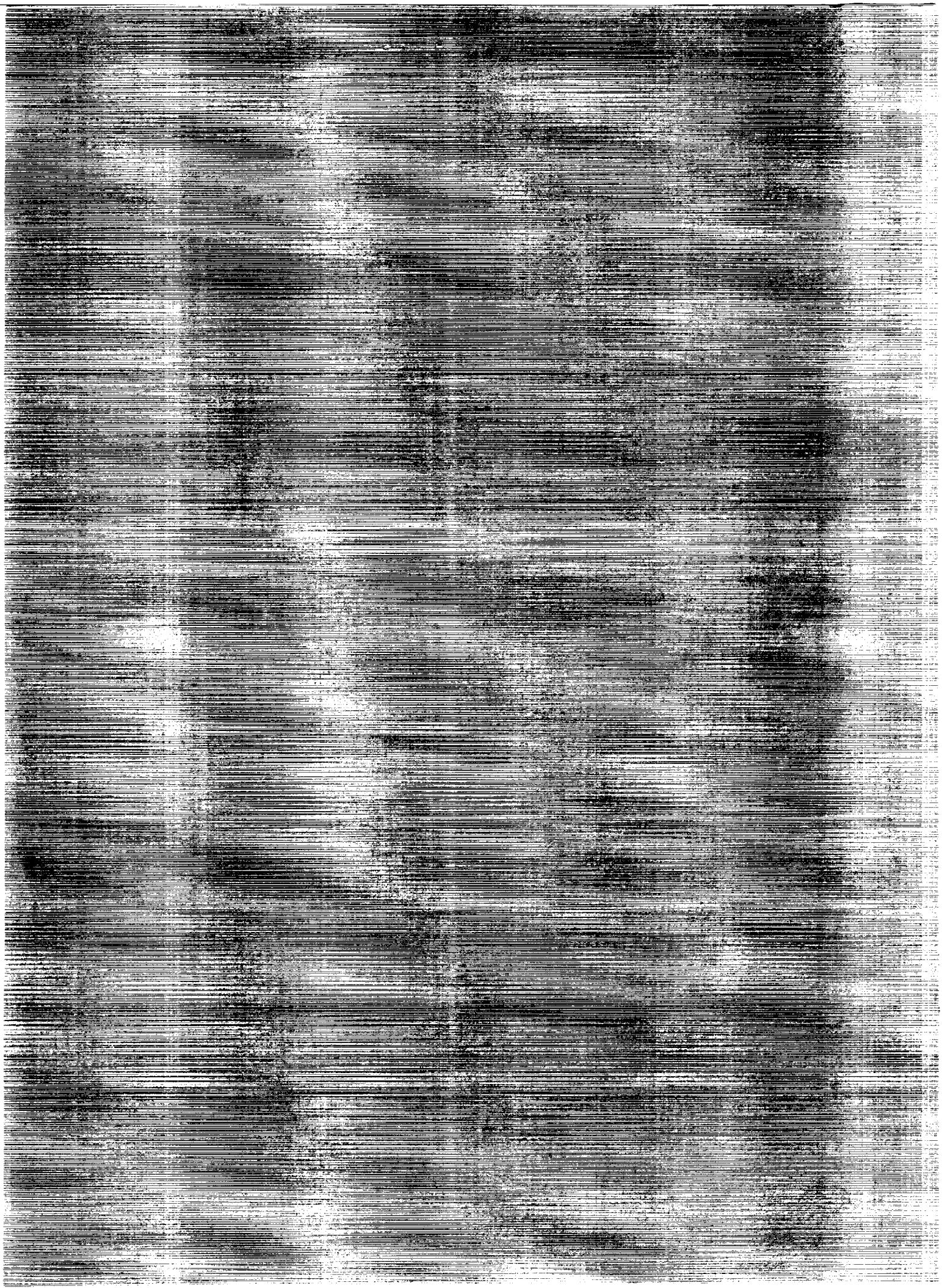

NASA Contractor Report 4389

Fabrication of Lightweight Si/SiC Lidar Mirrors

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PREFACE

This report has been prepared by the Research Center of Morton International Inc./CVD Incorporated, Woburn, MA, under Contract No. NAS1-18476 entitled, "Fabrication of Lightweight Si/SiC LIDAR Mirrors." This SBIR (Small Business Innovative Research) program was administered by NASA Langley Research Center, Hampton, VA. Mr. Dwayne E. Hinton was the Project Manager. Four other scientists involved in reviewing the research in this program were: Leon V. Taylor, James W. Cheely, Charles V. Woener and Antony Jalink, all of NASA.

At CVD Incorporated, the Program Manager responsible for carrying out this program was Dr. Jitendra Singh Goela. Dr. Raymond L. Taylor, Vice President, Research Center, provided supervision. Alex Teverovsky and Lee Burns provided assistance on various aspects of process design and engineering. Hemant Desai provided assistance on the flow pulsing technique. The small scale depositions were supervised by Roy D. Jaworski and his technicians, while large scale depositions were supervised by William Haigis, Thomas Lucia and their technicians. Carol McSweeney typed the text of this manuscript and Rose Douglas typed the tables and figure captions.

The Final Technical Report covers the period of performance from June 9, 1987 to December 8, 1990. The CVD Incorporated internal number for this report is TR-9081.

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1.0 INTRODUCTION

The objective of this SBIR NASA LIDAR (Light Detection and Ranging) Mirror Program was to develop the technology for fabricating lightweight Si/SiC LIDAR mirrors by a scalable chemical vapor deposition (CVD) process for use in NASA space-borne systems. The Si/SiC mirrors could consist of a faceplate made of either Si, SiC or SiC cladded with Si and a lightweight backstructure made of either Si or SiC. This program was organized in two phases. Phase I was a six months program which was successfully completed in January 1987. The objective of Phase I was to establish the feasibility of fabricating large, finished or near-finished Si and/or Si/SiC LIDAR mirrors via a scalable CVD process. Phase II was for a two-year duration and its objective was to further develop the CVD lightweight mirror fabrication technology and demonstrate the fabrication of a 40-cm-dia Si/SiC lightweight LIDAR mirror. This final report describes the R&D work performed during Phase II of this NASA program. The Phase I Final Technical Report was previously published.[1]

The CVD approach for fabricating lightweight Si/SiC mirrors in Phase II was to first develop the CVD Si/SiC mirror technology on a small scale ($\leq$ 7.5-cm-dia samples) in a research furnace, scale this technology to a small production furnace ($\leq$ 0.5-m-dia) and demonstrate the technology by fabricating a 40-cm-dia Si/SiC lightweight mirror. This overall approach required funds considerably more than were available in this SBIR Phase II program. Therefore a part of the technology development effort was shared with another ongoing program. This latter program was supported by the Air Force and its objective was to develop large, lightweight SiC mirrors for space applications. The emphasis in this latter program was on precision replication of SiC faceplates on prefigured, highly polished SiC mandrels.

There were three main tasks in this NASA Phase II program. In Task 1, small scale experiments were to be performed to resolve important issues associated with the fabrication of Si/SiC mirrors via CVD, an optimum approach to fabricate lightweight Si/SiC

mirrors was selected and then demonstrated by fabricating small models of Si/SiC mirrors in a research reactor. In Task 2, the scaling of the Si CVD process was to be demonstrated. Demonstration of the scaling of the SiC process and the lightweight structure (LWS) were to be accomplished as part of the AF-supported Program at CVD. In Task 3, using the approach developed in Task 1, a 40-cm-diameter mirror was to be designed and fabricated to demonstrate the scaling of the CVD mirror fabrication technology. This mirror was specified to be spherically concave with a radius of curvature of about 1.0-m. The mirror was to be delivered in the "as deposited" condition and no grinding or polishing of the faceplate was to be performed. Further, no metrology data was to be obtained on this mirror.

This NASA SBIR Phase II program has been successfully completed. A majority of the objectives of this program have been met. A total of twenty-seven (27) small scale experiments were performed to deposit either SiC or Si. These experiments have demonstrated the following: (i) SiC replication on curved and flat graphite samples, (ii) CVD bonding of SiC to itself, (iii) fabrication of SiC LWS via CVD, and (iv) Si cladding on SiC. A major accomplishment of this program was the successful fabrication of several 7.5-cm (3-inch) diameter models of flat and curved Si/SiC mirrors. A detailed fabrication procedure for these mirrors via CVD technology was developed. This procedure was used to fabricate a 25-cm-diameter SiC mirror in a production furnace. The diameter of the mirror was reduced from 40-cm to 25-cm because (i) it was more compatible with toolings and fixtures in our 0.5-m-dia furnace and (ii) all the important features of the CVD mirror fabrication technology could be demonstrated by fabricating a 25-cm-dia mirror.

The design of a 25-cm-diameter Si/SiC mirror was completed. This design was obtained by appropriately scaling a 50-cm-diameter SiC mirror design that was completed in the AF Program. The faceplate of this mirror was fabricated in a 0.5-m-dia production furnace by depositing SiC on a convex shaped graphite mandrel having a radius of curvature of 1.0-m. A graphite lightweight core consisting of hexagonal cells with triangular inner cells was designed using 0.5 mm thick graphite ribs. This core was fabricated, assembled and successfully bonded to the backside of the SiC faceplate. In this core, six equally spaced mounts were also provided for holding the mirror while

polishing/lapping its surface or performing metrology measurement. The lightweight backstructure was fabricated by depositing SiC on this graphite core in the CVD reactor.

The scaling of the CVD-Si process (Task 2) could not be demonstrated in this program. This is because considerable resources were spent in scaling the CVD-SiC process and the lightweight structure from the research furnace to the production furnace. Since the CVD processes to fabricate Si and SiC are very similar, most features of the CVD-SiC technology developed in this program are also applicable to the CVD-Si process. This coupled with the fact that CVD-Si technology was demonstrated at the 7.5-cm-dia scale, indicates that the scaling of the CVD-Si process is feasible.

Three Si/SiC mirrors were delivered to NASA: (i) A 7.5-cm-dia, lightweight Si/SiC mirror polished to a flat $\lambda/5$ ($\lambda = 0.6328$ μm) figure and finish ≈ 10 Å RMS. (ii) A 7.5-cm-dia, lightweight Si/SiC curved mirror with a radius of curvature of 1.0-m. The mirror was polished to $\lambda/5$ figure and finish of ≈ 10 Å RMS. (iii) A 25-cm-dia, lightweight SiC curved mirror with a radius of curvature of 1.0-m. All the above mirrors had a SiC faceplate and a lightweight SiC backstructure. The faceplates of the first two mirrors were also cladded with CVD-Si. In addition, seven quarterly reports were submitted and several presentations on the research work were made to NASA.

This NASA program has also met another important objective of the SBIR programs, that of commercializing the technology. Morton International/CVD Incorporated (MI/CVD) is currently marketing SiC lightweight mirrors (≤ 1.5-m-dia) for a variety of applications such as space telescopes and laser mirrors. Further, MI/CVD also supplies CVD-SiC and Si materials to optical companies for fabricating optical components such as mirrors and to ceramic material users for fabricating structural components.

This NASA SBIR program has resulted in the publication of four technical articles, one each in the Journal of American Ceramic Society and Applied Physics Letters, and two articles in the Proceedings of the Society of Optical Instruments Engineers Conferences. Four patent applications have also been filed, and one application has already been approved. The details about these publications and patents are provided in Appendix II.

In Section 2.0 which follows, important background information about LIDAR

mirrors, advantages of using Si and SiC as mirror materials, the CVD approach for the fabrication of Si/SiC mirrors, the process for CVD-Si and SiC and conclusions of Phase I research are provided. Section 3.0 includes the details of the small scale experiments. The results of scaling the SiC CVD technology and the fabrication of 40-cm and 25-cm-dia mirrors are discussed in Section 4.0. The summary and conclusions are provided in Section 5.0 and references are included in Section 6.0. Finally, Appendix I includes information about potential applications and commercialization of technology. Appendix II includes technical publications and patents that resulted from this effort.

2.0 BACKGROUND INFORMATION

2.1 LIDAR Mirrors

LIDAR (Light Detection and Ranging) has come to be recognized as an important diagnostic tool for remote measurement of a diversity of atmospheric parameters such as minor species concentrations, pressure, temperature and water vapor profiles, aerosol and cloud distributions and wind fields.[2-6] LIDAR techniques, such as measurement of back scattering, differential absorption and Doppler shifts, have applications to meteorology, atmospheric chemistry, oceanography and communications. The National Academy of Sciences Select Committee on the National Weather Service recently acknowledged a lack of current instrumentation capable of yielding a complete set of weather data and concluded "coherent LIDAR is the only concept in sight that promises to provide an operational, truly global wind determination system in the future."[7]

The earth's atmosphere can be probed with either terrestrial, airborne or spaceborne LIDAR systems. A spaceborne LIDAR is particularly attractive due to its ability to probe the atmosphere with wavelengths that cannot propagate through the lower atmosphere, its unique high spatial resolution and its capability for global or synoptic coverage. Consequently, in recent years the emphasis is shifting from the airborne to spaceborne LIDARs. In 1976, NASA convened an international panel which concluded: "LIDAR has promising applications to aeronomy, tropospheric and stratospheric research, with the tropospheric potential being particularly important due to the deficiency of other spaceborne techniques." This powerful diagnostic potential makes LIDAR systems a key element of the Shuttle/Spacelab research program for the 1990's. An important goal of this ongoing research program is to advance the understanding of the processes governing the earth's atmosphere and evaluate its susceptibility to man-made and natural perturbations.

A LIDAR system can be divided into three main components linked by well defined interfaces. These components are the laser, the receiving telescope and the detector. The most critical optical element in the receiving telescope is the primary mirror because of its size, weight, fabrication cost and thermal exposure to the outside world. The LIDAR

equation shows that the received signal is directly proportional to the area of the primary mirror. Therefore, to obtain reasonable signal levels for accurate measurement, it is important to use as large a primary mirror as feasible. This is particularly true when a spaceborne LIDAR is used to measure wind profiles in the troposphere on a global basis.

The performance of a LIDAR system also depends upon the optical configuration of its receiving telescope. Often, due to space limitations, such as in shuttle-borne LIDARs, the length of the telescope is fixed. Therefore, the optical designer must select a particular shape and speed of the mirrors to maximize the throughput of the telescope. In a Dall-Kirkham system consisting of an ellipsoidal primary, a spherical secondary and a parabolic collimator, a trade-off occurs between the linear obscuration and the blur circle diameter which depends upon the speed of the primary mirror. A fast primary mirror produces a small obscuration but a large blur circle due to increased coma and enhanced sensitivity to defocusing errors. The Dall-Kirkham system is also sensitive to the space between the primary and secondary mirrors. The Cassegrain system is similar to the Dall-Kirkham except that the shape of the primary and secondary mirrors is paraboloidal and hyperboloidal, respectively. The use of these shapes yields less coma for off-axis rays, although for on-axis rays, this system is virtually identical to the Dall-Kirkham. Both these configurations have been widely used by NASA scientists in their LIDAR systems. A Ritchey-Chretien configuration provides a better performance than the above two configurations because aspheric primary and secondary mirrors are used. The aspheric surfaces correct for coma and thus provide a diffraction limited performance. Less attention has been given to the Ritchey-Chretien system in the past due to increased cost and risk involved in fabricating aspheric surfaces using the current technology.

While the requirements for LIDAR telescope mirrors, in particular the primary mirror, depends upon the telescope optical configuration, the particular application and other systems constraints, certain generic requirements can be specified. Greco[3] has identified the following optical, thermal and mechanical properties for a LIDAR primary mirror: fabrication tolerance = 0.012 λ RMS at λ = 10.6 μm, high thermal conductivity, low thermal expansion coefficient, high stiffness and low density. In addition, the mirror

material must also be capable of fabrication to the shape, figure and size of interest in a cost-effective manner.

In recent years, for NASA spaceborne LIDAR systems, the emphasis has been shifting from the solid monolithic mirrors to lightweight optics. The latter type of mirrors are attractive because they are stiff and stable under thermal, gravity and mounting loads and yet weigh much less than the solid mirrors. The current EOS LASA facility baseline weight has been estimated at 4214 lbs, and the goal is to reduce this weight to about 3300 lbs. The baseline weight of the telescope in the EOS LASA facility has been fixed at about 750 lbs (with a 1.3-m-dia primary mirror) which can be substantially reduced by using a lightweight primary mirror. One aim of this SBIR research program was to advance the state-of-the-art of lightweight mirror technology such that lightweight mirrors could be rapidly fabricated directly in a CVD reactor.

2.2 Candidate Mirror Materials

A number of materials have been considered as candidates for LIDAR mirrors. Some materials, their important properties and figures of merit are listed in Table 1. The important figures of merit are: (i) density, ρ; (ii) pressure and bowing distortion parameter, E, which is the modulus of elasticity; (iii) thermal growth and bowing distortion parameter K/α, which is the ratio of thermal conductivity to coefficient of linear expansion; (iv) natural frequency and inertia loading parameter, E/ρ, which is the ratio of elastic modulus to density and (v) thermal stress parameter, $K/\alpha E$. It is desirable to have high values for all the above figures of merit except the density. It is evident from Table 1 that there is no one material which has the optimum values for all figures of merit. Diamond appears to be the best candidate material, but the technology to obtain large mirrors from diamond does not currently exist. Aluminum is attractive due to high thermal conductivity and low cost, but it is deficient due to a high value of thermal expansion coefficient. Beryllium is lightweight, has high stiffness and thermal conductivity, but Be mirror blanks are expensive and the coefficient of thermal expansion is large, although not as large as that of aluminum. In addition, both these latter metals require alternate plated layers into which are worked the actual optical surfaces.

TABLE 1.

IMPORTANT PROPERTIES AND PERFORMANCE PARAMETERS FOR POTENTIAL MIRROR MATERIALS

Candidate Material	Thermal Conductivity $W/cm^{0}K$ K	Coefficient of Thermal Expansion $K^{-1} \times 10^{-6}$ α	Specific Heat J/gk	Density $Kg/m^3 \times 10^3$ ρ	Modulus of Elasticity Pa x 10^{11} E	K/α W/m x 10^7	E/ρ $\frac{Kgf\ m}{Kg}$ x 10^5	$K/\alpha E$ $\frac{m^2}{sec}$ x 10^{-4}
Silicon	1.5	3	0.71	2.33	1.66	5	72.6	3.02
Mo	1.4	5	0.25	10.2	3.25	2.8	32.5	0.864
Cu	3.98	16.6	0.39	8.92	1.17	2.4	13.4	2.05
Be	2.18	12	1.83	1.85	2.9	1.82	160.0	0.63
Al	2.37	25	0.90	2.70	0.69	0.95	26.1	1.38
Ni	0.90	13	0.45	8.90	2.07	0.69	23.8	0.33
Cr	0.91	6	0.46	7.20	2.49	1.52	35.3	0.61
W	1.78	4.5	0.13	19.35	4.08	3.96	21.5	0.97
Ir	1.47	6	0.13	22.42	5.18	2.45	23.6	0.47
Ag	4.27	19	0.24	10.5	7.6	2.25	7.39	2.97
Au	3.15	14.2	0.13	19.3	8.29	2.22	4.38	2.68
Os	0.61	5	0.13	22.48	5.53	1.22	25.1	0.22
Diamond	6.0	1.5	0.52	3.51	9.54	40.0	277.0	4.2
Titanium	0.2	8.5	0.53	4.5	1.18	0.24	26.8	0.20
BeCu	0.12	16.6	0.42	8.2	1.31	0.07	16.3	0.06
SiC	1.5	4.3	0.76	3.2	3.8	3.49	121.0	0.92
Carbon/Carbon (chopped fiber)	1.38	-.54	0.84	1.99	1.73	25.6	8.86	148
Si_3N_4	--	3	--	3.4	3.0	---	90.2	--
ULE Silica (7971)	0.013	0.03	0.79	2.20	0.67	4.33	31.17	6.45
CERVIT	0.015	0.12	0.92	2.50	1.05	1.0	42.8	0.95
Zerodur	0.06	0.15	0.82	2.55	0.9	4.0	35.3	4.4

The ULE silica glass and CERVIT have low values for coefficients of thermal expansion and density, but these materials also have very low thermal conductivity and a relatively low value of elastic modulus. Further, ULE glass is compositionally and microstructurally inhomogeneous which leads to anisotropy in its thermal expansion. These inhomogeneities and spatial variations in the thermal expansion coefficient become worse as the size of the ULE glass mirror blank increases.

Zerodur glass ceramic is attractive due to its microstructural homogeneity and its low coefficient of thermal expansion, but it also possesses low values of thermal conductivity and elastic modulus. Additionally, the conventional process for fabricating optical components from Zerodur is quite slow, since it involves processing steps such as annealing and ceramization which are time-consuming. There is also a concern regarding thermal hysteresis in Zerodur.

Silicon is attractive because it has moderate values for all the figures of merit. Silicon is a lightweight and hard material having a low value of thermal expansion coefficient and a high value of thermal conductivity. If we compare silicon with ULE silica and Zerodur, materials of current choice for many LIDAR mirrors, we find that silicon is superior. Even though the thermal expansion coefficients of ULE silica and Zerodur glass are much smaller than that of silicon, the important parameter of interest for mirrors is the thermal distortion parameter, K/α, for which silicon has a slightly larger value. Further, the elastic modulus of silicon is larger by a factor of 2.5, and silicon blanks do not require any additional coatings for fabricating an actual optical surface. Finally, CVD-Si has been polished to a surface finish $\approx$ 2 Å RMS.[8-15]

There are two other materials, SiC and Si_3N_4, whose combined thermal and mechanical properties are also superior to those of Zerodur and ULE glass. Both of these materials are stronger, stiffer and harder than Si. CVD-SiC is particularly attractive because it has been polished to yield a surface figure $\leq \lambda/12$ and finish $\leq$ 1 Å RMS. However, the extreme hardness of SiC and Si_3N_4 make fabrication of optical surfaces in these materials slow and costly. Hybrid mirrors consisting of a silicon faceplate bonded to a SiC or Si_3N_4 solid substrate or a honeycomb structure are currently under development for space applications. These hybrid mirrors combine the extremely high

stiffness of SiC and Si_3N_4 with the high polishability of Si. As one component of the hybrid mirror, Si is attractive because (i) its thermal expansion coefficient matches that of SiC and Si_3N_4 (containing a small percentage of TiC) over all anticipated operating conditions and (ii) its modulus of elasticity is smaller than that of SiC and Si_3N_4. The first feature permits bonding or coating of Si to SiC or Si_3N_4 without generating any bi-metallic effects in the hybrid structure while the second feature minimizes bending distortion in the mirror.

The major disadvantage of silicon as a mirror material is its unavailability in large sizes. Several different fabrication technologies such as crystallization, chemical vapor deposition, sintering and hot pressing have been employed to produce Si, but the size of silicon blanks produced from these technologies is currently limited to only a few tens of centimeters in diameter. In LIDAR and astronomical telescope systems, mirrors in the 1-m-dia range are required. Such large sizes are beyond the present or likely capability of single crystal, sintering or hot pressing fabrication technologies. Further, single crystal silicon is susceptible to surface flaw induced fracture, while silicon produced from sintering or hot pressing techniques is of low purity, possesses voids and cannot be polished to a high degree of surface finish. The chemical vapor deposition process has previously been used to produce high quality polycrystalline silicon, but this technology has never been scaled to the sizes of interest for LIDAR applications.

2.3 CVD Approach to Fabricate Si/SiC Mirrors

Using Si and SiC materials, one can employ several technical approaches to fabricate Si/SiC lightweight mirrors via CVD. For instance, one can fabricate (a) the complete mirror from CVD-Si, (b) the mirror faceplate from SiC and backstructure from Si, (c) the mirror faceplate from Si and backstructure from SiC and (d) the mirror faceplate from SiC cladded with Si and backstructure from SiC. As will be shown in Section 3.0, all these approaches were tried at small scale in this program, but they yielded mixed results. For instance, fabrication of Si/Si and SiC/Si mirrors are feasible via CVD, but the Si backstructure does not provide as much stiffness for a given weight as can be achieved from a SiC backstructure. Further, the fabrication of a Si/SiC mirror

(Si faceplate and SiC backstructure) is not feasible because CVD-SiC does not bond well to CVD-Si. Thus all these approaches were discarded in favor of the approach (d) which is described in detail below. A master mandrel with one surface having a "negative" of the actual mirror figure is prepared from high density graphite (Figure 1). The "negative" surface will be polished to as good a surface figure and finish as possible and then coated with a mold release coating. This mandrel is loaded in a CVD furnace and SiC is deposited on it to fabricate a SiC plate of appropriate thickness. After the deposition is completed, both the mandrel and the SiC plate are unloaded. Then the backside of the SiC plate is ground to remove any nodules or irregularities from the surface. A honeycomb lightweight core made of graphite and coated with a release agent is then bonded onto the SiC faceplate, and this piece together with the graphite mandrel are again loaded in a CVD reactor. The SiC is then deposited to fabricate a SiC LWS on the backside of the SiC plate. Since SiC bonds quite well to itself, good adherence of the SiC LWS to the SiC plate is obtained. Next, the deposition is unloaded, and the graphite mandrel is separated from the SiC plate yielding a SiC/SiC mirror blank with a near-net-shape on the front surface. The graphite core is trapped inside the SiC lightweight structure. Since the coefficient of thermal expansion of graphite is larger than that of SiC, during cool-down from the deposition temperature, graphite contracts more than SiC and thus remains within the structure without inducing any stresses. If necessary, the graphite core could be removed by drilling holes at appropriate locations and then burning the graphite in air at 700 C. Next, the SiC faceplate is cladded with Si in a CVD reactor. Since it is relatively less time-consuming to polish Si than SiC, this step is more cost effective particularly when low f-number or aspheric mirrors are fabricated. Finally, the Si surface is optically fabricated to the desired figure and finish.

2.4 CVD of Si and SiC

Silicon and silicon carbide materials were fabricated in a CVD research reactor by the following reactions:

$$SiHCl_3(g) + H_2(g) \text{ -----> } Si(s) + 3\ HCl(g) \quad (1)$$

$$CH_3SiCl_3(g) + H_2(g) \text{ -----> } SiC(s) + 3\ HCl(g) + H_2(g) \quad (2)$$

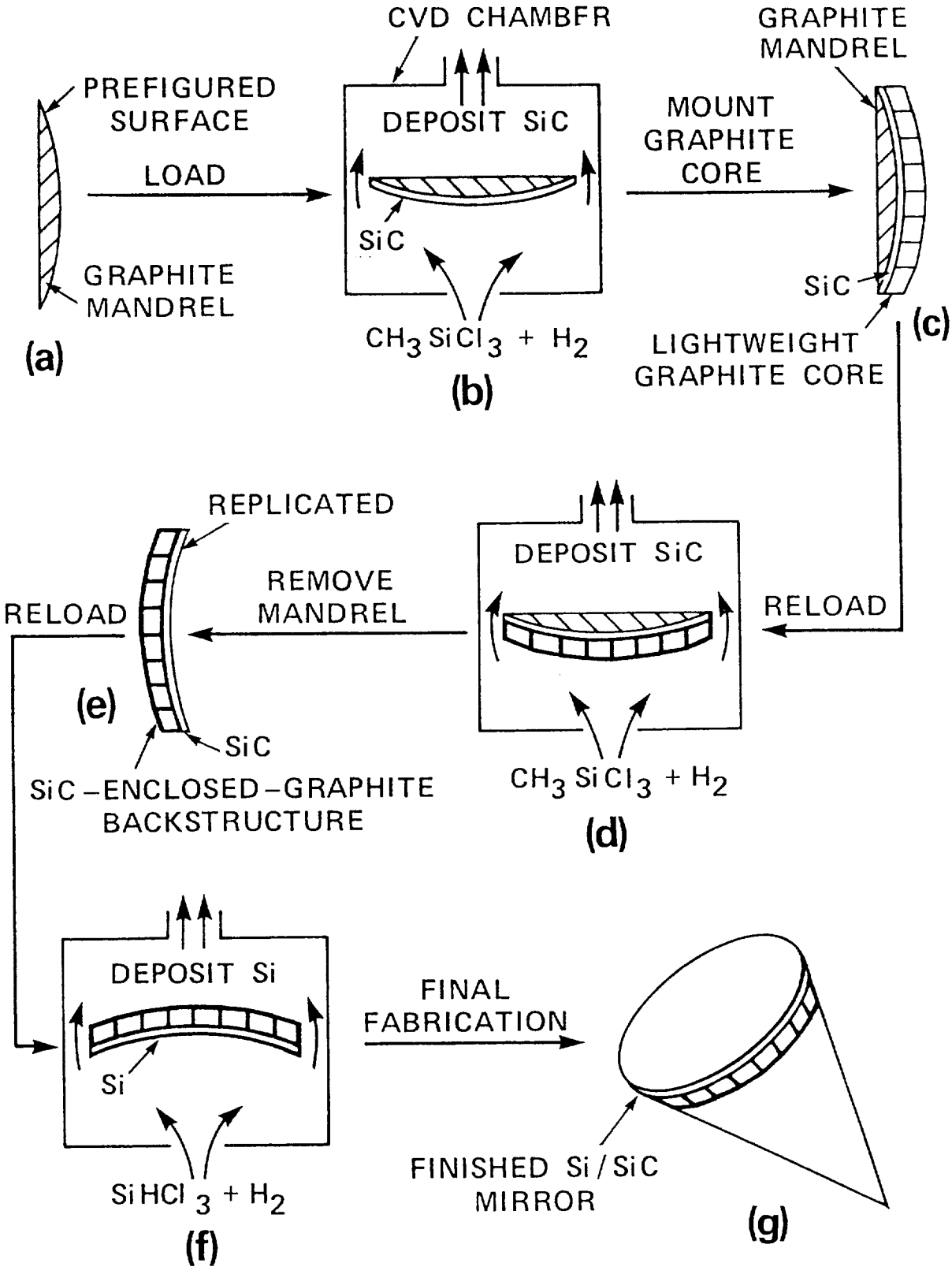

Figure 1. A flow chart to show the various steps required in the fabrication of Si/SiC lightweight mirrors via CVD

In Reaction (1), the source of silicon is $SiHCl_3$ which is a liquid at room temperature with a vapor pressure of 500 torr at 20 C (Figure 2). The $SiHCl_3$ was transported to the deposition area by bubbling argon through the $SiHCl_3$ tank. The important property data of $SiHCl_3$ are given in Table 2.

The source of SiC in Reaction (2) is CH_3SiCl_3 which is also a liquid at room temperature with a vapor pressure of about 140 torr at 20 C (Figure 3). The CH_3SiCl_3 was transported to the deposition area by bubbling argon through the CH_3SiCl_3 tank. The important property data of CH_3SiCl_3 is given in Table 3.

Reactions (1) and (2) have previously been used at CVD Incorporated to produce Si and SiC.[8-10] This prior work was done under two separate Air Force funded programs; one to develop Si for use in actively cooled high energy laser mirrors[8] and the other to develop SiC for use in space-based systems.[10] These materials were characterized for important properties which are listed in Tables 4 and 5. It can be seen from these tables that CVD-Si and SiC are theoretically dense, highly pure, fine grained materials with good mechanical, physical and thermal properties and excellent polishability.

Figure 4 shows the experimental set-up which was used to produce Si and SiC via CVD. It consists of a three-zone Lindberg furnace having a maximum temperature capability of 1500 C. The deposition set-up is enclosed in a 178-cm (7-inch) diameter alumina support tube. The deposition area consists of one mandrel box consisting of four graphite plates of equal size with dimensions 8.9-cm x 30.5-cm arranged as an open square box and up to four baffles arranged in series. The mandrel box is used to deposit Si or SiC on graphite and other substrates while the baffles are used to (i) recover the material which will otherwise go to the scrubber, (ii) partially cover the outlet of the mandrel box thus ensuring a more uniform thickness profile and (iii) fabricate lightweight Si/SiC mirrors in the impinging flow configurations.

The reagents $SiHCl_3/H_2$ or CH_3SiCl_3/H_2 are premixed and introduced into the deposition zone using a water-cooled injector. Keeping the gaseous mixture cold before it enters the deposition area facilitates fabrication of silicon or SiC over a wide range of temperatures. However, utmost care must be taken to ensure that the injector does not leak, because both $SiHCl_3$ and CH_3SiCl_3 react violently with water. The reaction products

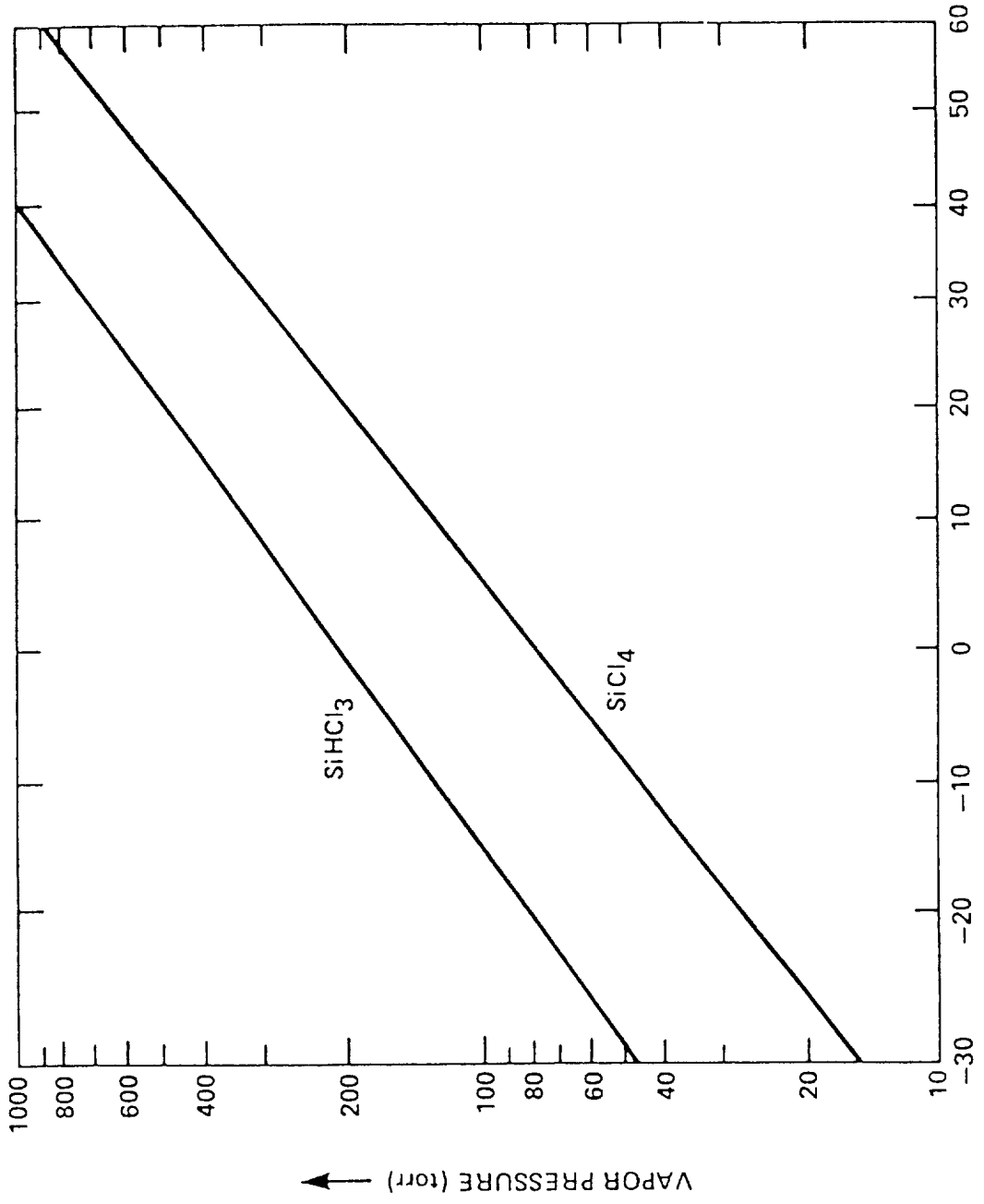

Figure 2. Vapor pressure of $SiHCl_3$ and $SiCl_4$

TABLE 2

IMPORTANT PROPERTY DATA OF $SiHCl_3$

Molecular Weight	135.45
Melting Point at 1 atm	- 50 C
Boiling Point	31.9 C
Density at 25 C, 1 atm	1.3298 g cm^{-3}
Vapor Density (air = 1)	4.67
Flash Point	- 27.8 C
Autoignition Temperature	341.11 C
Lower Flammable Limit in Air (% by volume)	9.8%
Heat of Vaporization at 1 atm	195.33 J g^{-1}
Viscosity at 25 C	0.19 CSTK
Specific Heat at 0 C	0.96 J G^{-1} K^{-1}
Heat of Formation	- 115.2 Kcal/mole
Surface Tension	14.3 dynes cm^{-1}
Coefficient of Expansion	1.9 x 10^{-3} K^{-1}
Refractive Index (0.6 μm) at 20 C	1.402

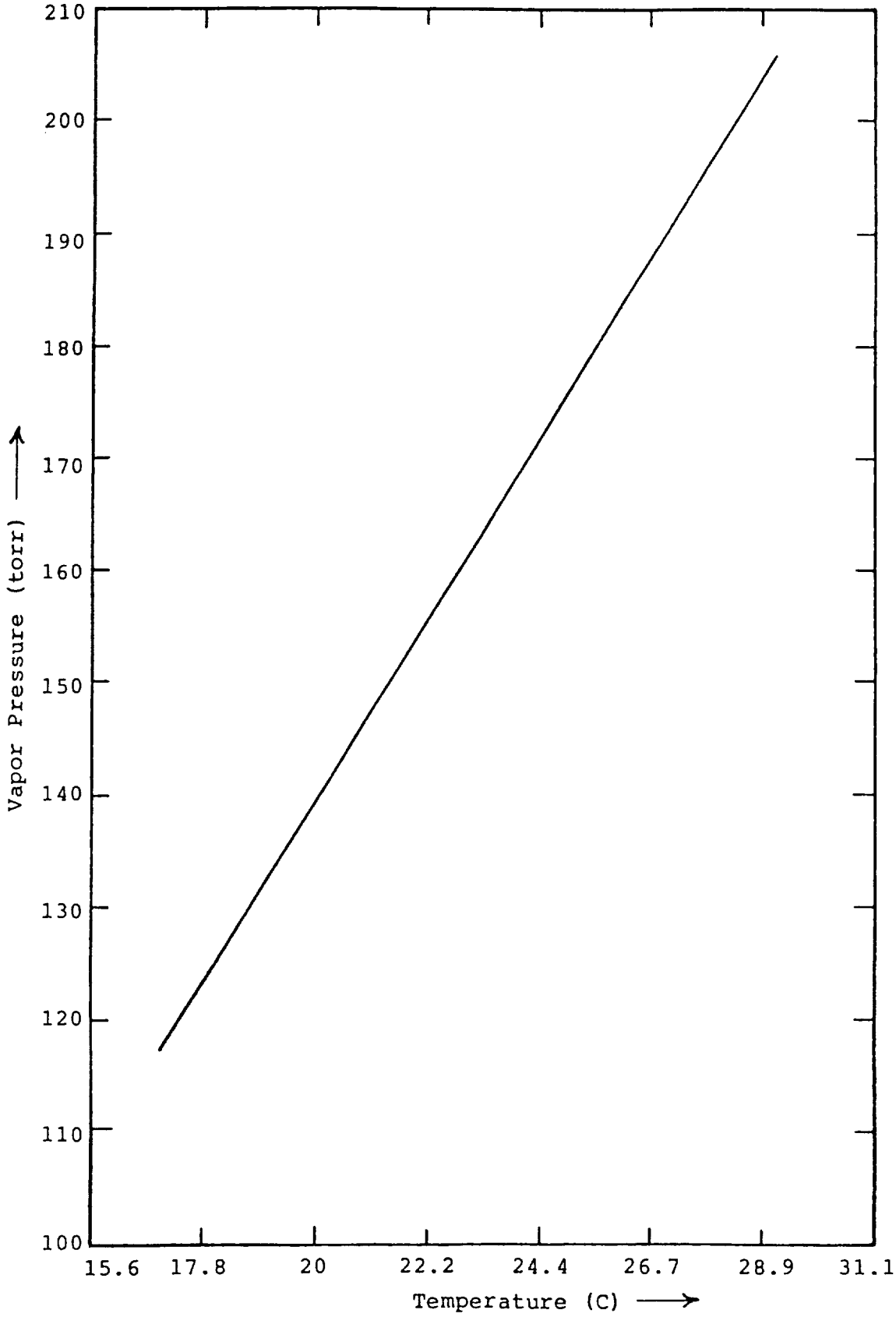

Figure 3. Vapor pressure of methyltrichlorosilane.

TABLE 3

Property Data of Methyltrichlorosilane (CH_3SiCl_3)

State at STP	Liquid
Molecular Weight	149.5
Melting point	- 77.8 C
Boiling point	66.4 C
Specific Gravity (H_2O = 1) at 25 C	1.273
Flashpoint	7 C
Viscosity	0.37 ctsk
Heat of Vaporization	= 7.4 K cal/mole = 206.9 J g^{-1}
Coefficient of Expansion	1.3 x 10^{-3} K^{-1}
Ionization Potential	11.36 eV
Specific Heat	0.92 J g^{-1} K^{-1}
Autoignition Temperature	455 C
Threshold tolerable limit	5 ppm (same as for HCl)
Conditions to avoid	Reaction with H_2O, alcohols etc.
Refractive index (0.6 μm) at 20 C	1.411

Source: Material Safety Data Sheet, Petrarch Systems Inc., Bristol, PA.

Table 4. Important Properties of CVD Polycrystalline Silicon

Property	Value
Density (g cm^{-3})	2.335
Hardness (Knoop) 200 g load	843
Chemical Purity	99.9997%
Trace Elements (ppm)	Cu (1.52) C (1.1) O_2 (0.475)
Flexural Strength (Ksi/MPa)	32/221
Fracture Toughness ($MNm^{-3/2}$)	0.93
Poisson's Ratio	0.24
Elastic Modulus (psi) (GPa)	23.3×10^6 161
Crystal Structure	Polycrystalline with preferred (220) orientation
Grain Size (μm)	Bimodal distribution 0.1 (X-ray diffraction)
Thermal Expansion Coefficient (K^{-1}) at 293 K	2.6×10^{-6}
Specific Heat (J g^{-1} K^{-1})	0.177
Thermal Conductivity (W cm^{-1} K^{-1})	1.0
Polishability (Time: 10 hours)	1.6-2.8 Å RMS
Total Integrated Scatter	16 Å

Table 5. Important Properties of CVD-SiC

Property	Typical Value
Crystal Structure	Face-centered cubic; polycrystalline
Sublimation Temperature (°C)	≈ 2700
Grain Size (μm)	5 - 20
Density (g cm^{-3})	3.21
Hardness (Kg mm^{-2})	
Knoop (500 g load)	2520
Chemical Purity	99.999% SiC
Trace Elements (ppm wgt)	Mn (1.2); Fe (7.3); Co (1.0); Ni (0.6); Cu (5.5); Zn (1.5)
Fracture Toughness, K_{IC} Values	
Micro-indentation (MN $m^{-1.5)}$	3.3
Controlled Flow (MN $m^{-1.5)}$	2.7
Elastic Modulus	
Sonic (GPa/10^6 psi)	466/68
4-point Flexure (GPa/10^6 psi)	461/67
Poisson's Ratio	0.21
Flexural Strength, 4-point (MPa/Ksi)	
@ 298 K	595/86
@ 1673 K	588/85
Coefficient of Thermal Expansion (10^{-6} K^{-1})	
@ 133 K	0.4
@ 173 K	0.8
@ 273 K	1.9
@ 573 K	4.1
@ 1373 K	4.6
Thermal Conductivity (W m^{-1} K^{-1})	
@ 123 K	120
@ 173 K	180
@ 298 K	144
Heat Capacity (J Kg^{-1} K^{-1})	
@ 123 K	250
@ 273 K	700
@ 773 K	1200
Thermal Stability	
(-190 C to 1350 C)	Excellent
Polishability	< 1 Å RMS
BRDF (3°-15° from specular)	≈ 1 x 10^{-5}

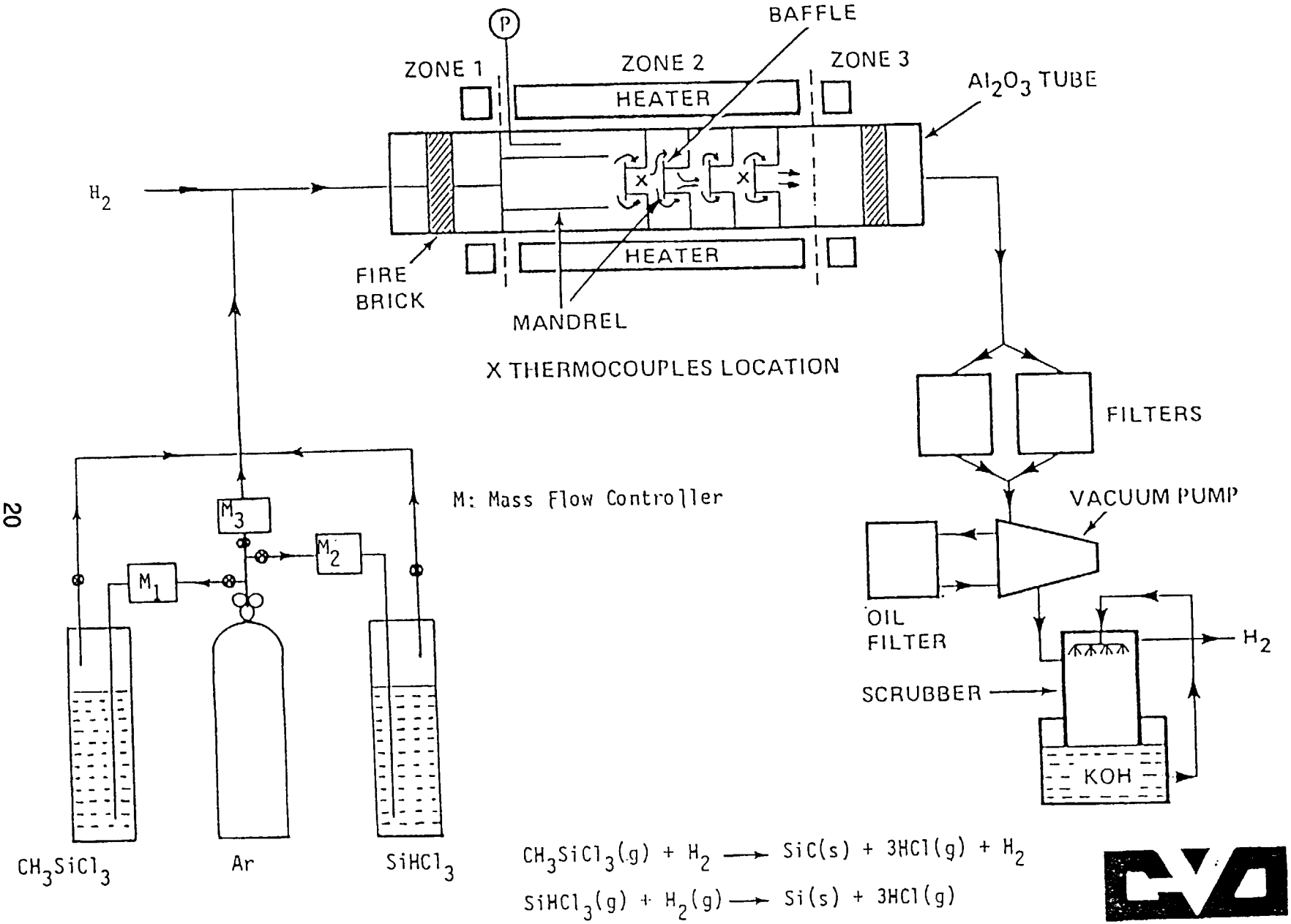

Figure 4. A schematic of research reactor that was used to deposit Si and SiC via CVD

and unreacted reagents that pass through the deposition area first go through a Pyrex wool filter to trap solid Si or SiC particles and then through a scrubber to neutralize $SiHCl_3$ or CH_3SiCl_3 and other chlorine compounds. Finally, the remaining gases (primarily H_2) are diluted with argon and vented to the atmosphere.

In the Air Force program to develop Si-CVD, the Si-CVD process was also scaled from a small, hot wall, horizontal research reactor to a small, vertical, production reactor, and Si pieces 344cm x 25-cm x 0.625-cm were produced. This material was characterized for important properties to ensure that the material was of the same quality as that produced in the research reactor.

2.5 Phase I Summary and Conclusions

Silicon and Si/SiC hybrid materials were fabricated via a scalable and low pressure chemical vapor deposition process to establish the feasibility of producing large, lightweight and near-net-shape Si and Si/SiC hybrid mirrors for use in NASA spaceborne LIDARs.[1] Replication of silicon on several flat substrates including quartz, graphite, Si, SiC, Si_3N_4, Mo, W and Pt, was performed directly in a CVD chamber, and near-net-shape replication was demonstrated on Mo, W and graphite substrates. Based on cost, ease of fabrication, availability in large sizes and the fact that a high degree of surface figure is not required for LIDAR mirrors, graphite was selected as the most appropriate substrate for replication in Phase II. Further, the effects of sample mounting configuration and the surface preparation conditions on replication were determined, and it was found that the degree of replication did not critically depend upon either of these factors.

A silicon/silicon carbide hybrid material was fabricated by depositing CVD-Si on SiC, CVD-SiC on Si, and CVD-Si on graphite followed by deposition of SiC. The deposition of SiC on polycrystalline Si did not yield a good Si-SiC bond. However, these results were preliminary and additional experiments were required to confirm this conclusion. The other techniques yielded a void-free and strong Si/SiC bonds which survived stresses during optical fabrication. One reason for this strong Si/SiC bond was determined to be diffusion of the deposit into the substrate during CVD growth. Optimum CVD conditions for fabricating a Si/SiC hybrid material were determined. For Si

deposition on SiC, the preliminary results showed that Si/SiC bonding was relatively insensitive to both the sample mounting configuration and the surface preparation conditions. For SiC deposition on Si, our analysis indicated that sufficient tensile stresses will be generated in Si to cause its cracking or disintegration due to a thermal expansion mismatch between Si and SiC. To eliminate this potential problem, while depositing SiC, use of dopants such as $TiSi_2$ is recommended.

A scaling analysis of the CVD process which produced Si and SiC was performed, and a qualitative understanding of the process parameters was developed. Important parameters which govern the process scaling in a CVD reactor were identified to be growth rate, furnace pressure, substrate temperature, flow pattern and reagent's molar ratio. Scaling laws were developed, and the feasibility of scaling the process to sizes in the 1-m-dia range was theoretically established.

In conclusion, Phase I effort produced very encouraging results. All the objectives of Phase I research were met. The research performed clearly established the feasibility of fabricating large, lightweight and near-net-shape Si/SiC hybrid mirrors via CVD.

3.0 TASK 1: SMALL SCALE EXPERIMENTS TO DEMONSTRATE FABRICATION OF Si/SiC MIRRORS VIA CVD

3.1 Introduction

The main objectives of this task was to resolve important issues associated with the fabrication of Si/SiC mirrors via CVD and then demonstrate the CVD mirror fabrication technology by fabricating small models of Si/SiC mirrors in the research reactor. In the beginning of this program, the aim was to concentrate on fabricating a Si/SiC lightweight mirror consisting of a Si faceplate and a SiC lightweight stucture. The important technical issues in this approach were (i) replication in Si, (ii) SiC bonding to Si faceplate, (iii) fabrication of SiC lightweight structure and (iv) Si/SiC lightweight mirror fabrication procedure. Small scale experiments to deposit Si and SiC were performed to resolve the above issues, but early in the program it became apparent that the above approach has low probability of success due to the fact that the SiC deposit did not adhere well to the Si substrate. Two possible reasons were identified for this behavior: (i) CVD-SiC was deposited at 1300 C which was close to the melting point of Si. Consequently, constant vaporization from the Si surface occurs which prevents SiC deposit from sticking. (ii) Since Si is very reactive, it reacts with H_2 and CH_3SiCl_3 to form a powdery deposit which prevents CVD-SiC deposit from sticking. Consequently, an alternate approach for fabricating Si/SiC lightweight mirrors was tried. In this approach the mirror faceplate is fabricated from CVD-SiC which is cladded with CVD-Si and the lightweight structure is fabricated from SiC. The important technical issues involved in this approach are (i) near-net-shape replication in SiC, (ii) SiC bonding to itself, (iii) fabrication of SiC lightweight structure, (iv) CVD-Si cladding on SiC and (v) lightweight Si/SiC mirror fabrication procedure. The details of Si and SiC deposition experiments that resolved the above issues are given below.

3.2 Silicon Deposition Experiments

In this program, a total of twelve Si depositions were performed in the research furnace. The details of these depositions are given in Table 6. The first eight Si deposi-

Table 6: Details of Si Deposition Experiments in the Research Reactor

Run No.	Material Deposited	Deposition Thickness mm	Run Time Hrs.	Substrate Temp. C	Furnace Pressure torr	Flow Rate, lpm				Substrate	Remarks
						H_2	CH_4	$SiHCl_3$ or CH_3SiCl_3	Ar		
9081-Si-1	Si	3.0	40	1050	200	7.0	0	1.2	0.0	Dryglide coated Si, SiC, alumina, high density graphite, uncoated Si.	Two coats of dryglide were used. Si deposit stuck to all substrates.
9081-Si-2	Si	-	-	-	-	-	0	-	-	-	Alumina tube cracked during loading.
9081-Si-3	Si	3.0	43	980	200	5.0	0	1.0	0.7	Drylide coated Si, SiC, alumina high density graphite. Uncoated alumina, Si, SiC and high density graphite.	10 coats of dryglide were sprayed. All dryglide coated samples except Si released from deposit. Other substrates except high density graphite stuck to deposit.
9081-Si-4	C Si	1.5	1.0 20	1010 1010	200 200	0.0 5.0	0.020 0.0	0.0 1.2	2.0 0.8	Uncoated Si, SiC, alumina, high density graphite.	CH_4 was passed for one hour to coat substrate with carbon. The deposit stuck to all substrates.
9081-Si-5	Si	1.0	34	960	200	5.0	0.0	1.25	0.85	Dryglide coated SiC, BN coated SiC and Si.	5 coats of dryglide. Si stuck to dryglide coated SiC. Thick coat of BN. Si separated from BN coated SiC and Si. Poor surface finish.
9081-Si-6	Si	1.0	32	960	200	5.5	0.0	1.36	0.85	Hot pressed BN, BN coated SiC.	One coat of BN was sprayed. The Si deposit stuck to both substrates.
9081-Si-7	Si	1.0	28	960	200	5.0	0.0	1.25	0.80	Polished pyrolytic graphite, hard carbon coated Si, dryglide coated Mo.	5 and 1 coats of dryglide on two Mo samples. Si stuck to all substrates except Mo. Poor surface finish.
9081-Si-8	Si	1.0	30	960	200	5.0	0.0	1.25	0.80	Polished pyrolytic graphite, gryglide coated Mo.	5 and 2 coats of dryglide on two Mo samples. Si separated from Mo substrates. Poor surface finish. Si stuck to polished pyrolytic graphite.

Table 6: Details of Si Deposition Experiments in the Research Reactor (contd)

Run No.	Deposition Thickness mm	Run Time Hrs	Substitute Temp (Mandrel) (C)	Furnace Pressure torr (C)	Flow Pulse Rate	Flow Rate, lpm H_2	Flow Rate, lpm $SiHCl_3$	Flow Rate, lpm Ar	Substrate	Remarks
9081-Si-9 (9095-1)	1.0	27	1000	200	ON: 30 sec. OFF: 10 sec.	7.00	1.2	0.5	12" long graphite mandrel. Two baffle with SiC mirror substrates. Three SiC pieces were kept on the mandrel to coat Si.	Gas manifold with multiple holes used. Argon flow 1.0 slpm used with last baffle to deposit selectively. One LWS bonded to SiC plate, and one bonded to Si. Silicon coated all the LWS and SiC.
9081-Si-10 (9095-3)	0.5	16	1000 1020	200	ON: 10 sec. OFF: 10 sec.	7.00	1.0	0.6	6" long graphite mandrel. Two curved replicated SiC plates kept on mandrel. Four SiC mirror substrates on 4 baffles.	Gas manifold with multiple holes used. One mirror is with curved faceplate, three with flat faceplates. Two flat faceplates are polished. One is replicated on graphite. One is separated from graphite. Si coated all the structure.
9081-Si-11 (9095-4)	1.0 (on baffle)	72	970-990	200	ON: 20 sec. OFF: 10 sec.	7.00	1.0	0.6	12" long graphite mandrel. One baffle with SiC mirror substrate. A few SiC pieces on mandrel.	Gas manifold with multiple holes used. One mirror with flat faceplate. Si coated all substrates. High Purity H_2 used.
9081-Si-12	1.0	25	1010-1030	200	ON: 20 sec OFF: 10 sec	7.00	1.0	0.6	Two curved, two flat SiC mirror substrates.	Si coated all substrates.

tions were performed to evaluate Si replication on a variety of coated and uncoated substrates such as SiC, Si_3N_4, BN, Mo, W, graphite and alumina while the remaining depositions were performed to demonstrate Si cladding on SiC mirror substrates. The aim of the replication experiments was to deposit the Si and then separate the deposit from the substrate yielding a high degree of surface replication in Si. The aim of the Si cladding experiments was to obtain good adherence between the Si deposit and the substrate.

In deposition Run #9081-Si-1, Si was deposited on dryglide* coated Si and SiC samples. Only two coats of dryglide were sprayed to seal the polished Si and SiC substrates. For comparison, substrates made of uncoated alumina, high density graphite, Si and SiC were also used. However, the deposit stuck to all substrates. Apparently, the amorphous carbon coating did not release the deposit. Carbon coated samples can also be viewed as "dirty samples," and one would expect these substrates to separate from the Si deposit but this did not happen.

Deposition Run #9081-Si-2 was aborted because the furnace support tube made of alumina cracked during loading. In deposition Run #9081-Si-3, ten coats of dryglide were sprayed on polished Si, SiC, alumina and high density graphite substrates. For comparison purposes, corresponding uncoated samples were also used in the same experiment. The Si deposit separated readily from all dryglide coated substrates except Si, while all uncoated substrates except high density graphite stuck to Si. This shows that a carbon coating of sufficient thickness is beneficial in releasing the Si deposit from SiC, alumina and high density graphite.

Examination of the replicated Si samples under a microscope showed that the surface quality of these samples was fair on dryglide coated SiC and uncoated high density graphite but relatively poor on dryglide coated alumina and high density graphite. However, the replicated Si samples did show high values of reflectivity in the infrared. A comparison of surface morphology of a high density graphite substrate and the replicated Si sample showed that surface features including defects were quite well

*dryglide is a suspension of carbon in a solvent.

replicated. However, the Si surface was not smooth, and small black particles which appeared to be of graphite were scattered all around the surface. Therefore, it is apparent that even on an uncoated high density graphite substrate, a high degree of replication may be difficult to achieve.

Similar results were also obtained on the replicated Si samples deposited on the dryglide coated SiC. Small amorphous carbon particles scattered over the surface were observed. Also visible were a few surface defects and scratches. The surface finish on this sample was much better than that of the Si replicated on dryglide coated graphite. However, further improvement is desirable.

Although the amorphous carbon coating was very effective in releasing the deposit from the mandrel, it tarnished the polished surface and also degraded the optical figure. One can improve the surface finish and figure if extremely thin continuous carbon coatings (≈ 10-1000 Å) could also release the deposit. These thin coatings could be applied either in a CVD reactor or by sputtering. However, the former method is preferred, because it could be done as part of the overall CVD replication process.

Therefore, in deposition Run #9081-Si-4, an attempt was made to apply a carbon coating via CVD prior to Si deposition. A mixture of CH_4 and Ar was passed through the reaction zone for about one hour prior to starting the Si deposition. The substrates used were uncoated Si, SiC, alumina and high density graphite. However, this technique did not succeed, and the Si deposit stuck to all substrates. It should be emphasized that the negative result does not imply that a carbon coating deposited by CVD will not work. Additional effort is required to determine the minimum carbon coating thickness which will readily release the Si deposit from the substrate.

In deposition #9081-Si-5, polished SiC was coated with five coats of dryglide. Reducing the quantity of dryglide will presumably improve the surface finish of the replicated Si, if the Si deposit separates from the substrate. The dryglide coated surface was also examined prior to deposition under a microscope and no pin-holes were detected in the coating. Two additional samples of polished SiC and Si covered with a thick coating of BN were also loaded in the reactor. Since BN is an inert material widely used for mold release, it is a good coating material to try.

Examination of the Si deposited substrates showed that Si stuck to the dryglide coated SiC but separated readily from the BN coated substrates. This shows that a relatively thicker coating of dryglide is necessary to obtain deposit release from SiC. The surface quality of the Si samples replicated on BN coated SiC and Si was found to be poor.

In deposition #9081-Si-6, it was decided to obtain more data on the BN coating. Therefore, a sample of hot pressed BN and another sample of polished SiC covered with one coat of BN were used as substrates. However, the Si deposit stuck to both these substrates indicating that a relatively thick coating of BN will be required to obtain release of the Si deposit.

In conclusion, a relatively thick coating of carbon or BN applied on different substrates, in particular, SiC can release the Si deposit. The use of these coatings, however, will not provide a high degree of replication in Si.

In deposition #9081-Si-7, it was decided to explore other approaches. One approach is to use polished pyrolytic graphite and hard carbon coated Si as substrates. Pyrolytic graphite produced by a CVD process is theoretically dense, void free and can be polished to a high degree. The hard carbon coating can be applied on Si by a CVD process. This coating seals the Si surface without appreciably affecting the surface finish of the Si sample.

Another substrate tried in deposition #9081-Si-7 was dryglide coated Mo. In Phase I it was shown that Si deposits released readily from Mo substrate but that the substrate surface was etched. Consequently, if a thin coating of dryglide is applied to polished Mo sample, it might prevent etching. Therefore in Run #9081-Si-7 four substrates (pyrolytic graphite, hard carbon coated Si, dryglide coated Mo (two samples) were loaded in the reactor. The substrates were kept on top of the mandrel and Si was deposited. Examination of the deposit revealed that Si stuck to all substrates except Mo. Further, the surface of the Mo substrate was not etched but it was spotty.

In Run #9081-Si-8, the polished pyrolytic graphite and dryglide coated Mo samples were again tried, but this time these substrates were mounted in a recessed configuration, i.e., below the surface of the mandrel. Again, Si deposit stuck to pyrolytic graphite but

readily separated from Mo. Examination of the Mo surface showed that it is not etched but is dull in color.

In summary, a variety of substrates and coatings were tried to obtain a high degree of replication in CVD-Si. All these results are summarized in Table 7. No substrate or coating could be identified which will do both, release the deposit as well as provide good surface finish. Near-net-shapes can be obtained on dryglide coated graphite, Mo and W substrates. Of these, since the surfaces of Mo and W become etched, dryglide coated grahite is the preferred substrate. Better replicated surface quality can be obtained by using dryglide coated polished SiC substrates. In all cases mentioned above, however, a final polishing of the Si replicated surface will be required. Since graphite is inexpensive, easy to polish and is readily available, it appears to be the preferred substrate for Silicon replication.

In deposition Nos. 9081-Si-9 through 9081-Si-12, the aim was to clad faceplates of SiC lightweight mirror substrates with CVD-Si and also to fabricate Si/SiC samples to be used for flexural strength measurement. The mirror substrates were mounted in an impinging flow configuration whereas SiC samples were placed on the mandrel. The deposition area had provision to mount up to four mirror substrates in series on four baffles as shown in Figure 5. Since the first mirror substrate directly faces the injector, an "injector mark" in the form of enhanced deposition is obtained on this faceplate.

In order to improve deposition uniformity and eliminate the injector mark, a manifold plate was designed and placed on top of the injector. This manifold plate had eight holes, each 1.25-cm (0.5-in) in diameter, located in an annular region of radii 1.25-cm (0.5-in) - 7.5-cm (3.0-in) from the injector. The flow impinged in the center of the manifold and then was equally distributed to these holes. This arrangement increased the effective diameter of the jet thereby reducing its velocity and thus the direct impingement on the first mirror.

In all of these depositions, the flow of $SiHCl_3$ was also pulsed, keeping the flow "off" time constant and equal to 10 seconds. A pulsed flow was used because it suppressed the growth of nodules, reduced the material grain size and thus provided a more uniform deposit on the faceplate. Both polished and replicated SiC faceplates were

Table 7: Silicon Replication Results

S. NO	MANDREL	DEPOSIT ADHERENCE * TO MANDREL	REMARKS
1.(a)	CVD Si	Adherent	
(b)	CVD Si Coated With Dryglide	Adherent	2 coats of dryglide
(c)	CVD Si Coated With Dryglide	Adherent	10 coats of dryglide
(d)	CVD Si Dipped In Dryglide	Adherent	
(e)	CVD Si Coated With BN	Separated	Thick coating of BN
(f)	CVD Si Coated With Hard Carbons	Adherent	0.5µm CVD Carbon coating
2.	Single Crystal Si	Adherent	
3.(a)	CVD SiC	Adherent	
(b)	CVD SiC Coated With Dryglide	Good Release	10 coats of dryglide Average surface finish
(c)	CVD SiC Coated With Dryglide	Adherent	5 coats of dryglide
(d)	CVD SiC Coated With BN	Adherent	Thin coat of BN
(e)	CVD SiC Coated With BN	Separated	Thick coat of BN Poor surface finish
4.	Hot Pressed BN	Adherent	
5.(a)	ATJ Graphite	Adherent	
(b)	ATJ Graphite Coated With Dryglide	Good Release	30 coats of dryglide Poor surface finish.
(c)	Polished Pyrolitic Graphite	Adherent	
6.(a)	Polished High Density Graphite	Separated	Average surface finish
(b)	High Density Graphite Coated With Dryglide	Separated	10 coats of dryglide Average surface finish
7.	Hot Pressed Si_3N_4	Adherent	

Table 7: Silicon Replication Results (contd)

S. No.	MANDREL	DEPOSIT ADHERENCE* TO MANDREL	REMARKS
8.(a)	Mo	Separated	Mandrel etched Near net shape
(b)	Mo Coated With Dryglide	Separated	5 and 2 coats of dryglide. Poor surface finish
9.	W	Separated	Mandrel etched Near net shape
10.	Pt	Mandrel Melted	
11.	Quartz	Adherent	Mandrel cracked
12.	Sapphire	Adherent	Mandrel cracked
13.(a)	Alumina	Adherent	High stress
(b)	Alumina Coated With Dryglide	Good Release	10 coats of dryglide Average surface finish
14.	Glass	Adherent	

*The quality of adherence was assumed very good if the coating did not delaminate, crack, or peel off during thermal cycling (0° to 100° C) and/or during cutting, grinding, lapping, and polishing operations.

used as substrates. Since the replicated SiC surface is normally coated with graphite due to replication on graphite, it is important to know whether it is nececssary to remove this graphite coating to obtain good adherence.

When Si is cladded on the faceplate of the mirror substrates, it is important that Si does not deposit on other areas of the mirror. Consequently, masking techniques are required to obtain selective Si deposition. The selective Si deposition also isolates the Si cladding on the faceplate from the rest of the furnace and thus prevents cracking of the clad layers. Two masking techniques were tried in deposition #s 9081-Si-9 through 12. First, a gas flow techniqu2e was used, in which two rows of overlapping holes were drilled on a small annular region around the substrate. Argon was passed through these holes to cover this region with an inert gas shroud which should prevent Si deposition on these areas. In the second technique, a 1.25-cm long hollow cylinder open at one end was fabricated from grafoil.[**] This cylinder had the same diameter as the substrate. This cylinder was bonded onto the backside of the SiC faceplate and sealed with graphite cement. The Si deposit occurred on this cylinder which protected the backside of the SiC faceplate and the LWS. The Si coated grafoil cylinder was readily removed by piercing it with a pointed tool.

On examination, all the Si depositions yielded very encouraging results. The Si cladding adhered quite well to both polished as well as replicated faceplates. This shows that the replicated facaeplates can be directly clad with Si without performing any further surface treatment. The gas flow technique did not yield selective Si deposit, but the masking technique using grafoil worked quite well.

A total of four Si/SiC lightweight mirrors were fabricated by following the above procedure. Two of these had flat faceplates while two were curved. The two flat mirrors were optically fabricated by MI/CVD and came out quite well. The Si cladding survived all the stresses associated with optical fabrication. The curved Si/SiC mirrors were sent to Opticraft, Woburn, MA for optical fabrication. One Si/SiC lightweight mirror with a

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[**]Grafoil is a thin, flexible foil made of graphite.

square shaped faceplate was also edge ground successfully to a round shape. This step did not chip or spall the Si cladding indicating that the Si-SiC bond is perhaps chemical in nature.

Since some spare capacity was available in these four Si depositions, it was decided to fabricate Si and SiC/Si lightweight mirrors. In the former case, the faceplate and LWS material is Si, while in the latter case, the faceplate material is SiC but the LWS is Si coated. Both these concepts also worked quite well. The Si coated lightweight graphite core bonded quite well to Si and SiC faceplates.

Several Si clad SiC samples were also fabricated in these four depositions. Twelve samples of this material were prepared to measure flexural strength and elastic modulus with a four point loading arrangement. The average elastic modulus of the material was measured to be 227.8 ± 10.8 GPa which lies between the elastic modulus of Si (≈ 159 GPa) and SiC (≈ 450 GPa). The flexural strength values were measured to be 229.4 ± 29.7 MPa and 338.3 ± 45.9 MPa when Si and SiC layers were stressed in tension, respectively. The details of these results are given in Reference 10 which is also included in Appendix II of this report. These data essentially indicate that bonding between Si and SiC is very strong and perhaps chemical in nature.

In summary, from these Si deposition experiments we can draw the following conclusions: (i) Silicon can be replicated on several substrates such as Mo, W and graphite. A thick coating of amorphous carbon facilitates the replication process. A high degree of replication could not be achieved in CVD-Si. (ii) CVD-Si bonds quite well to CVD-Si and SiC substrates. This adherence does not depend upon the substrate shape or the surface preparation. The flexural strength data indicated that the bonding between CVD-Si and SiC is perhaps chemical in nature.

3.3 SiC Deposition Experiments

The main objective of the SiC depositions was to demonstrate the following: (i) SiC bonding to Si substrate, (ii) fabrication of SiC lightweight structure, (iii) fabrication of near-net-shape SiC faceplate and (iv) CVD-SiC bonding to itself. A total of fifteen SiC deposition experiments were performed in the research furnace to resolve the above

mentioned issues. The details of these depositions are given in Table 8. The first five depositions were performed primarily to obtain a good bond between the CVD-SiC deposit and the Si substrate, although these depositions were also used to fabricate honeycomb structures of different shapes as well. Deposition Nos. 6-8 were performed to fabricate hexagonal cell honeycomb structures and near-net-shape SiC faceplates on graphite mandrels. Finally, depositions Nos. 9-15 were performed to fabricate both curved and flat SiC faceplates, lightweight structures and mirror substrates.

In deposition Nos. 9081-SiC-1 through 9081-SiC-3, bonding of CVD-SiC deposits onto Si substrates was attempted by applying coatings of carbon on Si substrate. In Phase I, small scale experiments showed that when SiC is deposited on CVD-Si, good adherence is not assured. However, since SiC bonds quite well to itself, coating the Si surface with carbon will lead to formation of SiC at high temperature which should bond well to the SiC deposit. Consequently, in deposition #9081-SiC-1, a Si faceplate was coated with an amorphous coating of carbon. A square cell honeycomb structure 5-cm x 5-cm in area was fabricated from graphite plates about 1.6-mm thick. Square cells with an aspect ratio (cell depth/cell width) varying from 1 to 4 were used to determine the effect of aspect ratio on deposition uniformity. The honeycomb structure was attached to a dryglide coated Si faceplate with graphite cement, and the assembly was mounted on the bottom mandrel plate close to the injector. Silicon carbide was deposited by flowing the reagent parallel to the mandrel surface.

Examination of the SiC deposit revealed that the honeycomb graphite structure was coated quite well with SiC. The top surfaces of all the walls were rounded and no sharp corners or edges were observed. This is an attractive feature of CVD technology, since it minimizes the possibility of stress concentration. The thickness uniformity appeared quite good from cell to cell, but within a square cell it was quite nonuniform. This shows that the flow in each square cell was mass transport limited. One can, in principle, deposit in a kinetically limited regime and improve the thickness uniformity of the deposit.

Examination of the Si-SiC bond showed that the SiC stuck to Si in some places. The honeycomb structure was sliced and lapped to see if delamination occurs. The Si-

Table 8: Details of SiC Deposition Experiments in the Research Reactor

Run No.	Material Deposited	Deposition Thickness mm	Run Time Hrs.	Substrate Temp. C	Furnace Pressure torr	Flow Rate, lpm				Substrate	Remarks
						H_2	CH_4	CH_3SiCl_3	Ar		
9081-SiC-1	SiC	3.0	30	1300	200	3.2	0	0.8	2.0	Square cell graphite honeycomb structure placed on dryglide coated Si faceplate.	The graphite structure was coated with SiC. The SiC deposit partially stuck to Si faceplate.
9081-SiC-2	SiC	3.0	30	1300	200	3.2	0	0.8	2.0	Square cell graphite honeycomb structures placed on dryglide coated Si faceplate.	The SiC deposit coated the graphite structure. SiC did not stick to Si faceplate.
9081-SiC-3	Carbon		1.0	1300	10	0	0.020	0	2.0	Polished Si, lapped Si.	Discontinuous carbon film on Si. Soot formation.
9081-SiC-4	Si	~ 0.05	0.5	1175	200	6.0	0.0	0.6 ($SiHCl_3$)	0.4	Hexagonal and triangular cell honeycomb structure placed on Si faceplate.	Si/SiC deposit coated the graphite structure. SiC partially stuck to Si. Injector clogged.
	Si/SiC	-	1.0	1175-1250	200	5.0	0.0	0.20-0.8	....		
	SiC	2.0	29.5	1250-1290	200	3.2	0.0	0.8	2.0		
9081-SiC-5	Si	~ 0.10	1.75	1160-1300	200	....	0.0	0.20-0.8	0.5 -2.0	Hexagonal cell honeycomb structure placed on Si faceplate.	Si/SiC deposit coated the graphite structure. SiC did not stick to Si.
	SiC	2.0	28	1300	200	3.2	0.0	0.8	2.0		
9081-SiC-6	SiC	1.5	16	1315	200	3.0	0.0	0.8	2.0	Hexagonal cell honeycomb structure placed on graphite.	SiC coated the graphite core. SiC deposit was quite thin at the base. SiC base cracked during separation.
9081-SiC-7	SiC	4.5	30	1315	200	3.0	0.0	0.8	2.0	Hexagonal cell honeycomb structure placed on SiC. Curved and flat pieces of graphite.	SiC coated all the substrates. SiC light-weighted structure fabricated SiC replicated on graphite.
9081-SiC-8	SiC	4.0	24.5	1315	200	3.0	0.0	0.8	2.0	Curved and flat pieces of graphite.	SiC replicated on graphite. Good separation and replication achieved.

Table 8: Details of SiC Deposition Experiments in the Research Reactor (contd)

Run No.	Deposition Thickness mm	Run Time Hrs	Substrate Temp C	Furnace Pressure torr	Flow Rate, lpm H_2	CH_4	CH_3SiCl_3	Ar	Substrate	Remarks
9081-SiC-9	2.0 - 3.0	25	1275-1335	200	3.0	0.0	0.8	4.0	One flat, one curved substrate one LWS core bonded to flat SiC facep	Three baffle configuration. Light-weighted structure on the third baffle had carbon rich SiC deposit.
9081-SiC-10	1.0	10	1300-1340	200	5.0	0.0	0.8	2.0	Two LWS core bonded to curved and flat SiC plates.	Two baffle configuration. Graphite LWS core on curved plate did not bond well and fell down. SiC coated the other structure.
9081-SiC-11	1.0 - 2.0	22	1300-1330	200	6.0	0.0	1.0	2.5	Two curved, one flat and one LWS made of H-490 graphite.	4 Baffle configuration-(curved-flat-curved-LWS). Good SiC coating achieved on all substrates. Flat replicated sample cracked.
9081-SiC-12	2.0	25	1300	200	6.0	0.0	1.0	2.4	Two flat substrates Two LWS bonded to curved plates.	4 Baffle configuration (flat-flat-curved-curved). SiC coated all substrates quite well.
9081-SiC-13	2.0	27	1300-1315	200	6.0	0.0	1.0	1.0 (HTS) 1.0 (central injector)	Two LWS bonded to flat plates. Two curved substrates.	4 Baffle configuration, (curved-flat-flat-curved) Gas flow: Ar 1.0 slpm through 4th baffle holes for deposit isolation. Deposition temperature in the exhaust dropped from 1315 at the beginning to 1295 after 10 hours. Expt performed in a pulsed mode. ON-20 sec, OFF-10 sec. Gas manifold with multiple holes used. All substrates were coated quite well with SiC.
9081-SiC-14	2.0	30	1275	200	6.5	0.0	1.0	2.0	Two curved, two flat plates	Pulsed flow ON: 5 sec, OFF: 10 sec. All substrates were coated well. Selective deposition technique using grafoil tried.
9081-SiC-15	2.0	26	1300	200	6.5	0.0	1.0	2.0	Four LWS bonded to flat and curved plates.	Pulsed flow ON: 5 sec., OFF: 10 sec. Three substrates were coated quite well.

SiC bond survived all the fabrication stresses. Thus, coating a Si faceplate with carbon is a potentially useful technique to obtain good bonding between Si and SiC.

In deposition Run #9081-SiC-2, the previous experiment was repeated. The results were similar to those obtained previously except that the SiC deposit did not stick to the carbon-coated Si faceplate. Therefore, it seems that coating a Si faceplate with amorphous carbon is not a very reliable technique to obtain good bonding between Si and SiC.

In Run #9081-SiC-3, the deposition of a thin continuous coating of carbon was attempted by passing CH_4 and Ar through the reaction zone for one hour. Polished and lapped samples of Si were used as substrates. Examination of the Si samples revealed a discontinuous carbon film on the Si surface. This film was covered by soot. The soot could easily be wiped off. Review of literature on carbon films deposited by CVD indicated that continuous laminar carbon films can be obtained by optimizing the CVD process conditions.

In deposition Nos. 9081-SiC-4 and 5, the CVD deposition was started with Si and gradually changed to that of SiC. The rationale for this approach was that since Si adheres well to Si and SiC to SiC, this technique should provide a good bond between Si and SiC.

In Run #9081-SiC-4, one hexagonal and one triangular honeycomb structure (Fig. 5) were cemented onto two different Si faceplates and loaded in the CVD reactor. The hexagonal structure was placed on the baffle plate facing the flow while the triangular structure was placed on a mandrel such that the flow was parallel to the structure. Initially, $SiHCl_3$ and H_2 were passed to deposit Si and gradually $SiHCl_3$ was replaced with CH_3SiCl_3 to deposit SiC. The deposition was terminated after 31 hours.

Examination of the honeycomb structures showed that Si/SiC deposit coated the graphite structures quite well. The coating was more uniform on the hexagonal structure than on the triangular structure. This difference was due to the location of the two structures with respect to the flow. On the baffle plate, the deposition was quite uniform across the structure but there was thickness nonuniformity along the depth of the structure. On the mandrel plate, the thickness was nonuniform, both across as well as

along the depth of the structure. This shows that lightweight structures with uniform deposition can be fabricated when they are facing the flow.

Efforts were made to separate the Si faceplate from the honeycomb structures. The Si faceplate attached to the hexagonal structure broke and a small piece of Si was separated from the faceplate showing that SiC partially stuck to Si. However, the triangular SiC structure stuck quite well to the Si faceplate.

In Run #9081-Si-5, the previous experiment was repeated with the following differences: (i) Only one honeycomb structure was loaded in the reactor and (ii) CH_3SiCl_3 was used as a source for both siliconized SiC as well as SiC. By varying the temperature and molar ratio of H_2 to CH_3SiCl_3, one can deposit either pure SiC or siliconized SiC.

Examinatiion of the hexagonal structure showed that Si/SiC deposit has coated the graphite structure quite well. However, the structure did not stick to the Si faceplate. A whitish powder was noticed in between the Si faceplate and the SiC structure. It is not clear how this whitish powder was created in the reactor at the start of the deposition. Perhaps, a small quantity of O_2 (air) was present in the beginning and produced SiO_2 powder.

Table 9 summarizes all the Si/SiC hybrid material fabrication results obtained in this program. From this table we see that one technique which is both effective and reliable in obtaining Si/SiC hybrid material is the deposition of CVD-Si on SiC. Other techniques worked with mixed results and could not be relied upon. This conclusion coupled with the fact that high quality Si replication was not obtained, indicated that the technical approach of fabricating a Si/SiC lightweight mirror, by replication of a Si faceplate on a suitable mandrel followed by fabrication of a SiC LWS on the backside of the faceplate, should be modified to that of fabricating a Si clad SiC faceplate and fabricating a SiC LWS on the backside of this faceplate.

The goal of deposition #9081-SiC-6 was to fabricate a SiC honeycomb structure on a graphite core and separate it from the graphite mandrel. The graphite core was fabricated from 0.5-mm (0.020-in) thick graphite ribs each 3.13-cm x 3.13-cm (1.25-in x 1.25-in) in area. A total of 12 graphite ribs were used to fabricate a structure consisting of a hexagonal cell with six triangular cells on the inside as shown in Figure 5. The

Table 9: Si/SiC Hybrid Material Fabrication Results

S. NO	SUBSTRATE	DEPOSIT	REMARKS
1	SiC	CVD Si	Good and Reliable Adherence
2	Graphite	Si/SiC	Good Adherence Material Cracked
3(a)	Polished CVD Si	SiC	No Adherence
3(b)	Ground CVD Si	SiC	No Adherence
3(c)	Dryglide Coated Si	SiC	Adherence Unreliable
3(d)	Ground CVD Si	Si/Si-SiC/SiC	Partial Adherence
3(e)	Ground CVD Si	Siliconized SiC/SiC	No Adherence

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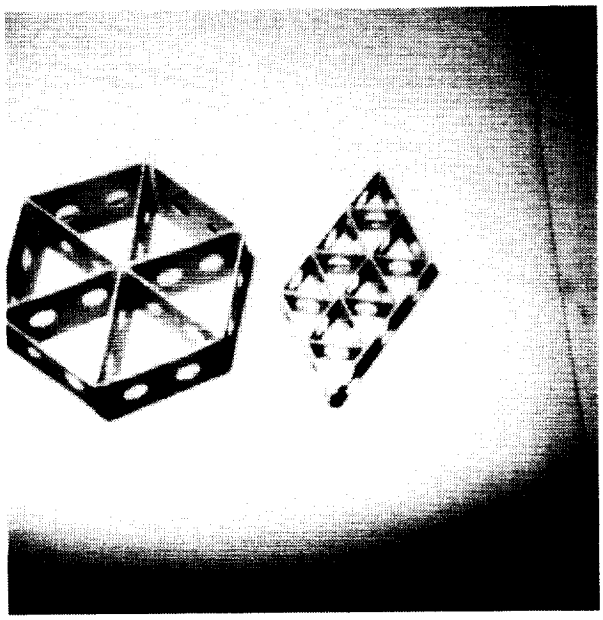

Figure 5. Hexagonal and triangular honeycomb graphite structures used to fabricate SiC light-weighting structures

graphite ribs were bonded together and to the graphite mandrel at the base with graphite cement. There were five holes, each of diameter 0.625-cm (0.25-in) drilled symmetrically on the graphite ribs as shown in Figure 6. There were two holes near the top, two near the bottom and one hole in the center of each graphite rib. The aim of these holes was to create paths for the gaseous reagents to flow freely within the cells thereby improving the deposition uniformity.

Examination of the structure after the SiC deposition showed that the graphite core had been completely enclosed by the SiC deposit. There was no evidence of cracking anywhere in the hexagonal or triangular cells. There were no sharp edges and all the corners appeared rounded. The deposition appeared quite uniform from cell to cell but was tapered along the depth of the cell, i.e. the deposit thickness was larger at the top than at the base of the cell. Further, contraction striations inside the holes and on the top edges of the cell walls were also observed. These striations were more pronounced on the top edges of the outside walls and on the outside than on the inside edges. Table 10 lists the measurements of the diameters of the holes on the six outside walls of the hexagonal cell. Note that all holes had a diameter of 0.625-cm (0.25-in) initially. We see that the diameters of the two top and two bottom holes are the same within 2.5%. However, there is a decrease in deposit thickness from the top holes (1.0-mm) to the bottom holes (0.50-mm). Although this variation, which was over a distance of 2-cm, is large, it may actually be desirable. Analysis performed at United Technologies Optical Systems under the AF Program showed that tapered walls increase the stiffness of the SiC LWS.

Attempts were made to separate the SiC LWS from the graphite mandrel. The structure did not have a clean separation. The SiC deposit at the bottom of the structure was quite thin and consequently cracked. This made it possible to examine the cross-section of the LWS at the base. There was very little deposit at the bottom of the six inside walls. Figure 7 shows the cleaved cross-section of the outside wall near the base. We see graphite 0.5-mm thick in the center and the SiC deposit about 0.65-mm thick on the outside and 0.32-mm thick on the inside. However, this difference may not be that important for our applications because both sides are joined together through the holes.

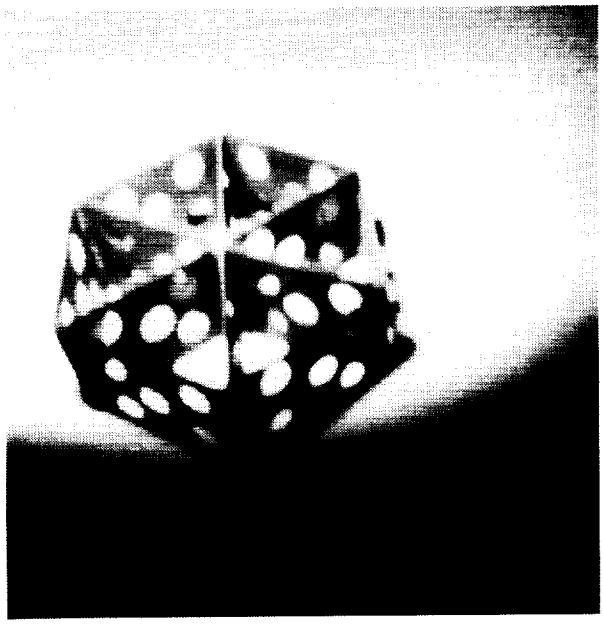

Figure 6. A hexagonal shaped lightweight structure made of 0.5-mm-thick graphite ribs. Cell length = 3.13 cm.

TABLE 10: DIAMETER OF HOLES ON THE OUTSIDE WALLS OF THE LIGHT-WEIGHTED STRUCTURE

All dimensions are in mm

HOLE NO.	LOCATION	RIB #1	RIB #2	RIB #3	RIB #4	RIB #5	RIB #6
1	TOP	4.33	4.37	4.35	4.27	4.19	4.31
2	TOP	4.19	4.37	4.41	4.29	4.18	4.29
3	CENTER	4.87	4.89	4.87	4.79	4.68	4.83
4	BOTTOM	5.29	5.29	5.35	5.26	5.20	5.23
5	BOTTOM	5.29	5.41	5.29	5.26	5.20	5.23

Figure 7. Cleaved cross-section of cell wall near the base. The black region is graphite core.

Further, the wall thickness at the top of the LWS was also mesured and compared. On the outside and inside walls the measured thicknesses were in the range 3.70-mm to 4.10-mm and 3.60-mm to 3.83-mm, respectively. Thus, the outside walls are slightly thicker than the inside walls.

In summary, there is a deposition non-uniformity at several places in the lightweight structure. Nevertheless, the structure is quite strong and rigid. It is not clear at this stage whether this deposition nonuniformity will affect the mirror performance in any manner. As will be shown later, one can use techniques to improve the thickness uniformity along the cell depth but the testing of the actual mirror will determine if improved thickness uniformity is desirable.

In deposition #9081-SiC-7, the graphite core was bonded onto a flat SiC plate with graphite cement and loaded in the CVD reactor. The surface of the SiC plate was generated flat but not lapped. The graphite LWS had two semicircular holes drilled at the bottom of each wall to permit more uniform flow of the gaseous reagents. The aim in moving the holes to the bottom was to improve deposition thickness at the base and thus the deposition uniformity along the cell depth. In addition, the following three graphite mandrels were also loaded in the CVD reactor to investigate replication on SiC. (i) A 7.5-cm-dia convex shaped (radius of curvature = 18.2-cm) mandrel made of T-6 ultra-high density graphite (porosity 9%, grain size = 2 μm, coefficient of thermal expansion = 6.5 x 10^{-6} c^{-1}). (ii) A curved mandrel with the above characteristics but made of ATJ graphite. (iii) A flat 7.5-cm x 7.5-cm mandrel made of T-6 ultra-high density graphite. All the mandrels were first polished, then coated with dryglide to make the surfaces impermeable and finally polished manually.

Examination of the SiC deposit revealed that the SiC has completely enclosed the graphite core of the LWS. A few cracks were observed on the SiC plate upon which the LWS was bonded. These cracks were on the outside and did not go through the LWS. The deposit thickness at the bottom of the inner triangular cells was measured to be about 1.0-mm (0.040-in) which is much larger than measured in the previous deposition. This shows that having holes at the bottom of the walls in the LWS increases the deposition thickness uniformity along the cell depth. Further, the LWS adhered quite well

to the SiC plate indicating that the effect of a reduction in the SiC-SiC bonding area due to the presence of the bottom holes was minimal.

Concerning the SiC replication on graphite, the curved graphite samples separated readily from the SiC deposit; however, the flat graphite sample did not. This was because the graphite mandrel was quite thin and a firm grip necessary for separation could not be provided to the graphite mandrel. The SiC deposit replicated on T-6 graphite was shiny and smooth while that on the ATJ graphite was rough. This is expected since the grain size and porosity of T-6 graphite is much smaller than that of the ATJ graphite. Further, the radius of curvature of the T-6 graphite mandrel and the replicated SiC were measured to be 18.03-cm and 17.83-cm, respectively. Since SiC is fabricated at 1300 C, this difference appears to be due to a thermal expansion mismatch between graphite and SiC. Figure 8 shows a picture of the convex shaped graphite mandrel and the replicated SiC plate.

In deposition #9081-SiC-8, a few more curved and flat samples of graphite were used to obtain replication in SiC. The aim of this deposition was essentially to confirm that SiC deposit could be separated from graphite readily and reproducibly. Two curved and one flat sample of graphite were used and all three samples separated readily from the SiC deposit. Measurement of SiC plate thickness at several places indicated that there is up to 50% variation in the deposit thickness. Since it is quite expensive and time-consuming to generate a curved surface in SiC, improved thickness uniformity will be required. This could be achieved by depositing SiC on a graphite mandrel placed perpendicular to the flow.

In Run Nos. 9081-SiC-9 through 9081-SiC-15, the SiC deposition experiments were performed to fabricate 7.5-cm-dia SiC mirror substrates which could then be used for cladding Si. In deposition #9081-SiC-9, the aim was to try a new deposition set-up, which could provide increased deposition area in a plane perpendicular to the flow. The deposition set-up that we used up to this point consisted of four mandrel plates which were placed parallel to the flow and one baffle which was placed perpendicular to the flow. This set-up was redesigned and four mandrel plates were replaced with two baffles. Thus, a total of three baffles in series were used. In this arrangement, the deposition

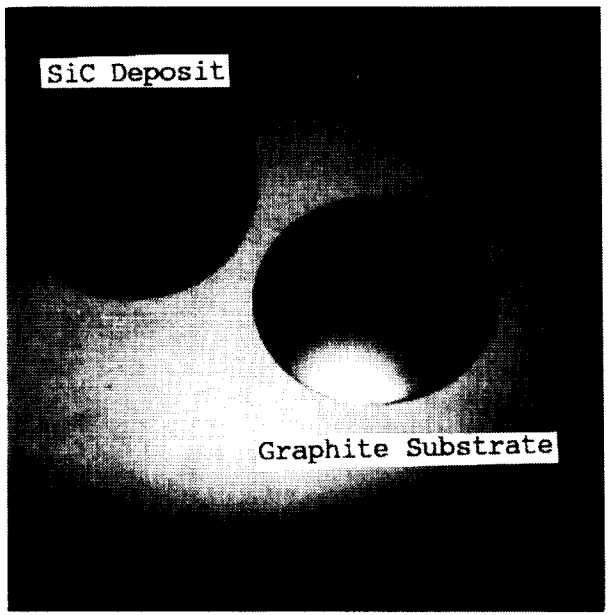

Figure 8. SiC replication of 7.5-cm diameter convex shaped graphite mandrel. Radius of curvature = 18.24 cm.

thickness could vary from baffle to baffle but within a baffle, the potential existed to obtain a fairly uniform deposit. Three graphite substrates -- a polished flat, a convex-shaped piece with radius of curvature about 18.29-cm and one graphite lightweight core bonded to a flat SiC plate -- were mounted on these three baffles. The flow rates of H_2 and MTS (methyltrichlorosilane) were kept the same as in Run #8, but that of argon was increased from 2.0 sℓpm to 4.0 sℓpm. The increased argon flow increased the average flow velocity and reduced the partial pressure of reagents thus providing a more uniform deposit from baffle to baffle. This run was terminated after 25 hours.

On examination, a very uniform coating of SiC was obtained on the first two baffles but there was carbon rich SiC deposit on the third baffle. A carbon rich deposit is formed when the reagents are H_2 deficient. Therefore, in deposition #9081-SiC-10, it was decided to increase the flow rate of H_2 to 5.0 sℓpm but reduce the flow rate of argon to 2.0 sℓpm. A two baffle configuration was used and graphite lightweight cores were bonded to the SiC faceplate (flat and curved) fabricated in the previous deposition. These SiC faceplates were not separated from the graphite mandrels. This deposition was terminated after 10 hours.

When the reactor was opened, it was found that the graphite LWS did not bond well to the curved faceplate and fell down early in the deposition. This structure was also coated with SiC. The other structure remained bonded to the flat faceplate and was coated with SiC.

To recover the SiC lightweight mirror substrate, it was necessary to separate it from the graphite mandrel. However, SiC deposited on both sides of the baffle plate. Since SiC is a very hard and strong material, methods used for recovering ZnSe and ZnS materials did not apply to SiC. Efforts were made to remove the excess SiC deposit by sawing off close to the periphery of the mirror faceplate. Once the excess SiC was cut off, the faceplate was separated from the graphite mandrel and a lightweight SiC mirror substrate ready to be coated with Si was obtained. However, this procedure was quite cumbersome. Consequently, it will be desirable to prevent SiC growth on the edges and back of the baffle plate such that the above procedure is not required.

In deposition #9081-SiC-11, the deposition area perpendicular to the flow was

further increased by using four baffles in series. Further, the grade of graphite used to fabricate mandrels was altered from CS grade to H-490. This latter grade has only 9% porosity as opposed to 17% for the CS grade, and provides a better finish and figure for replication. Two curved and one flat mandrel were mounted on three baffles while a graphite lightweight core was bonded to a SiC plate, which was in turn bonded to the baffle plate. The four baffle configuration increased the deposition length along the flow. Correspondingly the flow rate of H_2 was increased to 6 sℓpm while those of argon and MTS were increased to 2.5 sℓpm and 1.0 sℓpm, respectively. To facilitate recovery of the SiC mirror substrate, the baffle plate was designed with a channel along the edge as shown in Figure 9(a). Since the aspect ratio (depth to width) of this channel was large, a small SiC deposit at the bottom of this channel was anticipated. Thus, a break in the continuity of the SiC deposit was affected which facilitated in the recovery of the SiC mirror substrate. The deposition was terminated after 22 hours.

On examination, good SiC coating was obtained on all substrates. Although the thickness of the deposit varied from baffle to baffle, no carbon-rich-SiC (blackish color as opposed to dark grey) deposit was obtained. The SiC deposit was thicker on the second baffle from the injector. An injector mark in the form of discoloration and increased growth was seen on the first baffle. This is because the first baffle is very close to the injector. Close examination of the replicated flat and curved SiC deposits indicated cracks in the former. The crack marks were also visible on the flat graphite mandrel indicating that perthaps these cracks were generated at the beginning of the deposition.

The channel concept designed to break the continuity of the SiC deposit did not work satisfactorily. Although the thickness of the SiC deposit at the bottom of the channel was relatively small, the excess SiC could not be removed by manual operations. A sawing technique was used, but cracks developed in the SiC faceplate.

Deposition #9081-SiC-12 was performed to attach SiC LWS to the curved faceplates fabricated in deposition #9081-SiC-11 and also to replicate two new SiC flat plates. The deposition conditions were kept the same as in the previous run. To improve deposition on the first baffle, a manifold plate was designed and placed on top of the injector. This manifold plate had eight holes, each 1.25-cm-dia, located in an annular

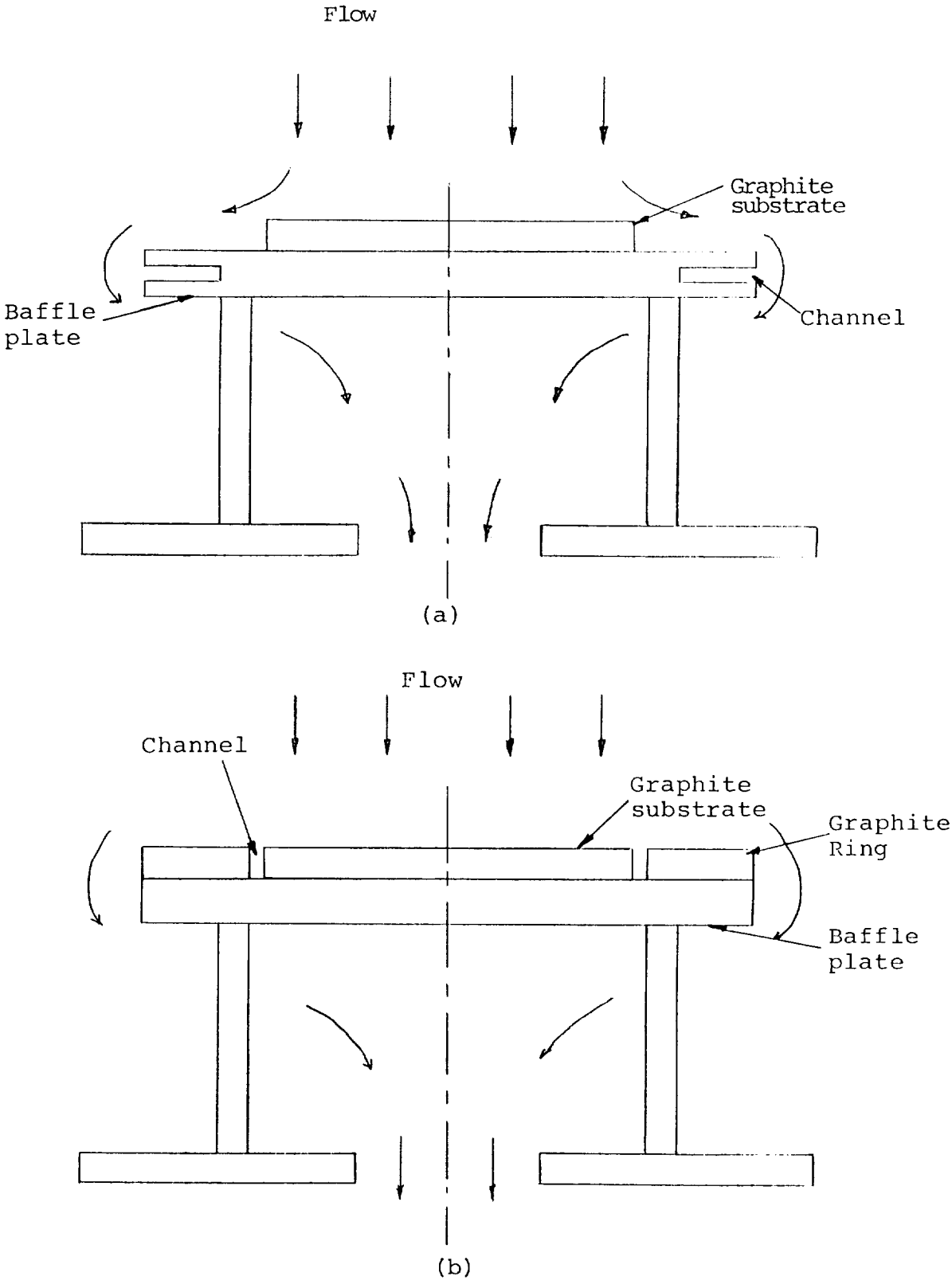

Figure 9. Channel concept to break continuity of SiC deposit (a) channel in the edge of the baffle plate, (b) channel around the graphite substrate

region of radii 1.25-cm (0.5-in) - 7.5-cm (3.0-in) from the injector. The flow impinged in the center of the manifold and then got distributed to these holes. This arrangement increased the effective diameter of the jet, thereby reducing its velocity and thus the direct impingement on the first baffle. Another channel concept was used to break the continuity of the SiC deposit. A channel was created on the baffle plate by bonding a graphite ring around the substrate plate as shown in Figure 9(b).

Examination of SiC deposit indicated that SiC had coated all four substrates quite well. No clogging of the holes in the manifold plate was observed. There was no indication of any injector mark on the substrate mounted on the first baffle. Thus the manifold plate worked quite well. Another benefit of this manifold plate was that there was a negligible erosion of the injector tip. Thus, the same injector could be used for many depositions. No cracks in SiC deposit on flat substrates was observed. The channel concept again did not work in a satisfactory manner. The SiC deposit was quite thick at the bottom of the channel. The excess SiC was again removed by sawing. One mirror substrate cracked due to a technician's carelessness, during the sawing operation while the other came out quite well.

In Deposition #9081-SiC-13, a gas flow technique was tried to prevent excess SiC growth. Two rows of overlapping holes were drilled around the substrate and argon was passed to shroud unimportant areas on the baffle plate. The total flow rate of reagents was the same as in the previous deposition. However, MTS flow was pulsed at a rate of two pulses per minute. The pulse width was 20 seconds. Pulsing of the flow reduced the grain size of the material and thus improved its properties. Two graphite lightweight cores were bonded to SiC flat faceplates, and two new graphite curved plates were used as substrates. The deposition was terminated after 27 hours.

On examination, it was found that SiC had coated all substrates quite well. The deposit appeared very similar to the other SiC deposits even though the flow was pulsed. The gas flow technique did not work as per our design. The argon flow was not distributed uniformly to all the holes but was concentrated in a few holes. To make the argon flow uniform, it was nececssary to increase the reservoir pressure and create a large reservoir of gas underneath the holes. However, since graphite is a porous

material, it cannot sustain a large pressure differential, especially at high temperatures. Further, we observed SiC deposit on and around all holes including those through which argon flowed. This showed that the flow rate of argon was not sufficient. However, a considerable increase in argon flow rate will reduce the partial pressure of the reagents and thus the deposition rate, increase the flow velocity, and affect the temperature distribution in the reactor. For these reasons, the gas flow technique to prevent SiC deposition on unimportant areas was not pursued further.

To recover the SiC lightweight and flat mirror substrates from the baffle plate, they were sent to Bomas Machine Specialties Inc., Somerville, MA. There, the excess SiC deposit was grounded off under controlled conditions. However, one LWS cracked when a technician accidentally tapped the SiC LWS with a hammer. The other flat mirror substrate released readily from the graphite mandrel.

In deposition run #9081-SiC-14, two flat and two curved graphite mandrels were mounted on the baffles. To prevent growth of SiC on the backside of the graphite mandrel, a hollow body was fabricated from grafoil and cemented on the backside as shown in Figure 10. Since grafoil is flexible, any stress that is generated in the material during cooldown will deform the grafoil body without significantly stressing the material. As can be seen from Figure 10, this body completely covered the backside of the mandrel, thus preventing deposition on the backside of the mandrel. Examination of the SiC deposit showed a thick layer of SiC on the front side of all the four mandrels. This layer was used to fabricate the faceplate of the mirror. On the backside, the deposition occurred on the grafoil body. The grafoil body along with its SiC deposit was removed readily by piercing it and breaking it into small pieces with a pair of pliers. No cracking of the SiC deposit on the front side of the mandrel was observed. These results showed the potential of this technique to selectively deposit SiC.

Four graphite LWS were then bonded to the four SiC deposits on the front side of the mandrel and new grafoil bodies were cemented on the backside. These mandrels were mounted in the reactor and another SiC deposition (Run #9081-SiC-15) was performed for 26 hours by pulsing the flows. The selective deposition scheme again worked quite well. The SiC mirror blanks were separated from graphite mandrels without

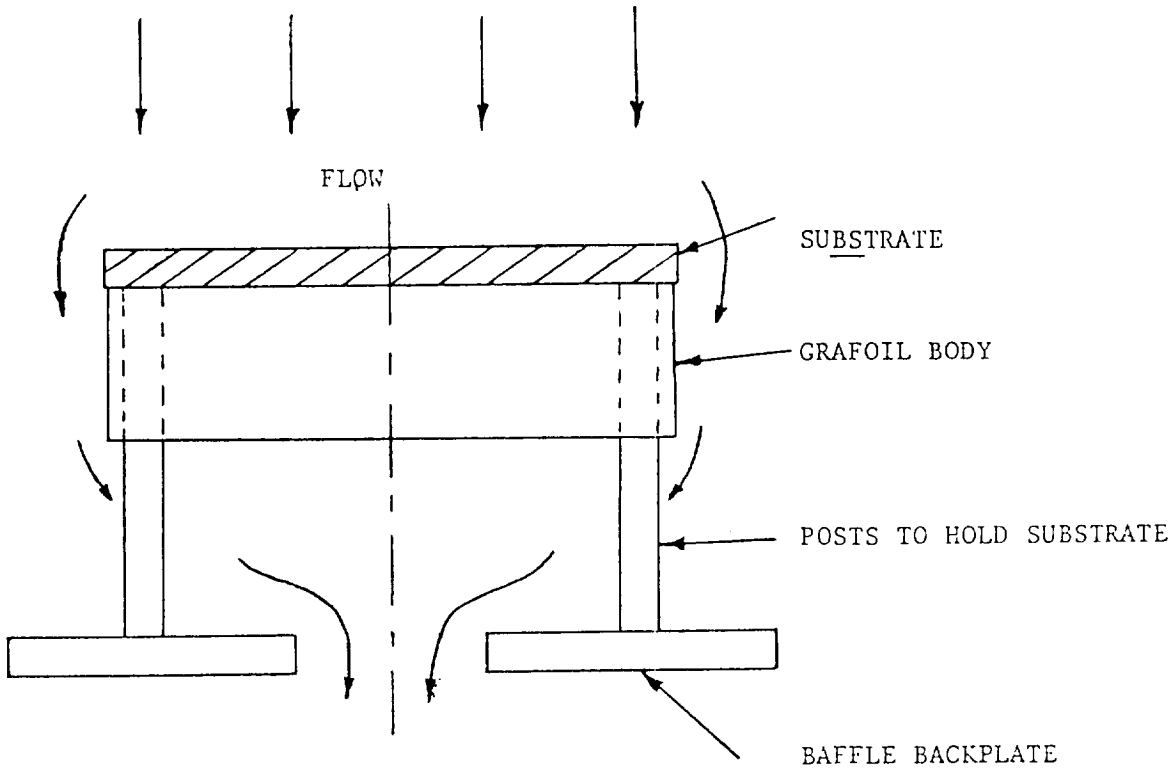

Figure 10. A schematic of the grafoil technique used to prevent growth on the backside of a substrate in an impinging vapor deposition system.

performing elaborate machining of the edges. This deposition resulted in fabricating three lightweight SiC mirror blanks ready for Si cladding. The fourth mirror substrate, which was placed farthest from the injector had carbon contamination in the LWS.

In summary, small scale SiC deposition experiments have been successfully completed. From these experiments, the following conclusions can be drawn: (i) SiC can be replicated on curved and flat graphite mandrels, and near-net-shape mirror faceplates can be fabricated. (ii) CVD-SiC bonds quite well to itself. (iii) A SiC lightweight structure can be fabricated by depositing SiC on a lightweight graphite core. The SiC deposit encloses the graphite core completely and no cracks are developed. (iv) The SiC deposit uniformity along the depth of the LWS can be improved by providing holes near the bottom of the LWS. (v) The SiC faceplate can be readily separated from the graphite mandrel by coating the graphite mandrel with a release agent and preventing the SiC growth on the backside of the faceplate. The growth on the backside of the faceplate can be prevented by using the grafoil technique which masks the relevant areas. (vi) Good quality Si-SiC two layer material with strong Si-SiC bond could not be fabricated by depositing CVD-SiC on Si substrates.

3.4 Small-Scale Si/SiC Lightweight Mirror Models

A total of four Si/SiC lightweight mirror substrates were produced in this program using the procedure described in Sections 3.2 and 3.3. Two mirrors were curved with a nominal radius of curvature of about 17.8-cm and the other two were flat. The diameter of the mirrors was 7.5-cm and the backstructure had one outer hexagonal cell and six inner triangular cells. Each side of the hexagon was 3.18-cm long and 2.54-cm wide. Two flow holes were provided on each rib of the backstructure. These holes had a diameter of .625-cm before SiC is deposited, and their center was located 0.625-cm from the faceplate. The thickness of the Si clad SiC faceplate varied in the range 1.5-mm to 5.0-mm. The thickness of the Si cladding was about 0.3-0.6-mm.

Two curved and one flat Si/SiC mirror substrate were sent to Opticraft, Woburn, MA, for polishing of the Si surface. The remaining flat Si/SiC mirror was polished at CVD Incorporated. The aim of the polishing effort at Opticraft was to fabricate a $\lambda/5$ figure on

all the mirrors. The two curved mirrors were polished to radius of curvatures of 17.95-cm (7.067-in) and 17.74-cm (6.986-in), a difference of 1.15%. This variation could have occurred due to any of the following reasons: (i) the curved graphite mandrels were not fabricated to a tight tolerance, (ii) the dryglide coating which was applied by a spraying technique might not be uniform, and (iii) the thickness of thte Si cladding was quite different for the two mirrors. However, it is encouraging to see that all these potential sources of variations made a small impact on the final radius of curvature of the mirror.

Opticraft succeeded in obtaining a figure of $\lambda/5$ without spending much polishing time. Thus, the goal of this program to obtain a figure of $\lambda/5$ was demonstrated on these scaled models. Further, the polishing of these mirrors did not produce any delamination, cracking, flaking or other defects in the Si cladding, indicating that over a 7.5-cm-dia surface, Si adherence to SiC is assured.

In addition to the above four mirrors, two more models of flat lightweight mirrors were fabricated in this program. One mirror substrate was completely made of CVD-Si while the other mirror had a SiC faceplate and a Si backstructure. The former mirror was polished at CVD Incorporated, while no polishing operation was performed on the latter. Successful fabrication of these mirrors demonstrated the following aspects of CVD technology: (i) CVD-Si can be replicated to a near-net-shape on graphite. (ii) CVD-Si can be bonded to itself. (iii) A Si LWS can be fabricated similar to a SiC LWS. (iv) CVD-Si can be bonded to SiC by depositing CVD-Si on a SiC substrate.

Figures 11 and 12 show the pictures of the polished Si/SiC lightweight mirrors fabricated in this program. Figure 11 shows two flat mirrors. By using the reflection of one mirror through the other, a typical front, back and side of a flat Si/SiC mirror are shown. The flow holes in the walls of the mirror are also clearly visible. Figure 12 shows five (5) Si/SiC mirrors. The two mirrors on the right are curved as can be seen from the reflection. The front mirror on the left is made of Si.

With the successful fabrication of Si/SiC mirrors, a detailed procedure for the fabrication of these mirrors via CVD was developed. This procedure is described as follows: First a mandrel is fabricated from H-490 grade graphite. On one side of this mandrel a surface is fabricated which is the "negative" of the actual mirror figure desired.

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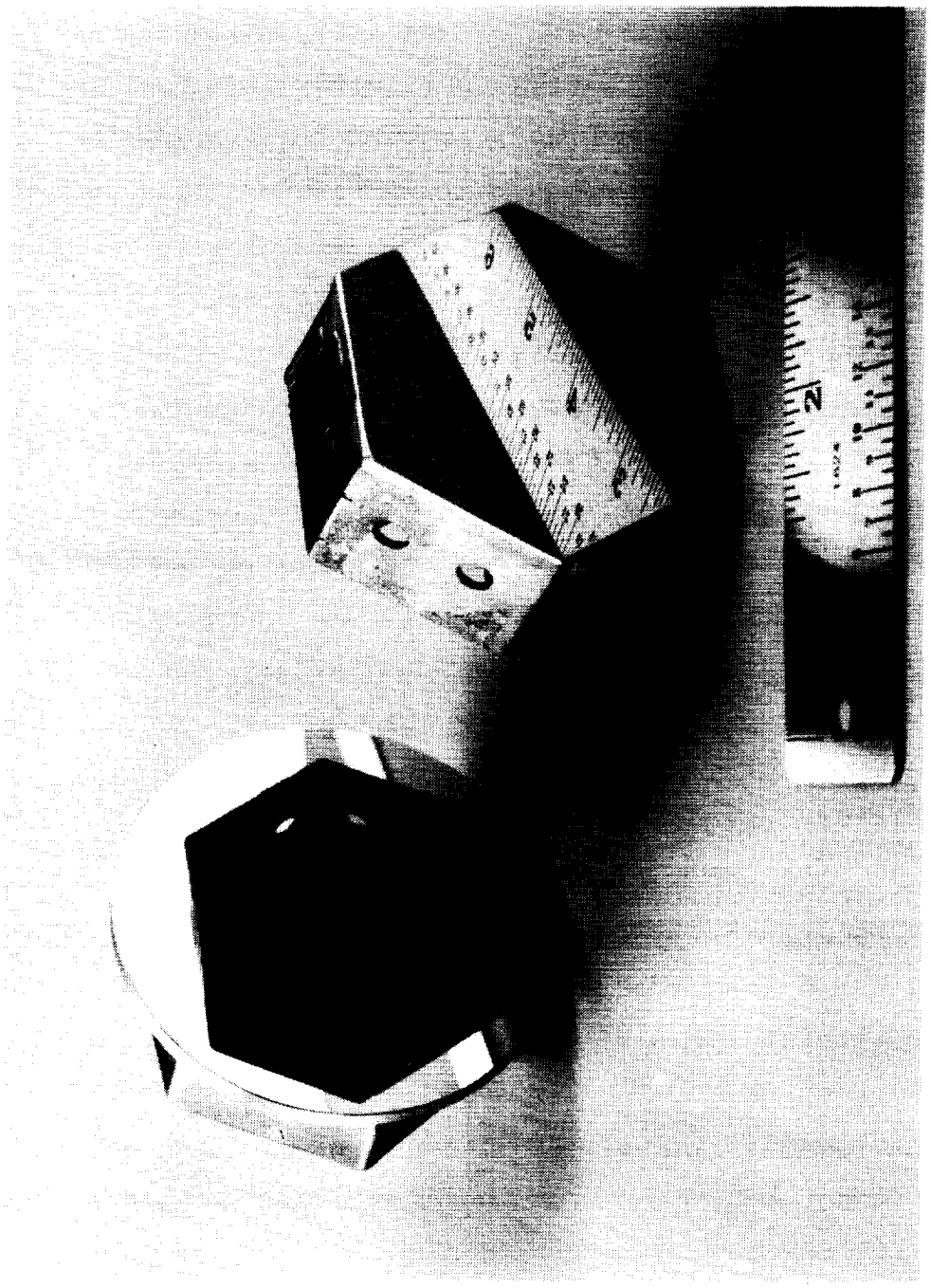

Figure 11. Pictures of two flat Si/SiC mirrors fabricated via CVD

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Figure 12. Silicon and Si/SiC lightweight mirrors. Two mirrors on the right are curved. The front mirror on the left is made of Si.

This mandrel is polished, coated with a mold release coating, such as a suspension of colloidal graphite, and mounted in a CVD reactor on a baffle. A grafoil body is used to mask the sides and the backside of the mandrel. In this reactor, the mandrel is heated to about 1300 C and a mixture of CH_3SiCl_3 and H_2 in the ratio 1:4 is introduced to deposit a predetermined thickness of SiC. This SiC deposit acts as a faceplate for the Si/SiC mirror. The mandrel along with the SiC faceplate is then removed from the CVD chamber, and the deposition side is cleaned and etched with KOH without removing either the faceplate or the grafoil body from the mandrel. Since the SiC faceplate is usually thin relative to its surface area, the graphite mandrel provides proper support during this cleaning operation. A hexagonal cell, honeycomb core is then fabricated from graphite ribs of thickness about 0.5 mm and bonded to the backside of the faceplate. This graphite core has several small holes in the walls to further reduce the weight of the structure and also to disburse the reagents during CVD deposition. The assembly consisting of the graphite mandrel, the SiC faceplate, and the graphite lightweight core is then mounted in the CVD reactor and a layer of SiC is deposited onto the graphite core. This deposition totally encloses the graphite core and also reinforces the bonding of the lightweight structure to the SiC faceplate. Next, the grafoil body is removed and the mandrel is separated from the SiC faceplate yielding a replicated figure on the faceplate. Since Si is relatively easier to polish than SiC, a coating of Si is applied to the mirror faceplate. In order to apply the Si coating, the grafoil body is again used to mask the SiC backstructure. Next, the reactor is heated to about 1000 C and a mixture of $SiHCl_3$ and H_2 in the ratio of 1:5 is passed through the deposition area. After a sufficient thickness of Si is deposited, the Si/SiC mirror substrate is unloaded from the reactor. Finally, the near-net-shape mirror is optically fabricated to the desired figure and finish. The above procedure was used to fabricate Si/SiC mirrors in the production furnace in Tasks 2 and 3.

3.5 Task 1 - Summary and Conclusions

Small scale experiments were performed to deposit Si by the reaction of $SiHCl_3$ and H_2, and SiC by the reaction of CH_3SiCl_3 and H_2 in a CVD research reactor. A total

of 27 experiments were performed to establish the feasibility of fabricating lightweight Si/SiC mirrors via CVD. These experiments demonstrated the following: (i) The initial approach to fabricate Si/SiC mirrors in which Si faceplate is replicated on a suitable mandrel and the lightweight structure made of SiC is bonded to the backside of the faceplate is not feasible because (a) a high degree of replication could not be obtained in CVD Si, and (b) CVD-SiC did not adhere well to Si. (ii) An alternate approach to fabricate Si/SiC mirrors in which the faceplate is made of SiC cladded with Si and the lightweight structure is made of SiC is feasible. Important issues relevant for making this approach a success were resolved. In particular, the following results were demonstrated: (a) SiC replication on curved and flat graphite samples, (b) CVD-SiC bonding to itself, (c) fabrication of SiC LWS via CVD and (d) Si cladding on SiC.

A major accomplishment in this task was the successful fabrication of several 7.5-cm-dia models of flat and curved Si/SiC mirrors. A detailed fabrication procedure for these mirrors via CVD technology was developed. Two Si/SiC mirrors, one flat and the other curved were also delivered to NASA for evaluation. Thus, all the objectives of this task were successfully met.

4.0 TASKS 2 AND 3: SCALING AND DEMONSTRATION OF CVD MIRROR FABRICATION TECHNOLOGY

4.1 Introduction

The main objective of Task 2 was to scale the CVD technology of fabricating the lightweight Si/SiC mirrors from the research furnace to a small production furnace. This scaling task required the following: (i) modification of the production furnace, (ii) scaling of the SiC CVD process, (iii) scaling of the Si cladding process and (iv) scaling of graphite lightweight core and SiC LWS. The objective of Task 3 was to demonstrate the scaling of the CVD mirror fabrication technology by fabricating a 40-cm-dia lightweight mirror in the production furnace. This task required the following: (i) design of the 40-cm-dia mirror, (ii) replication of SiC faceplate on a graphite mandrel, (iii) fabrication and assembly of graphite lightweight core, (iv) SiC deposition to fabricate LWS and (v) Si cladding on SiC faceplate.

The scaling of the CVD-SiC process and the LWS was to be demonstrated in the AF-supported Large Mirror Substrate Program. Since that program was delayed, it was decided not to delay this program and therefore, to proceed with the scaling of the CVD-SiC process. To conserve the program resources, it was decided to perform scaling experiments on a 40-cm-dia mandrel such that if the initial SiC depositions prove successful, a SiC faceplate will be obtained for the 40-cm-dia mirror. Further, the mirror design and furnace modification tasks were started in parallel.

4.2 40-cm Diameter Mirror Design

Table 11 shows the NASA requirements for a 40-cm-dia mirror which is a deliverable in this program. This mirror will be spherically concave with a radius of curvature of 1.0-m. The mirror surface will be "as deposited" silicon. No grinding or polishing of this surface was to be performed under this program.

Under the Large Mirror Substrate Program a 0.5-m-dia lightweight SiC mirror was designed. Table 12 lists important design data for this mirror. This design assumed a polishing load of 1 psi, a peak to valley intercell sag of 0.025 μm, a peak to valley self-

TABLE 11: REQUIREMENTS FOR 40-CM DIA. Si/SiC LIDAR MIRROR

MIRROR SUBSTRATE	
FACE PLATE:	Si CLADDED SiC
BACK UP STRUCTURE:	LIGHT-WEIGHTED SiC
MIRROR DIAMETER:	40 CM
AREAL MASS:	25 KGM^{-2}
MIRROR SURFACE:	SPHERICALLY CONCAVE
RADIUS OF CURVATURE:	1.0 M
SURFACE QUALITY:	AS DEPOSITED Si SURFACE
ENVIRONMENT	
TEMPERATURE:	15° ± 5° C
VACUUM ENVIRONMENT	
STS LAUNCH LOAD STRUCTURAL SURVIVABILITY	

TABLE 12: DETAILS OF 0.5 M DIA. MIRROR DESIGNED BY UTOS FOR THE LARGE MIRROR SUBSTRATE PROGRAM

SiC FACEPLATE	INCH	CM
THICKNESS	0.11	0.28
SiC LIGHT-WEIGHTED STRUCTURE		
WALL THICKNESS	0.064	0.163
CELL HEIGHT	1.60	4.06
CELL LENGTH	2.46	6.25
NO. OF EQUILATERAL TRIANGULAR CELLS	96	96
FLOW HOLE DIAMETER	0.328	0.833
NO. OF FLOW HOLES IN EACH CELL WALL	2	2
SiC MIRROR		
DIAMETER	20.80	52.83
CLEAR APERTURE	20.56	52.22
MIRROR SURFACE	SPHERICALLY CONCAVE	
RADIUS OF CURVATURE	314.57	799
WEIGHT	20 Kgm^{-2}	20 Kgm^{-2}
TOTAL MIRROR THICKNESS	1.71	4.34

weight gravity distortion between supports (25-cm spacing) of 0.025 μm and a minimum natural frequency of 25 Hz. These requirements were quite stringent and were an "overkill" for the NASA mirror. However, important features of this design could be used to design the 40-cm-dia LIDAR mirror. Therefore, to obtain a design for the 40-cm-dia Si/SiC mirror, the important dimensions of the 50-cm-dia mirror were scaled by a factor of 0.8. This scaling was performed keeping the following parameters the same: (1) cell aspect ratio, defined as the ratio of cell depth to the diameter of the inscribed circle, = 1.3, (2) number of lightweight cells = 96 and (3) thickness of graphite core = 0.5 mm. Combining constraints (1) and (2) reduced the cell length and height, each by a factor of 0.8. Keeping the cell wall thickness the same and reducing the diameter of the flow holes by a factor of 0.8 resulted in reducing the weight of the SiC LWS by a factor of 0.8 x 0.8 = 0.64. Further, corresponding to a reduction in mirror diameter from 50-cm to 40-cm, the thickness of the SiC faceplate by a factor of 0.8. The Si cladding thickness for the faceplate was selected to be 1.0 mm. This cladding thickness corresponded to an error of ± 2% in the radius of curvature and was considered sufficient to insure that, the SiC surface will not be exposed during polishing.

The design weight of the 50-cm-dia SiC mirror was 3.927 Kg which corresponded to a weight specification of 20 Kgm^{-2}. Of this, the weight of the SiC faceplate was 1.755 Kg and that of the SiC LWS was 2.172 Kg. Using the above scaling for the 40-cm-dia Si/SiC mirror, the weight of the SiC faceplate and the LWS structure were calculated to be about 1.404 Kg and 1.390 Kg, respectively. The weight of the Si cladding was 0.297 Kg. Thus, the total design weight of the 40-cm-dia Si/SiC mirror was 3.091 Kg. This weight corresponded to a weight specification of 24.60 Kgm^{-2}, which was within the goals of the NASA program. It is emphasized that if weight optimization is performed based upon NASA mirror specifications, there is a further potential to reduce the areal mass. Table 13 and Figure 13 lists/shows the important features of the 40-cm-dia Si/SiC LIDAR mirror design.

4.3 CVD Reactor Modifications

An existing pilot plant size CVD reactor (RF #4) was selected for performing the

TABLE 13: 40-CM DIAMETER Si/SiC MIRROR DESIGN FEATURES

	INCH	CM
Si CLADDED SiC FACEPLATE		
Si CLADDING THICKNESS	0.040	0.10
SiC FACEPLATE THICKNESS	0.088	0.22
FACEPLATE TOTAL THICKNESS	0.128	0.32
SiC LIGHT-WEIGHTED STRUCTURE		
WALL THICKNESS	0.064	0.163
CELL HEIGHT	1.28	3.25
CELL LENGTH	1.97	5.00
FLOW HOLE DIAMETER	0.275	0.70
HOLE CENTER DISTANCE FROM EDGE	0.40	1.02
NO OF EQUILATERAL TRIANGULAR CELLS	96	96
CELL ASPECT RATIO	1.3	1.3
Si/SiC MIRROR		
MANDREL DIAMETER	17	43.20
RADIUS OF CURVATURE	39.37	100
TOTAL MIRROR THICKNESS	1.408	3.58
CENTER DEPTH	0.93	2.36

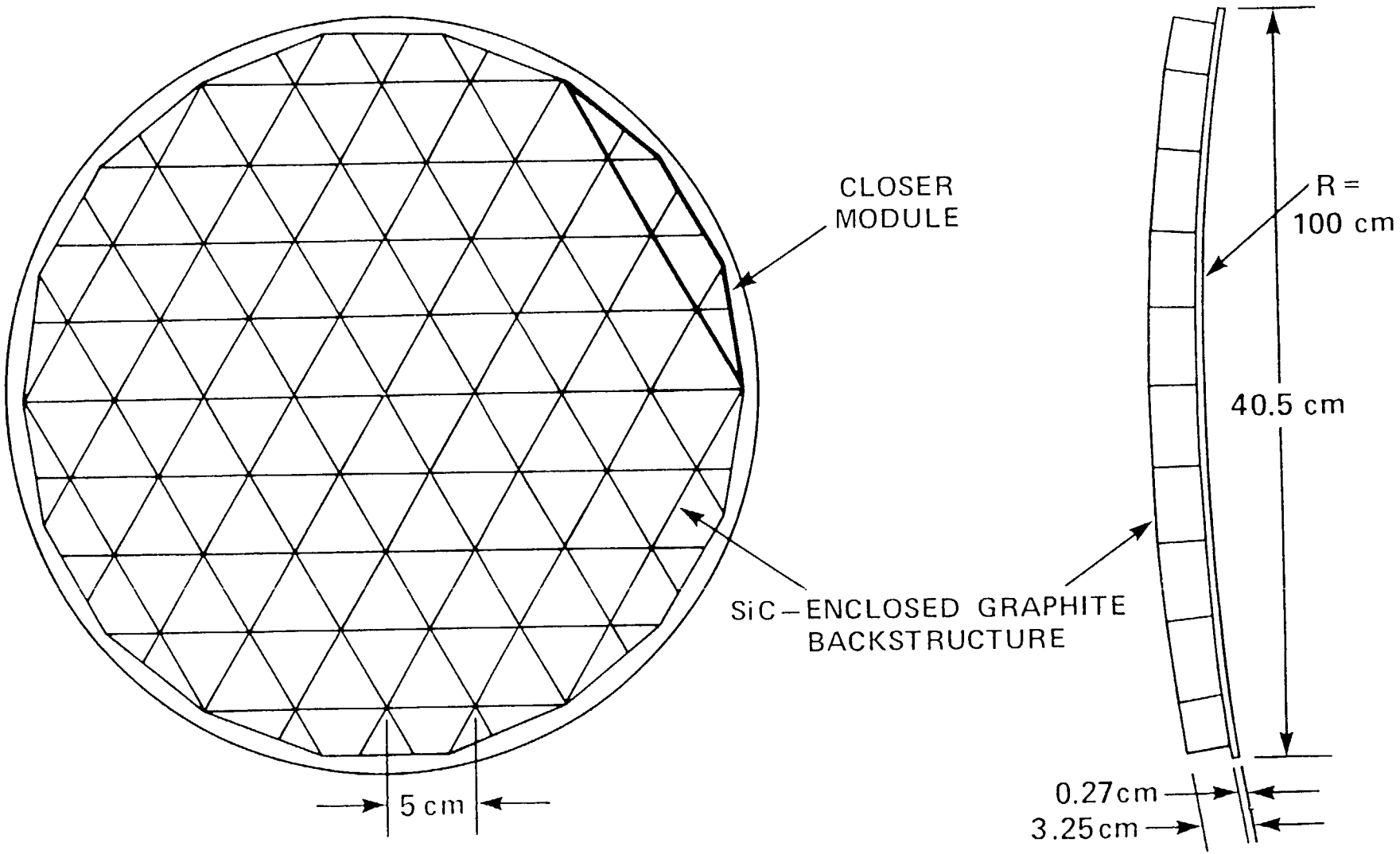

Figure 13. Details of a 40-cm diameter Si/SiC mirror design

scaling analysis and for fabricating the 40-cm-dia Si/SiC mirror. This facility was a vertical reactor with a working diameter of about 50-cm and length of 75-cm. The outer shell of this reactor was constructed from stainless steel and was water-cooled. This reactor was previously used to deposit Si and SiC and had a maximum temperature capability of 1500 C. The flow in this reactor was vertically upward and the deposition occurred on the walls of the chamber which were parallel to the flow direction. However, to obtain a uniform deposit required for our applications, the deposition should be performed in an impinging flow configuration where the deposition surface is perpendicular to the flow. Therefore, the modifications consisted of the design, fabrication and assembly of a new deposition set-up for this reactor.

The design of an impinging flow deposition setup was completed by the Engineering Department at CVD Incorporated. The salient features of this deposition setup were the following: (i) Up to three mandrels in series could be used at one time. The use of several mandrels increased the probability of success since it took three CVD depositions to completely fabricate a Si/SiC mirror blank. Further, different mandrels could be used to investigate different issues in the same deposition experiment, thereby reducing the number of experiments and, hence, the cost. The small scale experiments performed with several mandrels in series indicated that the deposition thickness varies from mandrel to mandrel. Typically, the deposition thickness peaked in the second mandrel from the injector. However, the variation in deposition thickness from mandrel to mandrel could be minimized by adjusting the temperature profile, the reagent flow rates, and the area ratio of the exhaust and the inlet ports. (ii) Four injectors were provided to obtain a more uniform flow pattern in the reactor. These injectors were fed from two CH_3SiCl_3 tanks, each tank feeding the two adjacent injectors. (iii) A manifold was incorporated to randomize the flow of the injectors, thereby providing a more uniform flow of reagents over large areas. The advantage of using this manifold was that the distance between the first mandrel and the injectors could be made small. If this manifold was not used, abnormal growth was obtained at those places where the injector directly impinged on the mandrel. (iv) The graphite heating elements were shortened to minimize the heating of the injector, thereby preventing their clogging.

Figure 14 shows a schematic diagram of the modified setup. The deposition arrangement was enclosed in a graphite tube of dimensions: 49.53-cm (19.5-in) i.d., 0.625-cm (0.25-in) thick, and 106.7-cm (42-in) long. The mandrel was 43.18-cm (17-in) in diameter and was made of H-490 grade of graphite. In between the mandrel and the tube, there were three graphite posts, each 1.91-cm (0.75-in) in diameter, and placed 120 degrees apart to support the mandrel assembly. The graphite tube isolated the deposition area from the graphite elements and the carbon felt insulation, thereby preventing deposition on them. The clearance between the mandrel and the graphite post was small, but it could not be increased due to the fixed diameter of the furnace.

The assembly of the deposition setup was performed by first assembling the mandrels on the furnace end cover, followed by loading the assembly into the furnace, and, finally, lowering the graphite tube from the top using a hoist. Since the clearance between the mandrels and the graphite elements was small, considerable difficulty was encountered in this loading operation.

4.4 SiC Depositions

A total of eleven depositions were performed in the production furnace to deposit SiC. The details of these depositions are given in Table 14. The first three depositions were performed in our production furnace RF #4 which was modified as described in Section 4.3, while the remaining depositions were performed in a new furnace, RF #5 which was especially designed for fabricating Si/SiC lightweight mirrors. In the first eight depositions, attempts were made to fabricate a 40-cm-dia mirror while in the remaining three depositions, a 25-cm-dia SiC lightweight mirror was fabricated.

In deposition #9081-L-1, a three mandrel setup was used as described in Section 4.3, and SiC was deposited at a mandrel temperature of 1313 C and a furnace pressure of 200 torr. The MTS was carried to the deposition area by bubbling argon through the MTS tank. The flow rate of argon through each MTS tank was 4 slpm which corresponded to an MTS flow rate of about 1.0 slpm through each of the four injectors. The flow rate of H_2 through each injector was 6 slpm. The deposition was terminated after 30 hours due to clogging of an injector. The furnace was cooled slowly at the rate

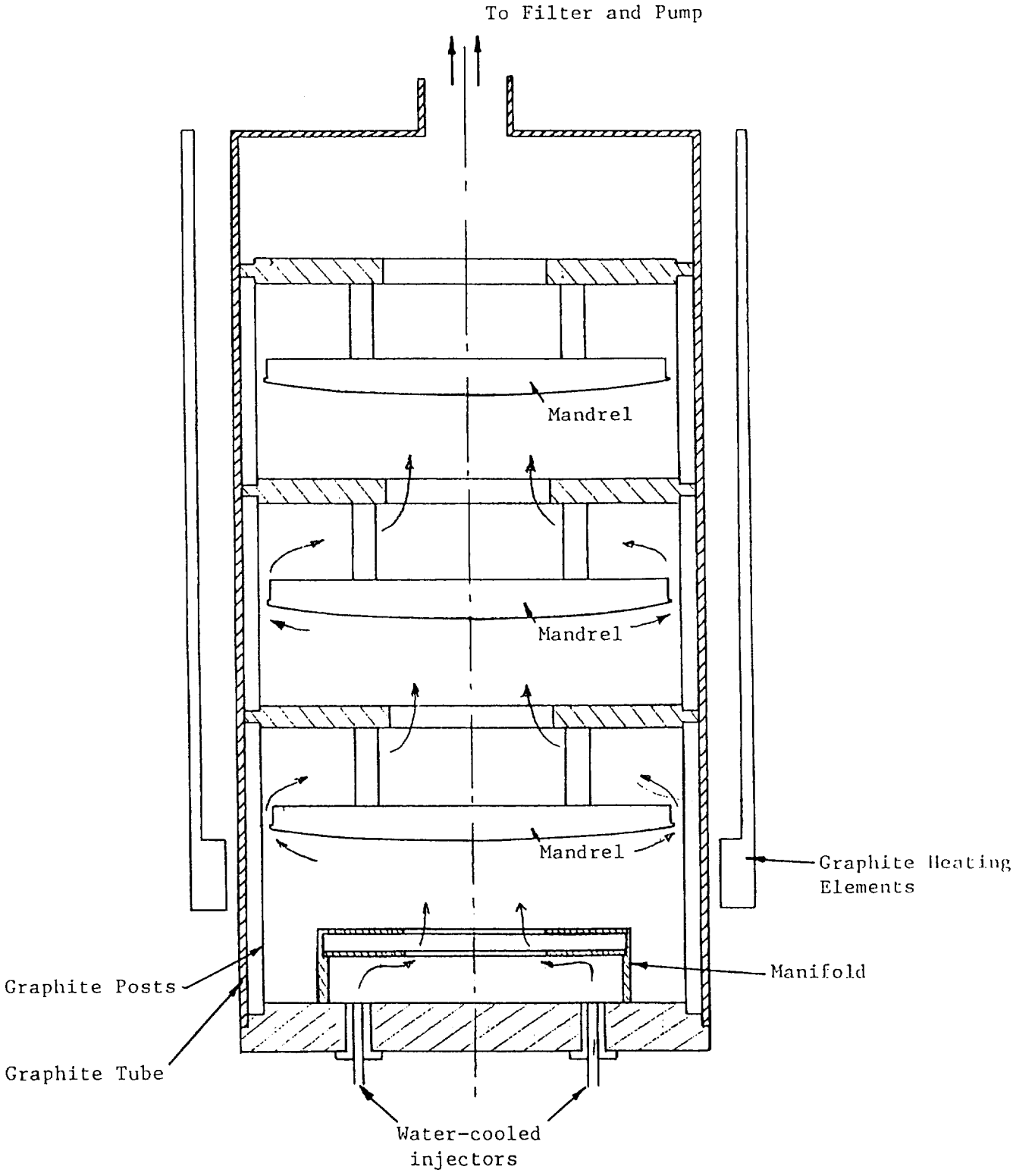

Figure 14. Impinging flow deposition setup used for the fabrication of 40-cm diameter Si/SiC LIDAR mirror in RF #4

Table 14. Details of Large Scale SiC Depositions

Run No.	Material Deposited	Deposition Thickness, mm	Run Time, hrs	Substrate Temp, C	Furnace Pressure, torr	Flow Rate, lpm H_2 CH_3SiCl_3 Ar	Remarks
9081-L-1 (RF #4)	SiC	2.0 - 3.0	28.0	1313	200	24.0 3.0 8.0	Injector clogged. Three mandrels in series used. Mandrel material: H-490 graphite. Material cracked. No carbon deposit.
9081-L-2 (RF #4)	None	-	5.0	1300	5.0	- - -	Bake-out run. No distortion in mandrel shape observed. Mandrel used: SiC-6 graphite.
9081-L-3 (RF #4)	SiC	2.0 - 3.0	33.5	1338	196	30.0 3.2 8.0	Material cracked on first two mandrels. Material on third mandrel thin but without any cracks.
9081-L-4 (RF #5)	SiC	2.0 - 4.0	30.0	1340	200	19.0 4.7 14.4	Four injectors used. Injector growth marks on SiC deposit. Thermocouples were defective. Actual deposition temp = 1450 C. Material did not crack.
9081-L-5 (RF #5)	SiC	2.0 - 4.0	29.0	1347	201	22.0 4.7 14.0	Four injectors used. Grafoil body used. Powdery and dendritic growth on the mandrel.
9081-L-6 (RF #5)	SiC	1.0 - 2.0	20.0	1320	200	41.0 6.9 83.5	Seven injectors used. SiC deposit enclosed the lightweight graphite core. The SiC LWS did not stick.
9081-L-7 (RF #5)	SiC	2.0 - 3.0	27.5	1320	201	28.0 5.56 56.6	Seven injectors and gas shroud used. Good quality SiC obtained, but the faceplate cracked. Gas shroud flow = 13 slpm
9081-L-8 (RF #5)	SiC	2.0 - 3.0	29.0	1335	200	28.0 5.48 56.6	Seven injectors and gas shroud used. Good quality SiC obtained, but the faceplate cracked. Gas shroud flow = 13 slpm.
9081-L-9 (RF #5)	SiC	3.0 - 5.0	61.0	1345	200	28.0 5.2 56.6	25-cm-dia mandrel used. SiC faceplate was fabricated and separated from mandrel.
9081-L-10 (RF #5)	SiC	1.0 - 2.0	25.0	1345	200	28.0 6.1 63.5	SiC LWS was fabricated on 25-cm-dia faceplate. Nonuniform SiC deposit obtained.
9081-L-11 (RF #5)	SiC	0.5 - 1.0	12.0	1345	201	31.5 5.04 63.0	Another SiC deposit on LWS performed. Uniform SiC deposit obtained. SiC mirror substrate fabricated and delivered to NASA.

of 50 c/hour to minimize stresses due to thermal expansion mismatch between the graphite and the SiC. After the furnace temperature reached ambient temperature, it was opened. Attempts were made to lift the graphite tube with a hoist, but it did not succeed because the tube was connected to the deposition setup through SiC deposit. With much difficulty, the tube was unloaded intact in one piece.

Examination of the mandrel area indicated that the SiC material was deposited on all three mandrels. There was no sign of a carbon deposit. This was a very encouraging development and essentially showed that a three mandrel setup can potentially work. However, the SiC material on all three mandrels was cracked. In fact, the SiC material deposited on the first mandrel had fallen onto the injector manifold. It was felt that this material must have fallen during unloading of the graphite tube. Therefore, an improved loading/unloading procedure for the graphite tube was developed.

Considerable nonuniformity in material thickness was observed in the SiC deposit. This thickness variation was not symmetrical about the central axis. The deposition thickness was quite large along one side of the mandrel on all three mandrels. Reagent depletion was ruled out as a cause of this variation. The data indicated that considerable flow of reagents took place from two adjacent injectors and very little flow came from the other two. A detailed analysis indicated that since two MTS tanks were feeding those injectors, all the reagents were essentially flowing from only two injectors. Thus, a better method was required to divide the flow from one MTS tank into two equal parts.

Attempts were made to determine the origin of cracks in the SiC material. First, it was observed that the cracks in the SiC deposit ran from the front of the mandrel to the side and then to the grafoil body on the back of the mandrel. This grafoil body was used to prevent SiC deposition on the backside of the mandrel. Apparently, the crack energy was so large that the crack propagated through three 90-degree bends without being deflected. It was also observed that the SiC material was stressed in tension which is due to a thermal expansion mismatch between the graphite and the SiC. The H-490 grade of graphite used to fabricate the mandrel has a thermal expansion coefficient (CTE) smaller than that of SiC. This grade of graphite worked quite well in small scale experiments, but, on a large scale, the tensile stresses became magnified and caused

cracking. It was concluded that the mandrel should be fabricated from graphite which has a CTE value equal to or larger than that of SiC.

A major modification of the deposition setup was performed to address the issues raised in the first large scale SiC deposition. This modification included the following: (i) To facilitate loading/unloading of the graphite tube, the tube was cut into four equal lengths and the mandrel assemblies were supported at the end of each tube as shown in Figure 15. To minimize deposition on the inside of the tube and to obtain complete isolation of the deposition area from the graphite heating elements, the inside and outside of the individual tube parts were lined with grafoil. In order to provide enough clearance for grafoil lining on the inside, the mandrel size was reduced by 2.5-cm to a 40-cm diameter. This mandrel is sufficient to yield a finished 40-cm-dia mirror. (ii) The SiC-6 grade of graphite marketed by TTA America was used as the mandrel material. This grade of graphite has thermal expansion coefficient values slightly larger than that of CVD-SiC. The SiC-6 graphite is available in large sizes, has low porosity and high density. (iii) A plenum of diameter 1.91-cm and length 30-cm was provided to divide the flow from one MTS line into two equal parts. (iv) The MTS injectors were extended by about 1.25-cm into the deposition area to prevent their clogging. (v) The size of the injector manifold was reduced to minimize depletion of reagents from the flow.

In Run 9081-L-2, a bakeout of the modified deposition setup was performed to determine if the thermal cycling has any effect on the shape of the mandrel. This was done on the recommendation of the graphite supplier, since SiC-6 graphite releases gaseous material at elevated temperatures. After the thermal cycling to 1300 C was completed, the mandrels were examined and no distortion in their shape was observed. Therefore, these mandrels were unloaded, a coating of amorphous carbon was applied, and the CVD chamber was made ready for a SiC deposition.

In Run 9081-L-3, a SiC deposition was performed to fabricate the SiC faceplate. The flow rates of H_2 and CH_3SiCl_3 were increased to 30 slpm and 3.2 slpm, respectively. The mandrel temperature was increased to 1338 C. The SiC deposition was terminated after 33.5 hours.

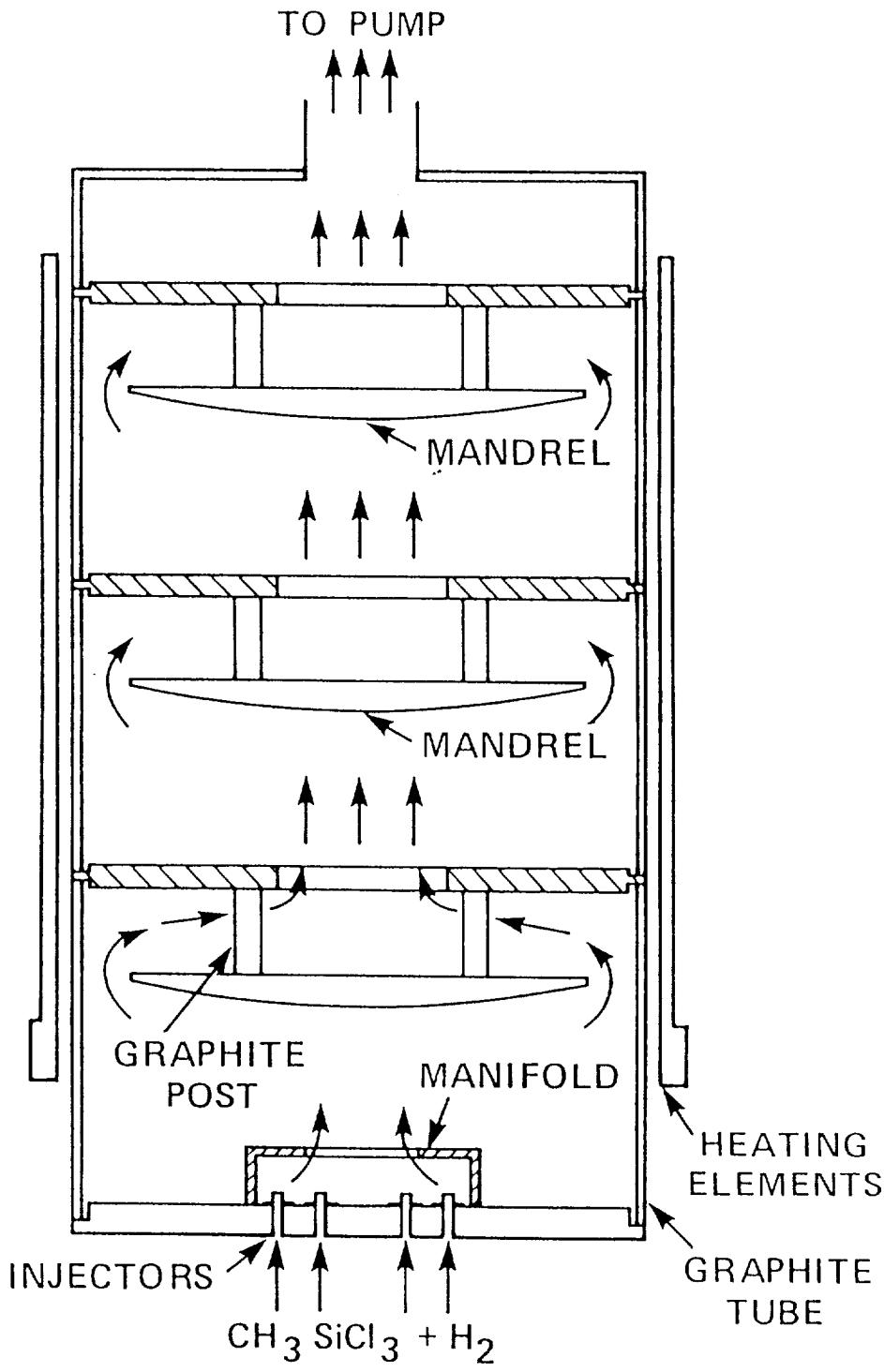

Figure 15. A schematic diagram of CVD deposition setup used in RF #4

On examination, it was found that the SiC material was deposited on all three mandrels. The deposited material on the first two mandrels cracked while the material on the third material was intact (Fig. 15). In fact, the material on the second mandrel had fallen down onto the baffle plate of the first mandrel. The thickness of the material on the first two mandrels was measured. On the first mandrel, the deposition thickness varied from about 3.7 mm in the center to about 2.3 mm on the edges. On the second mandrel, the thickness varied from 2.6 mm to 1.4 mm. However, on all the mandrels, the thickness profile was approximately circular indicating that the flow rate of reagents through the four injectors was about equal.

Figure 16 shows a picture of the SiC deposition on the third mandrel. No nodular growth was observed and the deposition surface was quite smooth. However, the SiC deposit appeared quite thin. On the convex side of this faceplate, a lightweight SiC structure could be fabricated. In order to do that, it was first necessary to assemble a graphite lightweight core which could then be bonded to the back of the SiC faceplate. Since in RF #4, the mandrel was mounted in an inverted position, there was concern that the lightweight graphite core could become loose and fall from the faceplate during heatup or SiC deposition. In order to prevent this situation, it was decided to use another SiC furnace (RF #5)*** in which the mandrel was mounted in an upright position (Fig. 17).

The graphite lightweight core was designed with graphite ribs about 0.5-mm thick. Three sets of this core were fabricated at an outside vendor facility. The graphite ribs were quite fragile and required careful handling during assembly. After going through a learning curve, one lightweight graphite core was assembled. This core, which was assembled on a curved surface, is shown in Figure 18. In this design, there is a large hexagonal cell which is filled with 96 triangular cells. Along the six sides of the hexagonal cell, closer modules were attached to maximize the coverage of the circular faceplate with lightweight cells (Figure 13).

Parallel with the above effort, two SiC depositions (Nos. 9081-L-4 and 9081-L-5) were performed in RF #5 to fabricate a SiC faceplate of appropriate thickness. Another

***This furnace was constructed in part by corporate funds.

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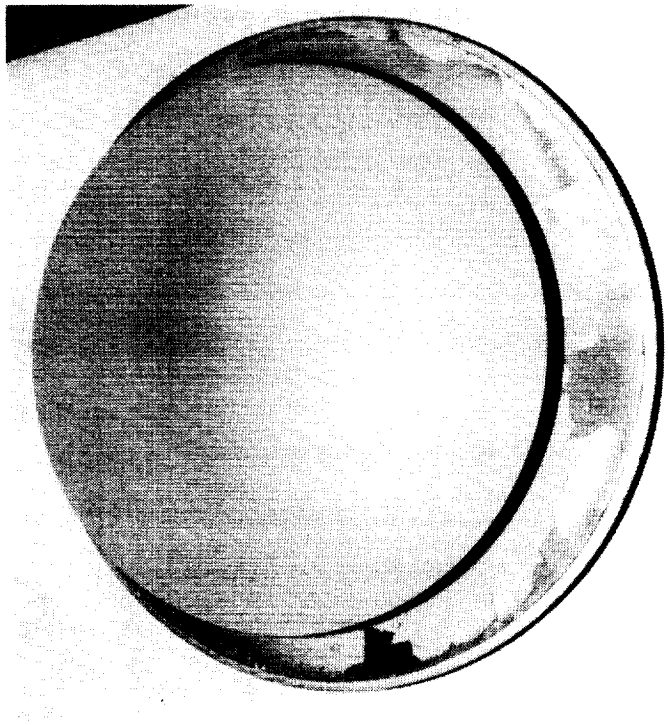


Figure 16. Silicon carbide faceplate for 40-cm-diameter mirror. The faceplate has not been separated from the mandrel.

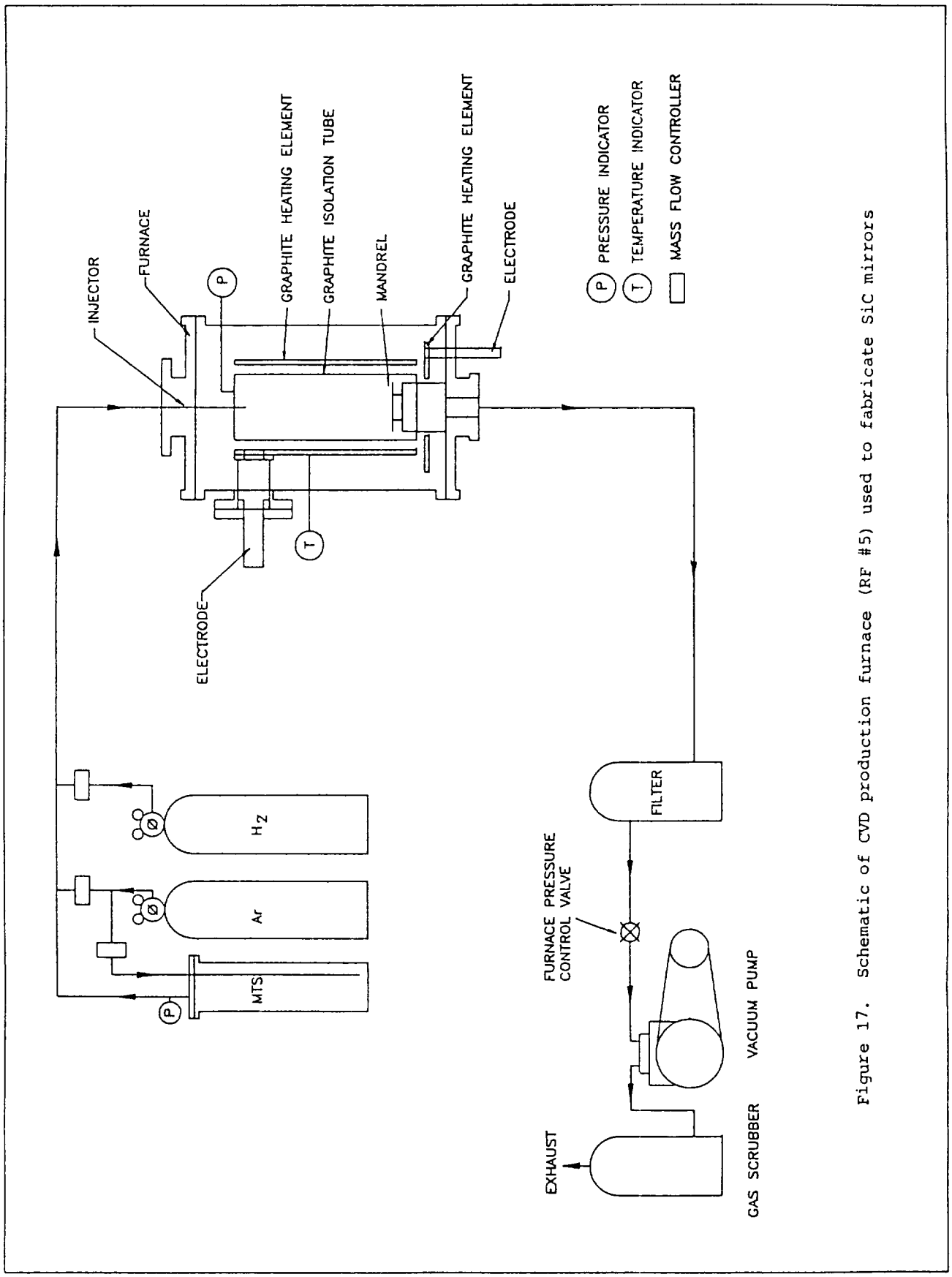


Figure 17. Schematic of CVD production furnace (RF #5) used to fabricate SiC mirrors

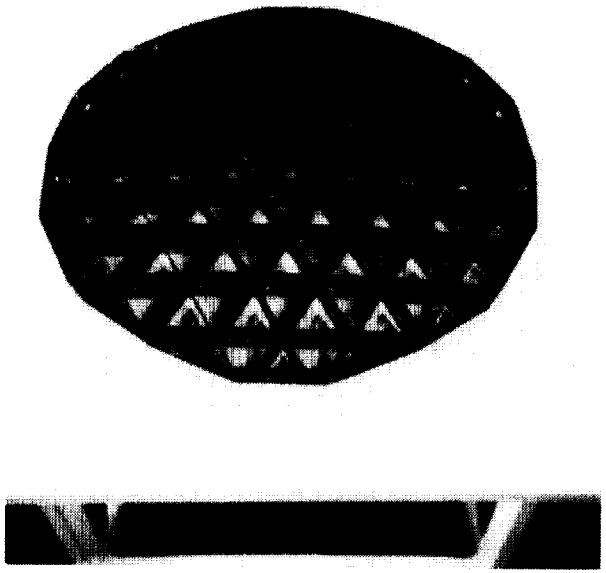


Figure 18. A lightweight graphite core for the 40-cm-diameter mirror

aim of these depositions was to evaluate the quality of the SiC deposit on a curved mandrel before fabricating the SiC LWS. In deposition #9081-L-4, the SiC deposit had the required thickness, but four injector growth marks were also observed. The deposited material did not appear similar to our usual SiC, and the deposition rate was also quite high. However, the SiC material on the mandrel did not show any evidence of cracking. A detailed analysis of the furnace data indicated that the thermocouples were malfunctioning, i.e., the deposition temperature was actually 1450 C as opposed to 1340 C indicated by the thermocouple.

In Run #9081-L-5, the above problem was corrected and another deposition was performed. However, this deposition also showed injector "growth marks" on the deposit. Consequently, to improve the deposition uniformity, a seven injector configuration -- one central injector surrounded by six outer injectors, was used in place of four injectors.

In Run #9081-L-6, an attempt was made to fabricate the SiC LWS and bond it to the SiC faceplate fabricated in Run #9081-L-3. In this run, a seven injector configuration was used. Further, the total flow rate of gases through the reactor was substantially increased to obtain a more uniform SiC deposition along the depth of the LWS. This deposition worked well in enclosing the graphite lightweight core, but the SiC deposit near the bottom of the LWS was quite thin. Further, the SiC LWS structure did not stick well to the faceplate.

In deposition Nos. 9081-L-7 and 8 two more attempts were made to fabricate a 40-cm-dia SiC mirror faceplate. In these depositions, a gas shroud technique that worked quite well in the Large Mirror Substrate Program was also used. In this technique, a small gap of width 1.0-mm was provided all around the mandrel. Through this gap an inert gas, argon, was passed at a high velocity. This flow prevented SiC deposition in this gap thereby isolating the SiC deposit on the mandrel from the rest of the furnace area. Good quality SiC was deposited on the mandrel, and the gas shroud technique worked quite well but the material cracked. Thus, a 40-cm-dia faceplate could not be fabricated. The cause of the material cracking could not be identified, although it appeared that material cracked during cooldown.

Since the funds in this SBIR program were nearly exhausted, it was decided to

reduce the diameter of the mirror to 25-cm and combine the mirror fabrication effort of this program with the effort in the Large Mirror Substrate Program. In this latter program, the replication experiments were being performed on a 25-cm-dia SiC mandrel. However, the 0.5-m reactor had capacity for two 25-cm-dia mandrels. Consequently, one mandrel location was used to fabricate SiC faceplate and LWS for the NASA program.

The 25-cm-dia mirror was designed by scaling the 0.5-m-dia mirror in the same manner as previously used for the 40-cm-dia mirror. The lightweight structure had 54 triangular cells. Each side of the triangular cell was 3.91-cm long and 2.29-cm high. Six circular mounting poles were provided in the LWS. Further, a hole in the backstructure was also provided to permit NASA to drill a hole in the center of the mirror faceplate.

The 25-cm-dia mirror substrate was fabricated in deposition Nos. 9081-L-9 through 9081-L-11. The mirror faceplate was fabricated in Run #9081-L-9. No difficulty was encountered in separating the faceplate from the mandrel. Figure 19 shows a picture of the SiC faceplate. A few nodules are visible on the surface but otherwise, the deposition surface is quite smooth. The nodules on the SiC faceplate were cleaned with a hand grinder and a graphite lightweight core was bonded to the SiC faceplate (Fig. 20). This picture clearly shows the six mounts that are an integral part of the LWS. In Figure 20 the graphite ribs in the center have not been removed to permit the center hole.

Deposition Nos. 9081-L-10 and 11 were used to overcoat the graphite core with SiC. Two depositions were required because in the first deposition, uniform SiC thickness was not obtained at all places in the backstructure. This is a consequence of the nonsymmetry associated with simultaneously depositing on two 25-cm-dia mandrels placed on a 50-cm-dia baffle. Figure 21(a) and (b) show the side and backside of the 25-cm-dia lightweight mirror. The weight of the mirror was measured and corresponded to a weight specification of 33 Kg m^{-2}. No Si cladding was performed and this mirror was delivered to NASA for evaluation.

In summary, the large scale experiments have demonstrated the scaling of the CVD-SiC process, the graphite lightweight core, the SiC LWS and the CVD-SiC mirror fabrication technology.

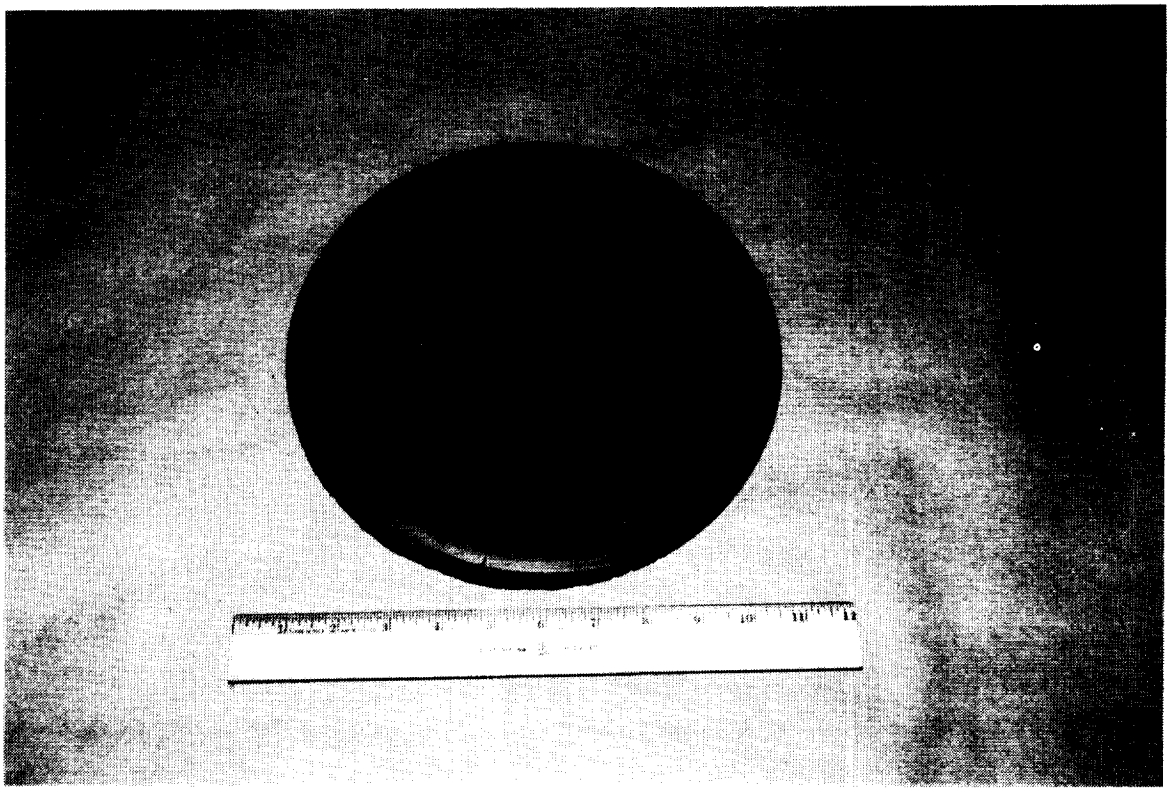


Figure 19. A picture of the SiC faceplate for the 25-cm diameter mirror

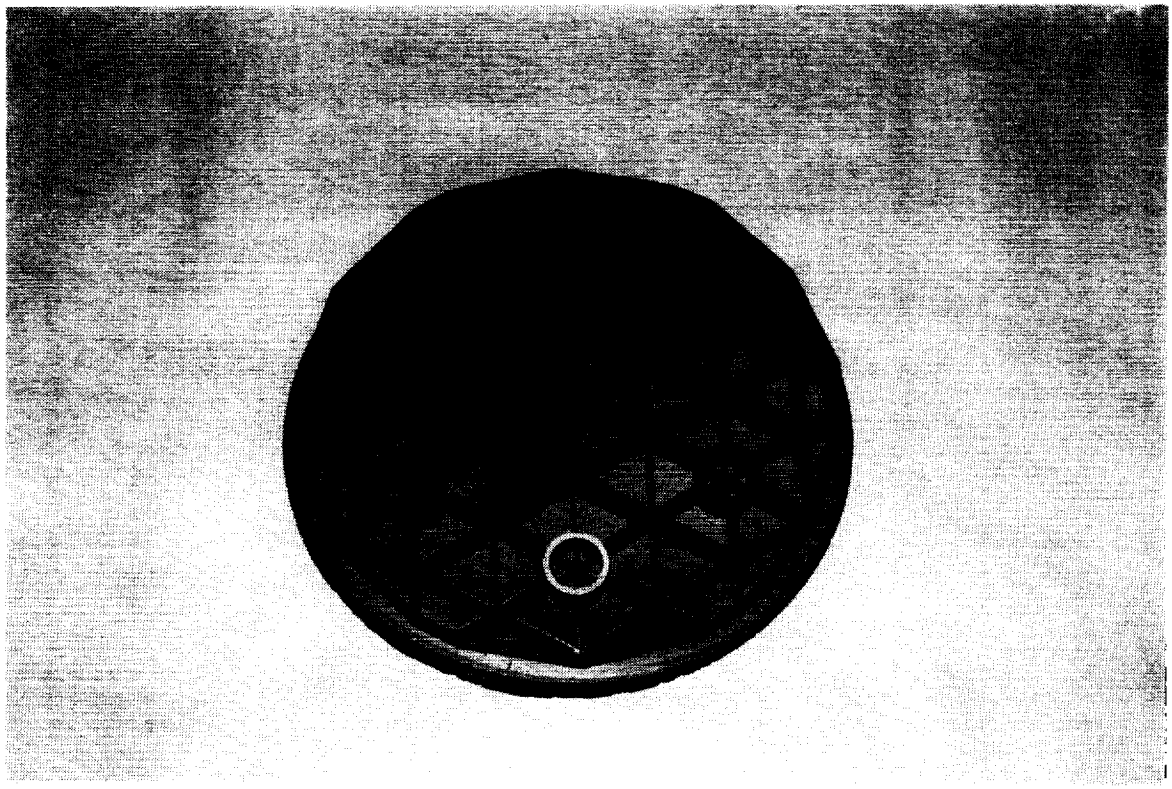
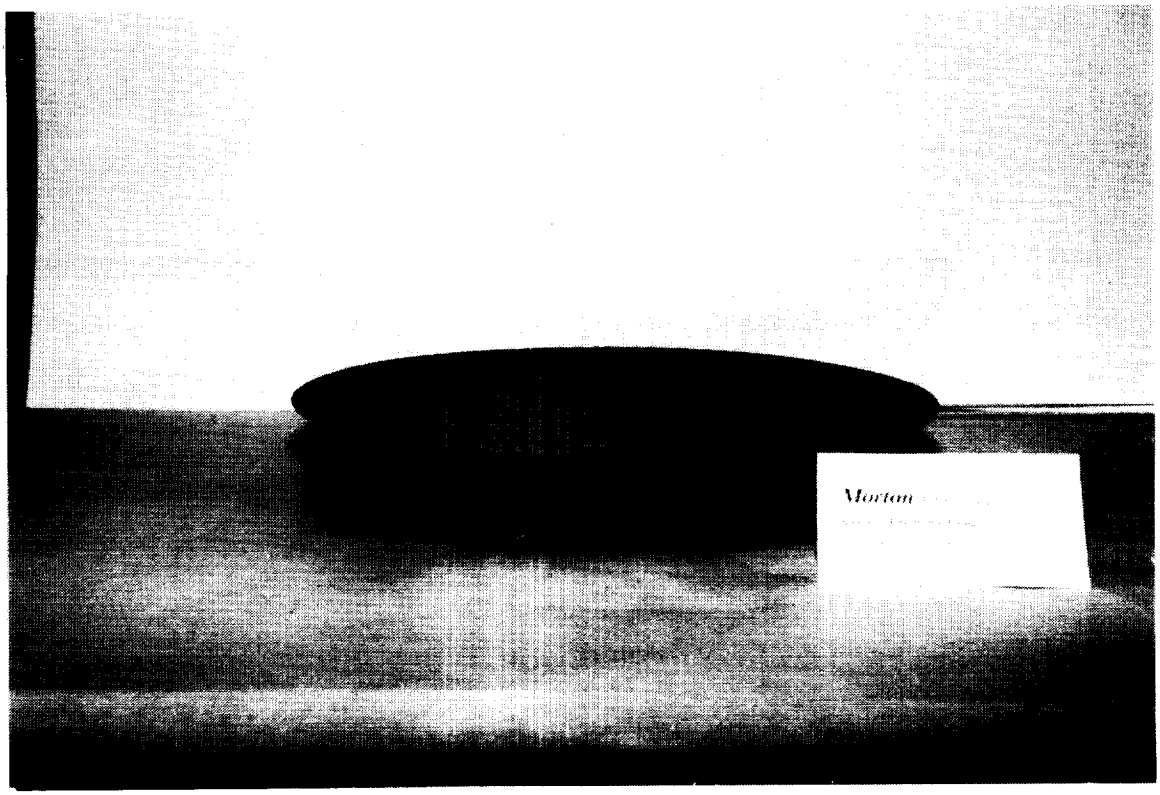


Figure 20. A picture of the graphite lightweight core bonded to the 25-cm diameter SiC faceplate



(a)

(b)

Figure 21. Two pictures of the 25-cm diameter lightweight mirror (a) side view, (b) back view

5.0 SUMMARY AND CONCLUSIONS

The CVD approach of fabricating lightweight Si/SiC mirrors was demonstrated. In this approach, a SiC faceplate is first replicated to a near-net-shape on a graphite mandrel and a lightweight SiC backstructure is bonded to the backside of this faceplate in a CVD reactor. Then, the SiC faceplate is cladded with CVD-Si and the Si surface is optically fabricated. Small scale Si and SiC depositions were performed to develop the Si/SiC mirror fabrication procedure and resolve important issues relevant for this technology. Specifically, the following features of the CVD technology were demonstrated: (i) near-net-shape replication of Si and SiC on graphite, (ii) CVD-Si cladding on CVD-SiC, (iii) fabrication of graphite lightweight cores, (iv) fabrication of SiC lightweight structures and (v) CVD-SiC bonding to itself. Several 7.5-cm-dia curved and flat Si/SiC lightweight mirrors were fabricated and polished to a figure of $\lambda/5$ (λ = .6328 μm) and finish of $\leq$ 10 Å RMS. Two such mirrors (one flat and one curved with a radius of curvature of 1.0-m) were delivered to NASA for evaluation.

The SiC CVD process was scaled from a small, horizontal research furnace to a small, vertical production furnace and SiC faceplate of 25-cm-dia and a radius of curvature of 1.0-m was successfully fabricated. The fabrication of graphite core and the SiC LWS was also demonstrataed at the 25-cm-dia scale. One 25-cm-dia lightweight SiC mirror was successfully fabricated to demonstrate the scaling of the CVD mirror fabrication technology. In this mirror, six mounts were also provided as an integral part of the SiC backstructure. This mirror blank was delivered to NASA for optical fabrication and metrological evaluation.

This SBIR program has been very successful. Most of the objectives of the NASA program have been met. The CVD mirror fabrication technology has also been commercialized and three products - CVD-SiC, Si and lightweight SiC mirrors are currently being marketed by CVD Incorporated (Appendix I). This research and development effort resulted in publication of four technical articles and filing of four patent applications (Appendix II). One patent application has already been approved.

Deliverables. The following items have either been delivered or are being

delivered to NASA concerning this SBIR Phase II program: (i) seven quarterly reports, (ii) several small samples of CVD-SiC for evaluation, (iii) two 7.5-cm-dia Si/SiC lightweight mirror models; the optical surface of one mirror was flat while the other had a radius of curvature of 1.0-m, (iv) a 25-cm-dia lightweight SiC mirror and (v) final technical report.

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8. J.S. Goela and R.L. Taylor, "Polycrystalline Silicon Improved Materials Property Data Base for Cooled Laser Mirrors," Air Force Wright Aeronautical Laboratories, Rept. No. AFWAL-TR-86-4131, CVD Incorporated, Rept. No. TR-031, March 1987.

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10. M.A. Pickering and R.L. Taylor, "Fabrication of Large Mirror Substrates by Chemical Vapor Deposition," Air Force Wright Aeronautical Laboratories Technical Report No. AFWAL TR-87-4016, April 1987.

11. J.S. Goela and R.L. Taylor, "Chemical Vapor Deposition for Silicon Cladding on Advanced Ceramics," J. Am. Cer. Soc. 72(9), 1747-50 (1989).

12. J.S. Goela and R.L. Taylor, "Rapid Fabrication of Lightweight Ceramic Mirrors via Chemical Vapor Deposition," Appl. Phys. Lett., 54(25), 2512-2514 (1989).

13. J.S. Goela and R.L. Taylor, "Fabrication of Lightweight Si/SiC LIDAR Mirrors," in SPIE Proceedings, Vol. 1062, International Society for Optical Engineering, Bellingham, WA, 1989, pp. 37-49.

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15. J.S. Goela and R.L. Taylor, "Large Scale Fabrication of Lightweight Si/SiC LIDAR Mirrors," in SPIE Proceedings, Vol. 1118, International Society for Optical Engineering, Bellingham, WA, 1989, pp. 14-24.

7.0 APPENDIX I

POTENTIAL APPLICATIONS AND COMMERCIALIZATION OF TECHNOLOGY

The CVD technology to fabricate lightweight Si/SiC mirrors as developed in this program has several applications. Lightweight Si/SiC mirrors can be used in the NASA LASA and LDR programs while large area SiC can be used for the NASA FUSE program. Silicon, SiC and Si/SiC hybrid materials may be used by the commercial sector, NASA and the military for fabricating optical components for high energy lasers, high power beam concentrators and deflectors, solar energy concentrators, LIDARs and astronomical telescopes. This research has the potential to fabricate aspheric surfaces in a cost-effective manner and to increase the current large mirror fabrication capability by at least an order of magnitude. The use of near-net-shape fabrication technology will permit fabricating a large number of mirrors in a cost-effective manner. This research can also be extended to the fabrication of SiC gratings and complex shaped Si/ceramic components for use in adiabatic engines and diffusion furnaces.

Technology Commercialization

CVD Incorporated has commercialized the CVD mirror fabrication technology developed in this SBIR program. Three products are currently marketed: (i) CVD-SiC, (ii) CVD-Si and (iii) lightweight SiC mirrors. The CVD-SiC is sold in the form of sheets of material up to 1.25-cm thick. The SiC plates of 0.5-m-dia are fabricated in the 0.5-m-dia facility and then cut up into smaller parts to satisfy the customers requirements. Our customers are primarily optical fabricators who use SiC to generate optical components, such as mirrors, and sell them to customers. The SiC plates larger than 0.5-dia are fabricated in a 1.5-m-dia facility which is also operational at CVD Incorporated.

CVD Incorporated is also involved in applying the SiC cladding on a variety of substrates for other industries and government programs. Since CVD-SiC has excellent optical properties such as polishability, it has been used to clad other forms of SiC such as reaction bonded, sintered and hot-pressed, and foam material. The SiC coatings have

been applied on graphite mandrels to obtain near-net-shape replication of complex heat exchanger passages and flow passages for adaptive optics applications. Graphite crucibles used in crystal growth have also been coated on the inside with SiC to prevent contamination.

CVD-SiC process is currently sufficiently developed that it can be transferred to manufacturing department with minimal additional work. This is currently under consideration for 1992. Commercialization of SiC technology is part of the long term goals of the company. As the SiC business develops, CVD Incorporated will invest in appropriate plant and equipment and transfer the process into manufacturing.

CVD-Si has been marketed to meet the special requirements for small samples and also for cladding substrates such as SiC and graphite. Small research furnaces which can provide rectangular pieces 30-cm x 7.5-cm in area and circular pieces about 10-cm in diameter are currently used to fabricate CVD-Si. However, there are plans to use 0.5-m-dia facility to fabricate large pieces of CVD-Si as the need develops.

Lightweight SiC mirrors are currently marketed to satisfy the needs of NASA, private industry and military. The mirror design is usually provided by the customer while CVD Incorporated fabricates the mirror blank according to the design specifications. An outside vendor is used to get the mirror optically polished, if the customer so requires. Currently, 0.5-m-dia facility is used for this purpose. However, 1.5-m-dia facility can also be used, if a requirement for large mirrors exist.

8.0 APPENDIX II

TECHNICAL PUBLICATIONS AND PATENTS DISCLOSURES

The research and development performed in this program resulted in publication of four technical articles as given below:

1. J.S. Goela and R.L. Taylor, "Chemical Vapor Deposition for Silicon Cladding on Advanced Ceramics," J. Am. Cer. Soc. 72(9), 1747-50 (1989).

2. J.S. Goela and R.L. Taylor, "Rapid Fabrication of Lightweight Ceramic Mirrors via Chemical Vapor Deposition," Appl. Phys. Lett., 54(25), 2512-2514 (1989).

3. J.S. Goela and R.L. Taylor, "Fabrication of Lightweight Si/SiC LIDAR Mirrors," in SPIE Proceedings, Vol. 1062, International Society for Optical Engineering, Bellingham, WA, 1989, pp. 37-49.

4. J.S. Goela and R.L. Taylor, "Large Scale Fabrication of Lightweight Si/SiC LIDAR Mirrors," in SPIE Proceedings, Vol. 1118, International Society for Optical Engineering, Bellingham, WA, 1989, pp. 14-24.

In addition two patents have been issued. One patent application has been approved and one application is pending. The details about these patents are given below:

1. J.S. Goela, M. Pickering and R.L. Taylor, "Lightweight Structures and Methods for the Fabrication Thereof," filed with US Patent Office, US Serial #626,393 (11 December 1990).

2. J.T. Keeley, J.S. Goela, M. Pickering and R.L. Taylor, "Selective Area Growth in a Vapor Deposition System," Patent #4,990,374 (February 5, 1991).

3. J.S. Goela, R.D. Jaworski and R.L. Taylor, "Method to Prevent Backside Growth on Substrates in a Vapor Deposition System," Patent #4,963,393 (October 16, 1990).

4. J.S. Goela and R.L. Taylor, "Fabrication of Lightweight Ceramic Mirrors via CVD" (application approved).

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Report Documentation Page

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Langley Technical Monitor: Dwayne E. Hinton

16. Abstract

A new, chemical vapor deposition (CVD) process has been developed for fabricating lightweight, polycrystalline silicon/silicon-carbide (Si/SiC) mirrors. The process involves three CVD steps: one to produce the mirror faceplate; the second, to form the lightweight backstructure, which is deposited integral to the faceplate; and the third and final step, to deposit a layer of optical-grade material, e.g., Si, onto the front surface of the faceplate. The mirror figure and finish are fabricated into the faceplate.

Using this process, 7.5-centimeter diameter flat and curved Si/SiC lightweight mirrors were fabricated successfully. The process was scaled to produce a 25-centimeter diameter SiC mirror having a radius of curvature of 1.0 meter. A SiC faceplate of 1.0-meter diameter and radius of curvature of 2.0 meters has been fabricated with this same process.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Chemical Vapor Deposition; Lightweighting; Optics; Polycrystalline Silicon Carbide	Unclassified-Unlimited Subject Category - 74

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