Review Papers

LASER-INDUCED CHEMICAL VAPOUR DEPOSITION OF SILICON CARBIDE

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INTRODUCTION

Silicon carbide, SiC is a promising material for protective coatings resisting high temperatures, corrosive and oxidative effects and wear. It is also of great interest in high temperature ceramic composites and engines. It is only very recently that SiC has arrested much attention also as a semiconductor for use in optoelectronic and electronic devices at high temperatures. High temperature stability makes it a candidate for wanted materials in e.g. solar cells, and high power electronic circuits. The attractivity of SiC is brought about by the occurrence of many crystallographic modifications - there are as many as 170 polytypes of different electronic properties which differ by stacking orders of an elemental pair of Si and C layers in the most densely packed directions [1]. A wide forbidden gap of SiC (up to 3 eV) is suitable for construction of e.g. blue light emitting diodes. Although the polycrystalline SiC is known to have been produced from the end of the last century and its main application is related to high chemical and thermal stability, the nowadays use of SiC responds mostly to demands of electronic and optoelectronic industries for high temperature semiconductors, and of heavy mechanics for high quality sinterable powders for high temperature components. Large scale fabrication processes affording films of desired properties or particles with desired composition, size and shape are, however, still not available and some earlier and present techniques have disadvantages in low product yields, formation of bulky agglomerates, and in an inadequate control and irreproducibility of the process.

This paper is a survey of the very recently developed laser technique enabling deposition of SiC materials from the gaseous precursors, which appears promising in production of high quality powders and layers of SiC. It presents the effects of reaction conditions on the nature and composition of SiC products.

RECENT TECHNIQUES FOR PROCUCTION OF SiC LAYERS AND POWDERS

Solid products (thin layers and powders) of SiC are most often produced by chemical vapour deposition (CVD) [2-6], plasma-enhanced chemical vapour deposition (PECVD) [7-11], or by sputtering techniques [12-15]. Thin layers of SiC have also been produced by other procedures as reactive-ion-beam-deposition [16, 17], laser evaporation (ablation) [18-20], or by photo-CVD technique using mercury or deuterium lamps [21-24]. The laser evaporation (ablation) consists of the interaction of laser radiation with a solid target material which is driven into the gas phase and subsequently condensed onto a substrate. The advent of lasers brought also promising developments in the gas-phase reaction techniques; the direct interaction of gaseous silicon and carbon-containing molecules with laser radiation involves many advantages over other gas-phase techniques and provides a basis of the method called the laser-induced chemical vapour deposition (LICVD).

LASER-INDUCED CHEMICAL VAPOUR DEPOSITION

LICVD is initiated as homogeneous or heterogeneous chemical reactions of volatible precursor to solid deposits which occur in the gas phase or on a hot substrate. The use of laser radiation for the inducement of LICVD can be divided according to three basic mechanisms into laser heterogeneous pyrolysis, laser homogeneous pyrolysis and laser photolysis.

In the pyrolysis, laser radiation impinges upon the desired substrate and heats it locally over the area of the laser beam (Fig. 1a), or it heats a local zone of the gas phase above the substrate (Fig. 1b). Gaseous reactant molecules in the proximity of the hot substrate area, or those contained inside the hot gaseous zone,

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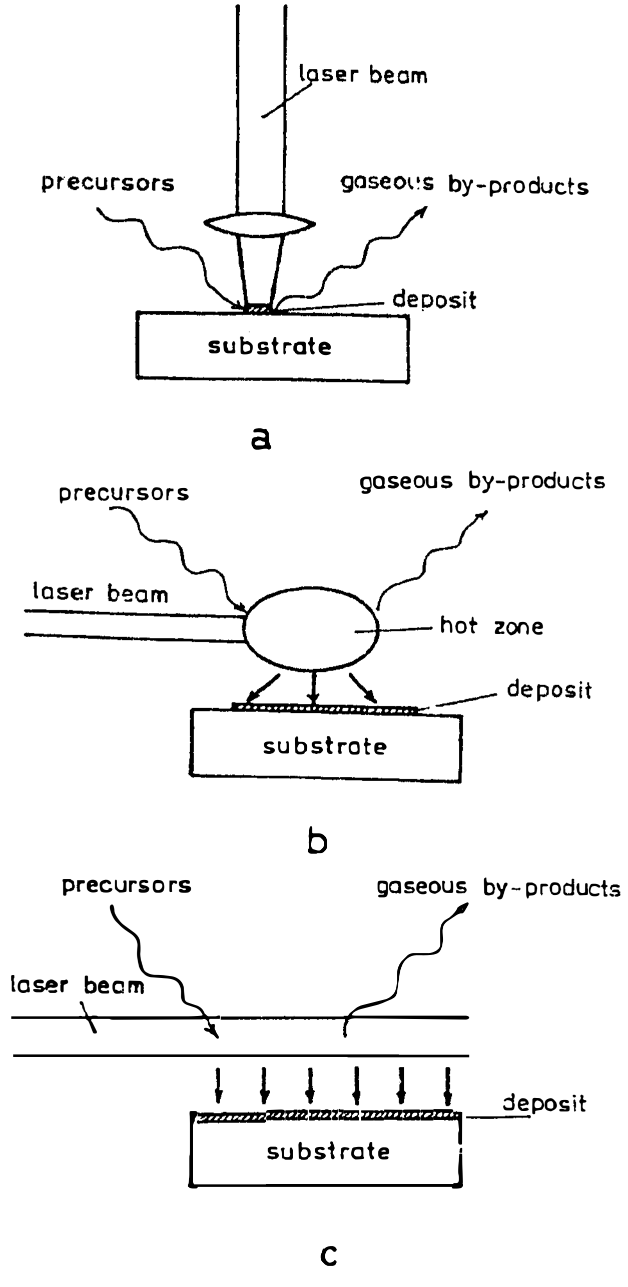

Fig. 1. Schematic diagram of LICVD process; heterogeneous pyrolysis (a), homogeneous pyrolysis (b) and photolysis (c).

undergo thermal reactions which result in the formation of the desired solid SiC and gaseous by-products. Heating of the substrate can be achieved by a focused radiation of a continuous-wave laser which is not absorbed in gaseous phase. Heating of the gas-phase zone is usually carried out via processes of infrared multiphoton absorption [25] or laser-photosensitized decomposition [26–28]. The former is initiated by a pulsed (usually CO_2) laser and the latter involves the use of either pulsed or continuous-wave infrared laser.

The laser beam absorbed by gaseous species in a single-photon event leads to the eletronic excitation of precursor molecules and to their photolytic fragmentation. The photolytic products can either react with other species present in the system, or can condense onto nearby surfaces (Fig. 1c). This type of LICVD is suitable for production of large area deposits (10's cm^2) and the lasers used for such a photolysis emit ultraviolet or visible radiation.

LICVD of SiC has several advantages over other gas-phase techniques. These are high deposition rates, easier initiation of the process, lower temperatures of the substrate and high quality of the deposit. While the temperatures of CVD processes range between 1000–1500°C, that of LICVD can be decreased by about 600°, often to even 350–500°C. This is of crucial importance for production of multilayered semiconductor structures used for electronic devices. The lower temperatures decrease mechanical tension between individual layers and make therefore unwanted deformation defects generation and migration as well as the redistribution of dopants less important. The better quality of layers in LICVD is related to the elimination of bombarding the growing layers by accelerated particles, which are important in PECVD or in ion-sputtering.

Pyrolytic LICVD occuring via homogeneous (the gas-phase) and heterogeneous decomposition of precursor (gaseous molecules adsorbed on substrate, i.e. adlayer molecules) is controlled in a different way. Provided that the intensity of laser radiation is constant across the beam and the decomposition takes place only in monomolecular layer of adsorbed molecules, the decomposition rate of adsorbed precursor (as well as the deposition rate) can be described as

$$R_a = \frac{N_a.\sigma_a.P_L}{h\nu.\pi.a^2}$$

where N_a is a number of molecules adsorbed on unit area, σ_a absorptivity of laser radiation, P_L laser output, $h\nu$ laser photon energy, a an effective beam diameter, and subscript a refers to the adlayers.

The deposition rate for homogeneous pyrolysis of precursor is given [29] by similar expression

$$\rho_g = \frac{N_g.\sigma_g.P_L}{h\nu.\pi.a^2}$$

where the subscript relates to the gas phase.

The fundamental parameter in the heterogeneous pyrolytic LICVD is local temperature within the reaction zone. The temperature rise on a substrate surface which is due to absorption of laser radiation, depends on the reflectivity and thermal conductivity of the irradiated solid. These quantities are initially

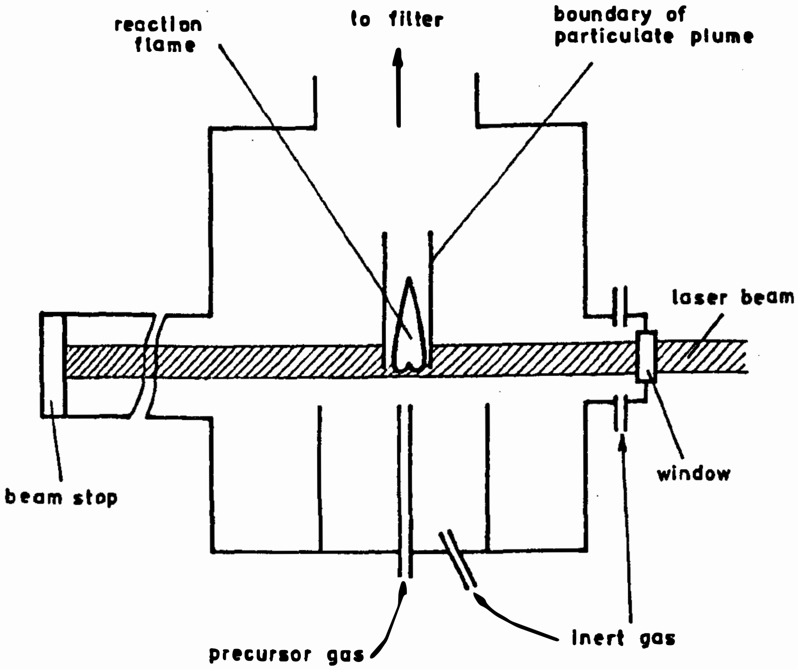

Fig. 2. Reactive vessel for LICVD of ultrafine SiC powders.

determined solely by the substrate, but will become progressively dependent on the deposited layers. The increase in temperature of the irradiated surface can be simply described [30] as

$$\Delta T = \frac{P(1 - R_s)}{2\sqrt{\pi} W_0 \chi}$$

where P is laser output, R_s a surface reflectivity of substrate (layer), χ thermal conductivity of substrate (layer), and W_0 the radius of the Gaussian laser beam at $1/e^2$ intensity. Provided that both R_s and χ are very little affected with temperature, the growth rate of the deposit is controlled by the well known Arrhenius law

$$v = v_0 \exp \frac{-E_a}{kT}$$

where v, v_0, T, k and E_a designate in the given order growth rate, temperature, Boltzmann's constant, and activation energy.

In the photolytic LICVD, where precursors are furnished from the gas phase, the efficiency of the deposition is determined by collisional parameters (deactivation) in the gas phase, the mutual configuration and distance between laser beam and substrate surface, sticking coefficient, and possible deexcitation processes on the surface. The simpliest direct deposition from the gas phase as a consequence of a single photon absorption is controlled by the linear relationship between the deposition rate and laser fluence. However, contributions of adlayer molecules to deposition process cannot be often neglected and they can affect both the deposition rate and chemical processes (photo-fragmentation and reactions of photoproducts) involved.

In a simple approximation [30], the net flux of reactants, Φ_P, is proportional to the laser fluence Φ_L:

$$\Phi_P = \frac{\Phi_L}{h\nu} (\eta_g F_g + \eta_a F_a)$$

where $h\nu$ is the photon energy, F is a probability factor for activated species to reach the reaction zone and to undergo reaction and η designates quantum yield. The subscripts g and a refer, again, to the gas phase and the adsorbed molecular layer. The quantum yields are usually independent of time and space coordinates.

DEPOSITION OF ULTRAFINE SiC POWDERS

Laser synthesis of ultrafine SiC powders with ideal characteristics (small particle size, freedom from agglomeration, a narrow range of sizes, a spherical shape and highly controlled purity) for use in ceramics has been demonstrated by employing the irradiation of continuous-wave CO_2 laser and different gaseous mixtures of precursors as silane/acetylene [31–33], silane/ethylene [32, 34–37] and silane/methane [34–36]. The resonant absorption of the laser radiation in the gaseous precursors produces silylene, SiH_2 and carbonaceous radicals which react in the gas phase

Table I

CW CO_2 Laser Synthesis of Ultrafine SiC Powders

Wavelength [μm]	Laser output [W]	Beam intensity [kW/cm^2]	Precursors	Total pressure [Torr]	Growth rate [g/hr]	Powder properties	Surface area [m^2/g]	Ref.
10.59	50	2–3	SiH_4/C_2H_2	200–800	12	β-SiC microcrystallites	50–100	31
10.53, 10.59, 10.63, 10.22, 10.72	40–50	1.6–2	SiH_4/C_2H_2	600–900	1.2–3.3	nonstoichiometric β-SiC[a]		32
10.59, 10.22			SiH_4/C_2H_2[b]	760	∼ 2	stoichiometric SiC or C[b]		
	50–200		SiH_4/C_2H_2	100–500		stoichiometric[c] crystaline β-SiC	120–240[d]	33
10.59	150	0.27 1.02 and up to 10^2	SiH_4/C_2H_4 SiH_4/CH_4	150 150		<0.1μm agglomerated spheres in chain network[e]	84 98	34,35
10.59		0.5–5.3	SiH_4/C_2H_4	300–600		polycrystalline β-SiC < 0.05 μm	44–106	37

[a] The maximum C/Si ratio 0.9 for an initial C/Si ratio 1.25.

[b] Amorphous carbon powders obtained with low SiH_4 flow rate when SiH_4 acts as a sensitizer for C_2H_2 decomposition.

[c] For $SiH_4/C_2H_2 = 2$.

[d] 120 m^2/g relates to low laser powers and flow rates, while 240 m^2/g refers to laser power 200 W and higher flow rates.

[e] Powders produced from C_2H_4 lack internal and surface porosity.

to products nucleating as well in the gas phase. This nucleation is ensued by a grow-up of the particles in the same phase. The most detailed description of the pyrolytic LICVD is reported in ref. [38]. Schematic of a powder synthesis cell with the location of a reaction hot zone (flame) is given in Fig. 2. The laser beam and the reactive gas stream diluted with an coaxially introduced stream of inert gas usually intersect orthogonally, and the powder particles are collected by using system of filters. SiC powders are produced according to the following gas-phase reactions:

$$2SiH_4 + C_2H_2 \rightarrow 2SiC + 5H_2$$

$$2SiH_4 + C_2H_4 \rightarrow 2SiC + 6H_2$$

$$SiH_4 + CH_4 \rightarrow SiC + 4H_2$$

The conditions for the homogenous pyrolytic LICVD of SiC powders and powder properties are gathered in Table I.

The inferences from individual studies are helpful for possible scaling-up the process. These are:

(1) The specific surface area of powder decreases at lower flows of precursors and high temperatures (higher laser power) [31].

(2) The product of the laser power and the residence time appears to correlate with the particle growth more than with the degree of crystallization [31].

(3) Attempts to produce stoichiometric SiC were not succesful using silane/ethylene mixtures, and varying the laser emision line from the P(14) to P(32) in the ν_4 absorption band of silane does not yield any change in properties (Si/C ratio, specific surface area) of the powder [32].

(4) The Si/C ratio of the powder is greater with silane-acetylene than with silane/ethylene mixture under comparable conditions, which is brought about by an easier dissociation of acetylene and a higher exothermicity of the silane-acetylene reaction [32].

(5) The material production is limited by the complete depletion of precursors rather than by an insufficient radiation supply or by a short residence time inside the laser beam [33].

(6) The particle size with silane/acetylene mixture can be reduced at higher laser powers and flow rates [33]; that with silane/ethylene mixtures increases with increasing laser intensity, flow rate and partial pressure of silane [37].

(7) The flame temperature increase with the enhancement of the gas flow rate or of laser inci-

Table II

Laser Production of SiC Layers

Laser	Precursor	Type of LICVD	Substrate	Substrate temperature [°C]	Properties of layer	Reference
ArF	Si_2H_6/C_2H_2	photolysis	sapphire, α-Al_2O_3 (0001)	1150	epitaxial or polycrystalline films	40,41
XeCl	CH_3SiCl_3	[a]	Si, MgF_2	20-130[b]	Si/C/H films	42
Co_2	SiH_4/CH_4	pyrolysis	SiC		periodic structures	43
Ar	CH_3SiCl_3 $(CH_3)_2SiCl_2$	pyrolysis	glass		fibrous microstructures	44
CO_2	CH_3SiCl_3	pyrolysis	SiC, W, C, SiO_2, ZrO_2		fiber features	45
CO_2	CF_3SiH_3	photolysis[c]	Al	ambient	network of agglomerates	46

[a] Probability both photolysis and pyrolysis.

[b] Initial temperature.

[c] Infrared multiphoton decomposition.

dent power results in the formation of materials containing more carbon [33].

(8) Powders formed with silane/ethylene mixtures contain some carbon, whereas those yielded with silane/methane mixtures contain no carbon at all [35].

These studies show that this laser CVD process produces powders with all desired parameters and they present it as a better technique from among the few known in which a desired product is afforded in better yield and at low cost with a laser than by other methods.

Ceramic powders have also been prepared by the photodecomposition of various chloro(methyl)silanes using an ArF excimer laser radiation. These precursors are cheaper and easier to manipulate than silane. The dissociation of the chloro(methyl)silanes $(CH_3)_nSiCl_{4-n}$ (n = 1–3) and $(CH_3)_2SiHCl$ and CH_3SiCl_2H is assumed to generate new chemical species which absorb the majority of the laser energy. Stripping of ligands from the silicon atom by the combined effects of photolysis, heating and collisions with radicals is very effective and makes the LICVD of SiC powders potential for application due to the efficient usage of laser energy and high conversions of chloro(methyl)silanes [39].

DEPOSITION OF SiC LAYERS

Deposition of SiC layers by LICVD processes can be achieved by using various lasers which drive the decomposition of precursor as pyrolysis, photolysis, and as a mixture of both mechanisms. SiC films have been grown by using ArF laser photolytic LICVD and disilane/acetylene mixtures [40,41]. In this process, the rate of the built-up of the film is limited by the diffusion of the supplied precursors. Solely epitaxial growth of adherent films takes place only when the laser is incident on the substrate, while in the parallel irradiating configuration the films are polycrystalline and easy to be peeled off. Hydrogenated Si/C/H films can be developed by the irradiation of a moderately heated substrate with XeCl laser in the presence of methyltrichlorosilane [42]. The dynamics of the deposition of these materials are affected by substrate temperature, laser beam intensity and dose, and it is assumed that this process is controlled by photolysis as well as pyrolysis in adlayers and by photolysis of gaseous precursor. Heterogeneous pyrolytic LICVD of silane/methane mixtures carried out by laser heating of a hot-pressed SiC substrate results in the formation of localized SiC features [43]. The absorption of laser radiation in silane has been prevented by tuning a CO_2 laser to 9.27 μm wavelength. It was observed that deposits show a nucleation stage for irradiation times of up to 10^2 ms. Fibrous microstructures of SiC were fabricated in the presence of inert nitrogen and carbon dioxide using Ar laser pyrolytic LICVD from gaseous methyltrichlorosilane and dimethyldichlorosilane [44]. The pyrolytic LICVD of SiC fibrous microstructures has also been achieved through heating various substrates by CO_2 laser in the presence of methyltrichlorosilane [45]. The growth rate of SiC in this case was observed to be decreased by dilution of the organosilicon precursor with hydrogen. Microstructures of SiC have also been prepared [46] by infrared multiphoton decomposition of trifluoromethylsilane in which event reaction proceeds as an explosion. The irradiation with a single pulse from TEA CO_2 laser initiates the total decomposition of the precursor via a special sequence of steps of reductive chemistry. The explosive decomposition occurs when the pressure of trifluoromethylsilane exceeds a certain limit which depends on the laser fluence and the wavelength.

The conditions for these LICVD processes as well as properties of the produced SiC layers are compiled in Table II.

DEPOSITION OF POLYCARBOSILANES

Polycarbosilanes [47,48] have been recently disclosed as precursors [49–51] for thermal production of SiC materials, and it has been stressed that these polymers which possess a cross-linked structure are better suited for the purpose than linear-chain ones [48].

Also these polymeric materials can be prepared by LICVD techniques from volatile organosilicon compounds. CO_2 laser-induced plasma in gaseous tetramethylsilane has been used [52] to produce SiC particles enveloped with carbosilane polymer. In this process with effective temperatures around 3000 K, quantum yield can be increased with higher energy in laser pulse and higher tetramethylsilane pressure. Additional heating to 2000°C is sufficient to change the initial product into SiC. Another LICVD technique for production of polycarbosilanes is cw CO_2 laser photosensitized (SF_6) decomposition of silacyclohexane [52] and silacyclobutanes $R_2\overline{SiCH_2CH_2C}H_2$ ($R = H, CH_3, H_2C{=}CH, HC{\equiv}C$) [53–56].

The former decomposes by a variety of steps to yield Si/C/H deposits incorporating most of the silicon of the parent. The latter decomposition affords intermediary silylenes $R_2Si{=}CH_2$ which undergo very efficient polymerization into cross-linked polycarbosilanes. Nucleation occurs in the gas phase and the organosilicon polymers is deposited onto cold walls of reaction vessel. However, the ability of these polycarbosilanes to degrade into SiC at elevated temperatures has not yet been studied.

CONCLUSION

Laser induced deposition of silicon carbide from the gas-phase precursors allows feasible synthesis of ultrafine powders, it awaits its scale-up and can significantly contribute to sintering fabrication of novel SiC materials. Laser assisted deposition of SiC layers with desired properties for microelectronics remains, however, to be broadly examined before its suitability for selective production of different SiC films (polytypes) can be suggested. LICVD of polycarbosilanes, intermediates to SiC, might accomplish another channel for SiC materials in case that the ensuing thermal degradation of polycarbosilanes into SiC is an easy process.

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