Preparation of zirconium carbonitride by laser chemical vapor deposition using alkyl-amide precursor as a single source

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The vapor-phase growth of zirconium carbonitride (ZrCN) on carbon substrates was demonstrated by laser chemical vapor deposition using an alkyl-amide precursor as a single source. The effects of deposition temperatures on the phase formation, microstructure, chemical bonding states, and deposition rates of ZrCN layers were investigated. An amorphous layer with a large amount of oxygen impurity was deposited at a temperature of 1373 K, whereas a crystalline ZrCN phase with a face-centered cubic structure (fcc-ZrCN) was formed with an excess amount of free carbon at a deposition temperatures of 1453 K. The fcc-ZrCN layer had cone-like nodular morphology with a dense cross section, where the dendritic microstructure comprising nano-sized ZrCN grains with finely dispersed carbon was formed in the fcc-ZrCN layer at 1453 K. The deposition rate of the fcc-ZrCN layer was 40 μm h⁻¹.

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Key-words : Zirconium carbonitride, Laser chemical vapor deposition, Metal organic precursor, Microstructure, High deposition rate, Composition

1. Introduction

Zirconium carbide (ZrC) is one of the most conventional refractory carbides and an ultra-high-temperature ceramics with a melting point of 3805 K. It has the excellent thermochemical stability, outstanding hardness, great mechanical strength, high thermal and electrical conductivities, weak damage sensitivity under irradiation, and a low work function (4.0 eV) relative to Si.¹⁻⁴ Owing to these properties, ZrC has attracted great interest for applications in the extreme thermal and chemical environments, such as air-augmented propulsion, rocket propulsion, atmospheric hypersonic reentry, nuclear applications in generation-IV reactors, cutting tools, and field emission array devices.¹⁻⁶ ZrC has a rock-salt (fcc)-type crystal structure with the space group of Fm̅3m, which is the same as the crystal structure of zirconium nitride (ZrN). ZrC and ZrN form a complete solid solution of a carbonitride (hereafter termed fcc-ZrCN) which possesses remarkable mechanical, tribological, chemical, decorative and biocompatibility properties.⁷⁻⁸ It is known that these Zr-based non-oxide ceramics (ZrC, ZrN, and ZrCN) are difficult to be sintered to form dense bulk components owing to its high melting point and low self-diffusivity. The development of coating layers and films of Zr-based non-oxide ceramics can yield high-value-added components and devices not only for high-temperature-durable applications but also for biomedical fields. Indeed, films of ZrCN and its composites with Zr and carbon have been reported to demonstrate the potentiality for applications to medical devices having the improved biocompatibility with enhanced fatigue lifetime and the significant short-term antibacterial effect.⁸⁻¹²

Chemical vapor deposition (CVD) has potential advantages to synthesize highly dense and conformal coating layers. Through the regulation of deposition parameters, CVD enables the deposition of layers with excellent controllability of the microstructure as well as elemental and phase compositions. However, CVD often suffers from the harmful natures of precursors and by-products because one of the most common precursor species is halides, such as fluorides, chlorides, and bromides. These hazardous precursors and gas species in CVD limit its range of applications and experimental conditions for research and development. For instance, zirconium tetrachloride (ZrCl₄) with the combination of CH₄ and H₂ has been adopted as the most common precursor for the deposition of ZrC. Experimental and theoretical studies have been conducted to produce ZrC films via CVD by using chloride precursors with hydrocarbon and H₂ gases.¹⁻¹³⁻¹⁸

On the other hand, films of ZrC, ZrN, and ZrCN can be grown by CVD using metal organic precursors
Zirconium amide compounds are a class of precursors used for the atomic layer deposition of ZrO2 thin films owing to their volatility and reactivity.\textsuperscript{23–25} Among zirconium-amide compounds for Zr-based non-oxides, metal dialkyl-amide complexes, e.g., tetrakis-(ethylmethylamino)zirconium [Zr(NEtMe)4] and tetrakis-(diethylamino)zirconium [Zr(NEt2)4], are promising as less-toxic and less-hazardous alternative precursors, particularly for the growth of ZrN.\textsuperscript{25–28} However, there are still limited studies on the synthesis of Zr-based non-oxides by CVD techniques using dialkyl-amide complexes as a single source without H2 and NH3 gases.\textsuperscript{23,28}

Laser CVD is a versatile coating technique that enables the high-speed vapor growth of a variety of functional and engineering ceramics that are durable against high temperature.\textsuperscript{29–33} The present study aimed to synthesize CVD-ZrCN layers by using a dialkyl-amide complex as a single metal organic precursor with only Ar as a carrier gas. Here, we demonstrate the high-speed vapor-phase growth of fcc-ZrCN layers on carbon substrates by laser CVD; the effects of deposition temperature on the phase formation, composition, chemical binding state, and microstructure of the deposited layer were investigated.

2. Experimental procedure

ZrCN films were deposited by using a custom-built cold-wall-type CVD instrument equipped with a Nd:YAG laser. Details of the laser CVD apparatus are described elsewhere.\textsuperscript{29} The precursor used for zirconium was Zr(NEtMe)4 (99.99%, Sigma-Aldrich). Zr(NEtMe)4 liquid was heated to 353–373 K in a bubbler, and the resulting vapor was carried into a CVD reactor chamber with Ar carrier gas at a flow rate of 8.3 \times 10^{-7} \text{m}^3 \text{s}^{-1}. The tubes and nozzles of the pathway for the gaseous precursor were maintained at 373 and 393 K, respectively. A carbon substrate [10 mmφ, 0.5 mm (t); graphite, Sankyou carbon Co., Ltd., Japan] was set on a heated stage at 673 K. The deposition surface of the substrate was irradiated with a Nd:YAG laser in the continuous-oscillation mode during the deposition. The spot size of laser irradiation was approximately 15 mmφ, which covered the entire deposition surface of the substrate. The deposition temperature was monitored using a pyrometer. The laser power density ranged from 83 to 117 W cm\textsuperscript{-2}, and the corresponding deposition temperatures range was 1373–1453 K. During the deposition, the total pressure in the reactor chamber was maintained at 300 Pa, and the deposition time was 600 s. The deposition parameters are summarized in Table 1.

### Table 1. Deposition parameters for ZrCN layers by laser CVD

| Parameter                        | Value                  |
|----------------------------------|------------------------|
| Precursor                        | Zr(NEtMe)4             |
| Evaporating temperature          | ~373 K                 |
| Ar carrier gas ow rate           | 8.3 \times 10^{-7} \text{m}^3 \text{s}^{-1} |
| Substrate                        | carbon (graphite)      |
| Laser power density (P\textsubscript{L}) | 83–117 W cm\textsuperscript{-2} |
| Deposition temperature (T\textsubscript{dep}) | 1373–1453 K |
| Total pressure (P\textsubscript{t})     | 300 Pa                 |
| Deposition time                   | 600 s                  |

Figure 1. XRD patterns of layers deposited by laser CVD at temperatures of (a) 1373 and (b) 1453 K.

### 3. Results and discussion

The deposition temperature, which varied with laser power density, is one of the dominant process parameters that determine the characteristics of laser-CVD layers. Figure 1 shows the effect of deposition temperatures on the XRD pattern of the deposited layer. The XRD pattern of the layer deposited at 1373 K (laser power density of 83 W cm\textsuperscript{-2}) shows a broad reflection peak around 2θ = 31°, indicating the formation of an amorphous layer on the graphite substrate, as shown in Fig. 1(a). At 1453 K (laser power density of 117 W cm\textsuperscript{-2}), peaks appeared at 33.3, 38.6, and 55.8°, which corresponded to 111, 200, and 220 reflections, respectively, of an fcc phase [Fig. 1(b)]. From the reflection peak positions, the lattice parameter of the deposited layer deposited at 1453 K was estimated as 0.4657 nm, which is between the reference lattice parame-
As the alkyl-amide precursor, Zr(NEtMe)4, contains the C and N elements, the solid solution of ZrC and ZrN with the fcc lattice, ZrCN, was formed. Assuming that the relationship between the lattice parameter and C/N composition of zirconium carbonitride follows the Vegard’s law, the deposited layer was ZrC0.71N0.29.

Figure 2 shows XPS spectra for layers deposited at 1373 and 1453 K. Wide-scan spectra (not shown) exhibited photoelectron peaks assigned to Zr, C, and N elements from the Zr(NEtMe)4 precursor and oxygen, which occurred as an impurity from the residual gas species in the chamber because the present CVD technique was not a high-vacuum process (base pressure: \( \sim 20 \) Pa) without hydrogen gas. There is a significant difference in photoelectron spectra of Zr3d between the amorphous layer prepared at 1373 K and the fcc-ZrCN layer prepared at 1453 K, as shown in Fig. 2(a); the Zr3d spectrum of the amorphous layer had apparently three peaks, whereas that of the fcc-ZrCN layer comprised two peaks with shoulder at a higher binding energy. Zr3d peaks appear as a doublet consisting of Zr3d3/2 and Zr3d5/2 peaks, which can be deconvoluted on the assumption that the spin–orbit splitting in the Zr3d spectra is the same for all of the phases and equal to 2.4 eV and the integrated intensity of the Zr3d3/2 peak relative to that of the Zr3d5/2 peak is equal to the spin–orbit multiplicity of 2/3.\(^{30}\) Hereafter, the position of the Zr3d3/2 is only referred, assuming that the Zr3d3/2 is placed at 2.4 eV higher binding energies in the Zr3d doublet term, for simplicity. In the fcc-ZrCN layer prepared at 1453 K, a deconvoluted doublet curve with the peak position of 179.4 eV were dominant, which was assigned as a carbide (Zr–C).\(^{4},^{8},^{37},^{38}\) There were also the Zr3d doublet curve at 180.6 eV, which was assigned to a nitride (Zr–N),\(^{8}\) while the binding energy (181.2 eV) of the other doublet curve was consistent with that reported to be the Zr–C–O (181.2 eV) configuration in the literature.\(^{39}\) In the amorphous layer prepared at 1373 K, there was the Zr3d doublet curve located at 182.6 eV, which were attributed to the oxide configuration (Zr–O),\(^{8},^{39}\) and Zr3d doublet curves of Zr–C and Zr–N, in which the Zr–O configuration was dominant (56.6 % of the areal ratio). The slight deviation between the spectrum and fitting curve in the Zr3d spectrum for the amorphous layer prepared at 1373 K might be due to the existence of the other configurations such as Zr–C–O and Zr–O–N.\(^{39}\) Although the deconvolution with additional sets of doublet curves (Zr–C–O and Zr–O–N) may provide more precise analysis, the Zr3d spectra confirmed the formation of Zr–C and Zr–N bonding states in the deposited layers.

The photoelectron spectrum of C1s [Fig. 2(b)] in the amorphous layer deposited at 1373 K showed two peaks. The peak located at 282.2 eV was assigned to zirconium carbide (C–Zr), while the peak at 285.1 eV could be associated to oxidized carbon species (C–O) and/or C–N sp2 bonds.\(^{8},^{37},^{38}\) The photoelectron spectrum of C1s spectrum of in the fcc-ZrCN layer was deconvoluted into three peaks; compared with the amorphous layer deposited at 1373 K, the additional peak located at 284.2 eV was attributed to C–C bondings.\(^{4},^{38}\) The elemental composition of the fcc-ZrCN layer deposited at 1453 K, as estimated by XPS, was Zr:C:N:O = 1.00:2.13:0.15:0.22, implying that free carbon was co-precipitated with ZrCN during the vapor-phase growth.

In Fig. 2(c), the photoelectron spectra of N1s in layers prepared at 1373 and 1453 K showed a single peak located at 396.7 ± 0.2 eV, which is typical for nitrides (N–Zr).\(^{36},^{40}\) There was possibility that the N1s spectra could be deconvoluted into two curves attributed to nitrides and...
The intensity of the photoelectron peak of O1s in the fcc-ZrCN layer deposited at 1453 K was significantly lower than that in the amorphous layer deposited at 1373 K, as shown in Fig. 2(d). The photoelectron spectra of O1s were deconvoluted into two curves; the peak at lower binding energy located at 530.5 ± 0.3 eV was attributed to the Zr–O bonding, while that at higher binding energy at 531.6 ± 0.2 eV could be associated with the existence of O–C (531.5 eV)\(^{30}\) and O–N (531.3 eV) configurations.\(^ {36}\) The elemental composition of the amorphous layer deposited at 1373 K was Zr:C:N:O = 1.00:168:0.10:1.06. The amorphous layer contained a large amount of oxygen and comprised mainly the oxide forms with less amounts of carbides (Zr–C) and nitrides (Zr–N) configurations. At deposition temperatures above 1453 K, crystalline fcc-ZrCN layers were formed with a lower content of O impurity, which might be due to the reduction by hydrogen and hydrocarbons resulting from the decomposition of the precursor having alkyl groups.

**Figure 3** depicts surface and cross-sectional SEM images of ZrCN layers deposited by laser CVD at temperatures of (a–c) 1373 and (d–f) 1453 K. Images (b) and (c) are magnified views of (a) and (d), respectively, and (c) and (f) are fracture cross sections corresponding to (a) and (d), respectively.

Dispersed (395.9 eV).\(^ {36}\) The intensity of the photoelectron peak of O1s in the fcc-ZrCN layer deposited at 1453 K was significantly lower than that in the amorphous layer deposited at 1373 K, as shown in Fig. 2(d). The photoelectron spectra of O1s were deconvoluted into two curves; the peak at lower binding energy located at 530.5 ± 0.3 eV was attributed to the Zr–O bonding, while that at higher binding energy at 531.6 ± 0.2 eV could be associated with the existence of O–C (531.5 eV)\(^ {30}\) and O–N (531.3 eV) configurations.\(^ {36}\) The elemental composition of the amorphous layer deposited at 1373 K was Zr:C:N:O = 1.00:168:0.10:1.06. The amorphous layer contained a large amount of oxygen and comprised mainly the oxide forms with less amounts of carbides (Zr–C) and nitrides (Zr–N) configurations. At deposition temperatures above 1453 K, crystalline fcc-ZrCN layers were formed with a lower content of O impurity, which might be due to the reduction by hydrogen and hydrocarbons resulting from the decomposition of the precursor having alkyl groups.

**Figure 3** depicts surface and cross-sectional SEM images of ZrCN layers. At 1373 K, the amorphous layer had a cone-like morphology with cone sizes of several micrometers, where each cone consisted of nodular grains with sizes of several tens and hundreds of nanometers, as shown in Figs. 3(a) and 3(b). Cracks were formed mainly along the gaps between the cones [Fig. 3(b)], whereas the fracture cross section had the apparently dense morphology [Fig. 3(c)]. The cracks were resulted from the residual stress by the significant difference in thermal coefficients between the layer containing a large amount of oxides and the carbon substrate. Although the surface morphologies of the crystalline fcc-ZrCN layers were similar to the nodular morphology with a cone-like structure observed for the amorphous layer at 1373 K, no cracks propagating in the cones and along the gaps of cones were observed in fcc-ZrCN layers [Figs. 3(d) and 3(e)]. In the cross sections, the thicknesses of crystalline fcc-ZrCN layers were 6.6 μm (deposition rate: 40 μm h\(^{-1}\)) at 1453 K, whereas the thickness of the amorphous layer formed at 1373 K was 35 μm (deposition rate: 210 μm h\(^{-1}\)).

**Figure 4** shows SEM images of the cross section, polished by Ar ion milling, of the fcc-ZrCN layer deposited at 1453 K. Here, a thin layer with a dark contrast was formed on the top surface, which would be due to the redeposition of the sputtered ZrCN by the Ar ion milling. The polished cross-sectional image revealed the formation of a dense and uniform layer with a few closed pores having sizes of several tens of nanometers [Fig. 4(a)]. A slight contrast was observed in the grains; relatively dark areas with sizes less than several tens of nanometers [designated by white arrows in Fig. 4(b)] were finely dispersed in gray-contrast grains. The small dark areas are considered to be free carbon that is simultaneously precipitated with the main fcc-ZrC phase.

**Figures 5(a) and 5(b)** depicts a high angle annular dark field (HAADF) image and the corresponding selected-area electron diffraction (SAED) pattern of the fcc-ZrC layer deposited at 1453 K. A dendritic microstructure compris-
Fig. 4. Cross-sectional SEM images of ZrCN layers polished by Ar ion milling at a deposition temperature of 1453 K; (b) is a magnified view of (a).

Fig. 5. STEM images and SAED pattern of the ZrCN layer prepared at a deposition temperature of 1453 K; a HAADF image at a low magnification (a), an SEAD pattern (b), a pair of HAADF (c) and BF (d) images at a high magnification, and HAADF images at low (e) and high (f) magnifications.
of C3H6 is prior to that of ZrC. In the present study, the morphology as same as pyrolytic carbon, because nano-sized ZrC particles embedded in the carbon matrix was.

The SAED pattern [Fig. 5(b)], the concentric rings corresponded to (111), (200), (220), and (311) planes of a fcc-ZrCN layer [Fig. 5(a)], which was consistent with cross-sectional SEM images as shown in Fig. 4. In the SAED pattern, the contrast of fcc-ZrCN and carbon were vice-versa in the HAADF and bright-field (BF) images at a higher magnification. A pair of HAADF and bright field images (BF) at a higher magnification are depicted in Figs. 5(c) and 5(d). In Fig. 5(c), grain sizes of the fcc-ZrCN phase with a gray (bright) contrast was less than 10 nm, while dark-contrast carbon was finely dispersed in the nano-sized fcc-ZrCN grains; the contrast of fcc-ZrCN and carbon were vice-versa in the BF image [Fig. 5(d)]. Sun et al. synthesized ZrC nanoparticles reinforced carbon (ZrC/C) composite coatings by CVD using ZrCl4-C3H8-H2 source with an Ar carrier gas. They reported that Zr/C microstructure comprised nano-sized ZrC particles embedded in the carbon matrix with the morphology as same as pyrolytic carbon, because the nucleation of pyrolytic carbon by the decomposition of C3H6 is prior to that of ZrC. In the present study, the decomposition of the Zr(NEtMe)4 single source simultaneously contributed to the nucleation of both fcc-ZrCN and carbon, resulted in the formation of the dendritic microstructure comprising nano-sized fcc-ZrCN grains with finely dispersed carbon. HAADF images taken at the interface between the ZrCN layer and the substrate (carbon) are depicted in Figs. 5(e) and 5(f). Near the interface, there were a bright-contrast thin region (200–300 nm) adjacent to the substrate and slightly dark-contrast region approximately 1 μm thick. This feature at the interface was also observed in the SEM image [Fig. 4(a)]. At the initial stage of the deposition, the laser was irradiated directly to the substrate. As the deposit (layer) were grown and became thick, the laser energy was partially or primarily absorbed by the deposited layer. The change in characteristics of the deposition surface, such as optical absorbance and thermal conductivity, and the interaction with the laser would cause the deviation on the behavior of vapor-phase growth. At the initial stage, the deposition at a Zr-rich composition would occur on the substrate surface, corresponding to the bright-contrast thin region; then, the deposition shifted to the slightly carbon-rich composition corresponding to the dark-contrast region. As the deposited layer became thick, the layer was grown at a steady state, forming the microstructure with the chemical composition and bonding states as shown in Fig. 5(a) and Fig. 2.

**Table 2.** Deposition of ZrCN and ZrN films by CVD techniques using alkyl-amide precursors in the literature

| Precursor | Gases | Deposition temperature | Phase | Substrate | Deposition rates (μm h⁻¹) | Reference |
|-----------|-------|------------------------|-------|-----------|---------------------------|-----------|
| Zr(NEt2)4 and HNEt2 | H2, N2, Ar | 823–1023 K | fcc-Zr(C,N) | Fe | N.A. | Mathur et al. 2004²² |
| Zr(NMe2)4 | H2, N2, Ar | 523–648 K | ZrN | p-type Si(100)/SiO2 | ~3 | Kim et al. 2004¹⁹ |
| [Zr(η⁵-(PrN)2CNMe2)]₂[NMe2]₂ | N2 | 673–873 K | amorphous ZrN | p-type Si(100) | N.A. | Banerjee et al. 2012¹² |
| [Zr(NMe2)3]₂[NMe2]₂ | NH3 | 873–1073 K | fcc-ZrN | p-type Si(100) | N.A. | Banerjee et al. 2012¹² |
| Plasma CVD | Zr(NEtMe)4 and Zr(NEt2)4 | H2, N2, Ar | 573–673 K | fcc-ZrCN | Fe | ~1 | Berndt et al. 1995³⁰ |
| Zr(NEt2)4 | H2, N2, NH3 | ~673 K | ZrCN | glass, quartz, Si, Al, steel | ~1.2 | Wendel et al. 1992⁴¹ |
| Laser CVD | Zr(NEtMe)4 | Ar | 1373 K | amorphous carbon | 210 | This study |
| Zr(NEtMe)4 | Ar | 1453 K | fcc-ZrCN with carbon | 40 | This study |

**Table 2** summarizes some studies, including the present one, on the deposition of ZrC and ZrN films by CVD techniques using alkyl-amide precursors. By thermal CVD, Mathur et al. deposited zirconium carbonitride (fcc-ZrCN) films on Fe substrates by using Zr(NEt2)4 and HNEt2 as precursors at a deposition temperature of 823 K.²² They reported that the formation of pure ZrC was relatively less probable because of the rather low deposition temperature. Kim et al. reported the deposition of ZrN films on Si (100) substrates by using Zr(NMe2)4 at deposition temperatures of 523–648 K; they characterized the CVD-ZrN films mainly by Fourier transform infrared spectroscopy, although it is unclear whether the deposited ZrN films were crystalline.¹⁹ Banerjee et al. suggested alkyl-amide precursors (heteroleptic Zr amide-guanidinate complexes) for the deposition of ZrN.²³ Plasma CVD using alkyl-amide precursors has been also employed for the growth of zirconium carbonitride films.²⁹,⁴¹ The deposition temperatures for the vapor-phase growth of ZrCN films by plasma CVD were in the range of 573–673 K, which is lower than the range for thermal CVD; however, the deposition rates were not high (around 1 μm h⁻¹).²²,⁴¹ In the present study, high-temperature deposition by CVD using a high-power infrared laser resulted in the formation of thick ZrC layers with a solid solution of N and excess carbon. The deposition rates were 210 μm h⁻¹ for the amorphous layer and 40 μm h⁻¹ for fcc-ZrCN layers,
which were more than an order of magnitude higher, compared to those reported in the literature. In the present laser CVD scheme, the substrate surface was locally heated by irradiation with the near-infrared high-power laser beam at the wave length of 1068 nm. Because the nozzle temperature was maintained at 373 K, the incoming precursor gas molecules were maintained at relatively low temperatures to reach the high-temperature reaction field of the substrate surface, avoiding neither decompositions nor homogeneous reaction in the gas phase. Though the effects of interactions between the laser beam and precursors as well as chemical reactions on the vapor-phase growth behavior of ZrCN are unclear, the locally activated field created by the laser irradiation on the substrate surface precursor molecules enables the high-speed growth of fcc-ZrCN.

4. Conclusions

The vapor growth of ZrCN layers was demonstrated by a laser CVD technique using an alkyl-amide precursor as a single source. An amorphous layer was deposited at a deposition temperature of 1373 K, whereas the deposition temperature of 1453 K resulted in the formation of a crystalline fcc-ZrCN phase. The XPS results confirmed the bonding states of Zr–C and Zr–N and suggested an excess amount of free carbon in the fcc-ZrCN layer formed at 1453 K. The fcc-ZrCN layers had cone-like nodular morphologies with dense and conformal cross sections, where the dendritic microstructure comprising nano-sized ZrCN grains with finely dispersed carbon was formed. The deposition rates for the fcc-ZrCN growth were 40 μm h⁻¹, which were more than an order magnitude higher, compared to those reported for films by CVD techniques using alkyl-amide precursors in the literature. The present study achieved the high-speed growth of crystalline ZrCN layers by a CVD technique using a single source and revealed nanostucture and detailed chemical compositions of the fcc-ZrCN layers.

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