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Review

Mini Review: Crystal growth from liquid and vapor phases

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Abstract: Crystalline materials at both the macro- and nanoscale have attracted growing interest due to their competitive advantages in cutting-edge applications. This literature review examines two representative crystal growth techniques—the heat exchange method and chemical vapor deposition—along with their prominent applications: high-quality bulk crystalline silicon and transition metal dichalcogenide (TMDs) crystalline nanofilms. The review provides an overview of the general mechanisms, the developmental trajectories of each method, their current status, and future prospects, with the aim of understanding the associated challenges, limitations, and emerging opportunities.

1. Introduction

Large-scale artificial crystallization processes rely on removing heat from liquid-phase substances, a principle underlying most major crystal growth methods, such as the Bridgman–Stockbarger method, the Czochralski technique, and the heat exchange method. The Bridgman–Stockbarger method employs a temperature-gradient furnace and a moving crucible, extracting heat as the crucible travels from a high-temperature zone to a low-temperature zone. The Czochralski technique uses a seed crystal dipped into the melt, with heat being removed from the molten material into the environment and the seed. By contrast, the heat exchange method employs a heat exchanger to absorb heat from the melt and enable a controllable crystal growth process. Although the Czochralski technique has been broadly applied in the manufacture of semiconductor materials such as silicon and gallium arsenide, the heat exchange method (HEM) is regarded as a compelling alternative thanks to its advantages in eliminating crystal defects arising from mechanical perturbation, thermal stress, and non-uniform heat distribution. Nevertheless, major challenges hindering its further development center on the adhesion phenomenon in specific materials and the advancement of crystal growth kinetic theory.

Building chemical bonds by suppressing molecular kinetics from high-energy phases underpins most nanoscale artificial crystal synthesis techniques, including physical vapor deposition (PVD), chemical vapor deposition (CVD), and atomic layer deposition (ALD), which share a kinetic commonality with large-scale crystallization processes. With the rapid evolution of sophisticated applications—such as ultra-high-quality semiconductors, nanoscale sensing, and biomedical surface modification—interest has grown substantially in critical candidate materials, including graphene, gallium nitride, nanophase metals and alloys, and transition metal dichalcogenides. A critical challenge arises from the inherent variability of nanomaterials, which can compromise excellent performance in advanced applications and poses a significant barrier to their practical deployment.

This literature review focuses on the HEM process for high-quality bulk crystal manufacturing and CVD for crystalline nanofilm synthesis, with particular interest in their applications in semiconductors, photovoltaics, and sensing. Specifically, the review will concentrate on high-quality bulk monocrystalline sapphire and silicon produced by the HEM method, and on transition metal dichalcogenide (TMD) crystalline monolayer thin films synthesized via the CVD technique, to investigate their growth mechanisms, limitations, and potential applications. An overview of the general mechanisms, developmental trajectories, current status, and future prospects will be included, aiming to elucidate the challenges, limitations, and emerging opportunities.

HEM and CVD, as two representative methods for large-scale and nanoscale crystallization, exhibit similar crystal-forming behaviors that give rise to commonalities in both thermodynamics and reaction kinetics, in turn influencing material properties, application domains, and growth processes. Consequently, a methodology that investigates these two methods in parallel serves as a cohesive framework to closely connect them in both theoretical research and applied science development, offering the potential for substantial economic benefit.

2. Mechanism and Materials

Heat exchange method was firstly proposed by D. Viechnicki and F. Schmid in 1969 for high quality sapphire manufacture, then was used to manufacture Silicon in 1970s. Because of its inherent superiority in mechanism, HEM was considered as an ideal process for manufacturing high quality bulk crystal such as silicon and sapphire. This method uses a heat exchanger to absorb the heat from melted materials, promotes a controllable solidification and crystal growth process. As the heat exchanger involved in, the solidification process become to a turntable process by changing the cooling power of heat exchanger, which brought the advantages including eliminating crystal defects from mechanical perturbation, thermal stress, and heat distribution. Furthermore, the method can be straightforwardly engineered and applied to nearly all materials by change heat exchanger parameter, cooling media, and crucible materials. A typical Sapphire HEM furnace, which was depicted schematically in **Figure 1** originally created by Joyce *et al.*,[1]consists of heating elements, thermal insulated furnace chamber, cooling systems derived heat exchanger, and observation system.

A crucible set on the top of heat exchanger; seed materials and start materials were placed within in the crucible. The seed was made by monocrystalline material and placed bottom center of crucible, which was directly upon the heat exchanger. The starting material was made by amorphous or polycrystalline, placed on both top and surrounding of seed material. The original HEM process was reproduced in **Figure 2** originally created by

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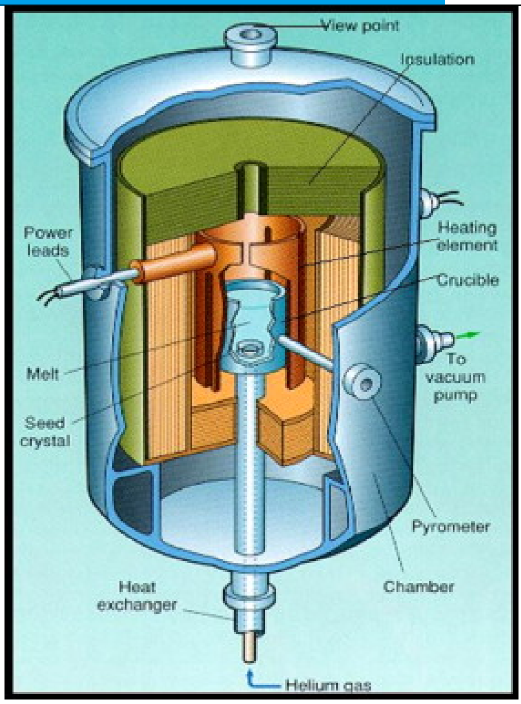

Figure 1. Schematic of a typical HEM furnace for Sapphire manufacture, consists of heating elements, thermal insulated furnace chamber, helium gas derived heat exchanger, and pyrometer and viewpoint-based observation system.

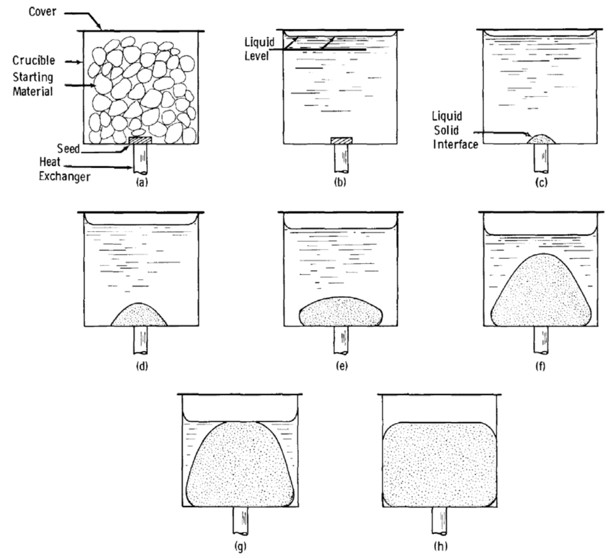

Figure 2. A typical HEM Process; a. status of crucible, cover, starting material, and seed prior to melting; b. starting material melted; c. seed partially melted for nucleation process; d. first stage of growth; e. second stage of growth covered bottom; f. third stage of interface expanding; g. fourth stage of growth liquid solid interface breaks liquid surface; h. crystal growth completed.

Viechnicki *et al.*,[2] the starting material was melting initially, the seed material was served as a nucleation core; heat was transferred from melted material to nucleation core accompanied by a crystalline process from nucleation core to surroundings. The numerical computation based on Leibniz–Reynolds transport theorem has been employed to investigate the crystalline process for vary kinds of materials such as silicon and sapphire. General continuous equations can be written in Equation 1, in which dV is volume element, dA is surface element, f is transport function, $v_b=v_b(x, t)$ is velocity of area element, n is the unit vector. In recent twenty year, the finite element method and high-performance computation bring it to the next level.

$$\frac{d}{dt}\int_{\Omega(t)} f\,dV=\int_{\Omega(t)}\frac{\partial f}{\partial t}\,dV+\int_{\Omega(t)}(v_b \bullet n)\,f\,dV \qquad (1)$$

Eqn. 1. General continuous equations for Leibniz–Reynolds transport theorem

In addition to Sapphire and Silicon, High-melting sesquioxides (such as Sc2O3, Y2O3, and Lu2O3), Neighborite (NaMgF3), NBW(NaBiW2O8), NBP(Na5B2P3O13), Bismuth germanate, and Cadmium telluride, have been produced by HEM process.[3-8] The **table 1** detailed listed materials can be growth by HEM method, and their supplementary equipment such as crucible and furnace.

CVD as a one of majority thin film and nanoscale crystalline synthesis method has a similar mechanism in thermodynamics and reaction kinetics with HEM. Specifically, similar mechanism applied in both heat up and crystalline process. The majority deference between two methods concentrated on the phase of materials. The HEM involved in a heating process from solid phase to liquid phase and a crystalline process from liquid phase. The engineering issues concentrated on the methods to calculating the latent heat and balancing the growth and evaporation under specific pressures. As well, the CVD involved in an evaporating process from solid phase to gas phase, a crystalline process from gas phase to solid, and a reaction involved two reactors (precursors). In addition, the reaction kinetics between precursor would be considered in all CVD process, as well would be considered in some specific HEM process of which growth material reacts with crucible materials such as the pair of silicon and quartz. Because of the similar growth mechanism, the computational analysis tool for HEM can be shared to CVD, such as the Leibniz–Reynolds transport theorem, therefore brings economics benefit as above mentioned.

Compared to HEM's heating-growth two-step process, a typical thermal CVD has a slightly complex process including heating up, reaction, and growth. Specifically, a typical CVD process can be separated into three main stages; in first stage, gas phase reactant was vehicle to reactor by carrier gas and mix with precursor; in second stage, reactant react with precursor and forming products and by-products; in third stage, products deposited on the substrate forming the thin film and by-products vehicle out to the reactor by carrier gas. The process of deposition on substrate was considered as a crystalline process from gas phase directly. A typical thermal CVD system, which was depicted schematically in **Figure 3** originally created by *Sun et al.*,[9] consists of gas delivery and vacuum systems, reaction furnace, energy source, an exhaust system, and control system.

Theoretically, CVD has less limitations in reactors and precursors than other nano crystalline methods; as well provided tunable ability to produces which significantly increased capacity in application. Therefore, most materials involved in surface reactions can be synthesized using CVD technique, such as silicon, sapphire, graphene, diamonds, TMD materials (such as molybdenum disulfide and tungsten disulfide), and polymers (such as PEDOT and Ppy). Their application covered nearly all research hotspot across from quantum computation to medical diagnosis. For example, carbon-based nanomaterials,

Table 1. Materials can be growth by HEM method

Materials	Crucibles	Protect gas	Reference
Bismuth germanate ($Bi_4Ge_3O_{12}$, BGO)	Platinum	vacuum	Schmid et al. [3]
Cadmium telluride (CdTe)	Hexane	Argon	Baars et al.[4]
Sodium–bismuth tungstate ($NaBiW_2O_8$; NBW)	unknown	vacuum	Rahab et al.[5]
Sodium–phosphate ($Na_5B_2P_3O_{13}$; NBP)	unknown	vacuum	Rahab et al.[5]
Yb-doped Lu2O3 (Yb:Lu_2O_3)	Rhenium	N_2:H_2 (0.9:0.1)	Peters et al.[6]
Neighborite ($NaMgF_3$)	unknown	vacuum	Oçafrain et al.[7]
Sesquioxide of yttrium family	Rhenium	N_2:H_2	Peters et al.[8]

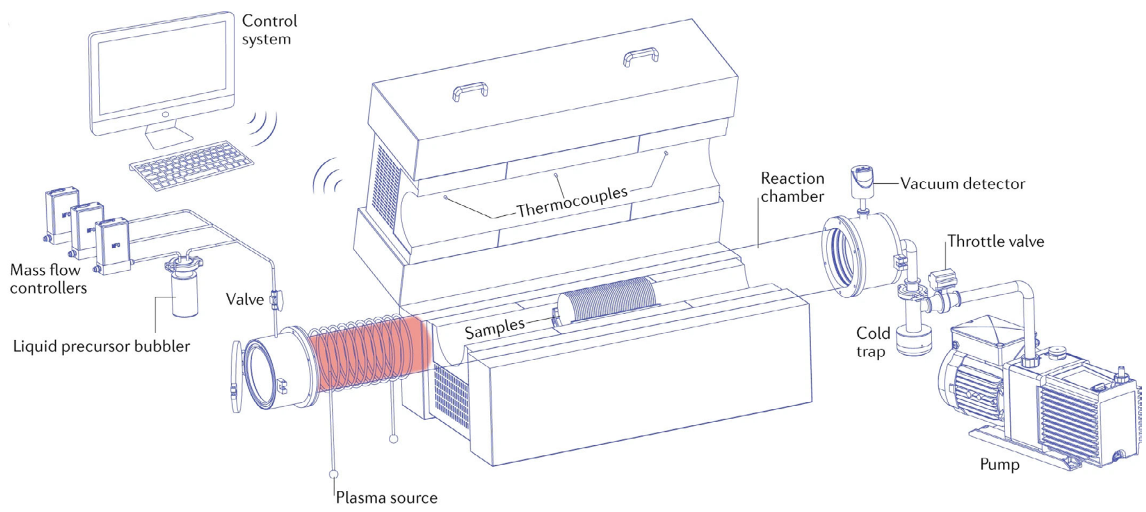

Figure 3. Schematic diagram of a typical thermal CVD system, gas delivery and vacuum systems, reaction furnace, energy source, an exhaust system, and control system.

such as diamond, graphene, and carbon nanotube, can be synthesized using CVD with precursors combination of alkenyl group and hydrogen. Further decreased manufacture pressure from super high (>5 GPa) to moderate pressure (<0.01 GPa) and manufacture temperature from over 1500°C to 900°C for diamond; as well the tunable ability reflected in the potential to produce graphene and carbon nanotube using same equipment with minus change in parameters.

Among those materials, TMDs was considered as one kind of most competitive candidates in several novel applications because of their physical properties such as direct band gap, heterostructures, and strong spin–orbit coupling. Those excellence properties promised their potential in high performance electronic components, photonic and optical and electrochemical sensing; their application includes flexible/transparent field-effect transistors, biosensors, color sensor, wavelength sensor, specific gas sensor. Molybdenum disulfide (MoS2) and tungsten disulfide (WS2) nanocrystals have been proved capability as photodetector and transistors. For example, *Cheng et al.* integrated MoS2 nano-transistors on quartz and flexible substrates to achieve a highly performance self-aligned device, *Shokri et al.* investigated electronic and transport properties of simple gas molecules such as CO and NO in monolayer MoS2 and theoretically revealed its potential as gas sensors, *Bilgin et al.* using CVD synthesized monolayer optoelectronic-grade molybdenum disulfide, and their follow-up research by *Hejazi et al.* proved the capability in precise wavelength measurement.[10-13] Moreover, Molybdenum diselenide (MoSe2) has been proved the identical potential in similar applications. Owing to its higher electrical conductivity from Se, MoSe2 has an even broader application in energy storage, photoelectronic, and electrocatalysis.[14] For example, *Choi et al.* discovered MoSe2 embedded CNT ball has ability in sodium-ion storage with excellence stability and reversible feature, which provided potential in battery applications; *Tang et al.* reported the hydrogen evolution reaction activity of MoSe2, which provided possibility to become electrocatalysis; Du *et al.* investigated the optical absorbance of monolayer MoSe2 in both computational and practical and provided theoretical support for photoelectronic.[15-17]

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Conflicts of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potenti al conflict of interest.

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