HIGH PRESSURE APPARATUS AND METHOD FOR NITRIDE CRYSTAL GROWTH

CROSS-REFERENCE TO RELATED APPLICATION

The present application is a continuation-in-part application claiming priority to U.S. patent application Ser. No. 12/133,364, filed Jun. 5, 2008, commonly owned and hereby incorporated by reference for all purposes.

BACKGROUND OF THE INVENTION

The present invention relates generally to techniques for processing materials in supercritical fluids. More specifically, embodiments of the invention include techniques for controlling parameters associated with a material processing capsule disposed within a high-pressure apparatus enclosure. The invention can be applied to growing crystals of GaN, AlN, InN, InGaN, AlGaN, and AlInGaN, and others for manufacture of bulk or patterned substrates. Such bulk or patterned substrates can be used for a variety of applications including optoelectronic devices, lasers, light emitting diodes, solar cells, photoelectrochemical water splitting and hydrogen generation, photodetectors, integrated circuits, and transistors, among other devices.

Supercritical fluids are used to process a wide variety of materials. A supercritical fluid is often defined as a substance beyond its critical point, i.e., critical temperature and critical pressure. A critical point represents the highest temperature and pressure at which the substance can exist as a vapor and liquid in equilibrium. In certain supercritical fluid applications, the materials being processed are placed inside a pressure vessel or other high pressure apparatus. In some cases it is desirable to first place the materials inside a container, liner, or capsule, which in turn is placed inside the high pressure apparatus. In operation, the high pressure apparatus provides structural support for the high pressures generated within the container or capsule holding the materials. The container, liner, or capsule provides a closed/sealed environment that is chemically inert and impermeable to solvents, solutes, and gases that may be involved in or generated by the process.

Scientists and engineers have been synthesizing crystalline materials using high pressure techniques. As an example, synthetic diamonds are often made using high pressure and temperature conditions. Synthetic diamonds are often used for industrial purposes but can also be grown large enough for jewelry and other applications. Scientists and engineers also use high pressure to synthesize complex materials such as zeolites, which can be used to filter toxins and the like. Moreover, geologists have also used high pressure techniques to simulate conditions and/or processes occurring deep within the earth's crust. High pressure techniques often rely upon supercritical fluids, herein referred to as SCFs.

Supercritical fluids provide an especially ideal environment for growth of high quality crystals in large volumes and low costs. In many cases, supercritical fluids possess the solvating capabilities of a liquid with the transport characteristics of a gas. Thus, on the one hand, supercritical fluids can dissolve significant quantities of a solute for recrystallization. On the other hand, the favorable transport characteristics include a high diffusion coefficient, so that solutes may be transported rapidly through the boundary layer between the bulk of the supercritical fluid and a growing crystal, and also a low viscosity, so that the boundary layer is very thin and small temperature gradients can cause facile self-convection and self-stirring of the reactor. This combination of characteristics enables, for example, the growth of hundreds or thousands of large a-quartz crystals in a single growth run in supercritical water.

Supercritical fluids also provide an attractive medium for synthesis of exotic materials, such as zeolites, for solvent extractions, as of caffeine from coffee, and for decomposition and/or dissolution of materials that are relatively inert under more typical conditions, such as biofuels and toxic waste materials.

In some applications, such as crystal growth, the pressure vessel or capsule also includes a baffle plate that separates the interior into different chambers, e.g., a top half and a bottom half. The baffle plate typically has a plurality of random or regularly spaced holes to enable fluid flow and heat and mass transfer between these different chambers, which hold the different materials being processed along with a supercritical fluid. For example, in typical crystal growth applications, one portion of the capsule contains seed crystals and the other half contains nutrient material. In addition to the materials being processed, the capsule contains a solid or liquid that forms the supercritical fluid at elevated temperatures and pressures and, typically, also a mineralizer to increase the solubility of the materials being processed in the supercritical fluid. In other applications, for example, synthesis of zeolites or of nanoparticles or processing of ceramics, no baffle plate may be used for operation. In operation, the capsule is heated and pressurized toward or beyond the critical point, thereby causing the solid and/or liquid to transform into the supercritical fluid. In some applications the fluid may remain subcritical, that is, the pressure or temperature may be less than the critical point. However, in all cases of interest here, the fluid is superheated, that is, the temperature is higher than the boiling point of the fluid at atmospheric pressure. The term "supercritical" will be used throughout to mean "superheated," regardless of whether the pressure and temperature are greater than the critical point, which may not be known for a particular fluid composition with dissolved solutes.

Although somewhat effective for conventional crystal growth, drawbacks exist with conventional processing vessels. As an example, processing capabilities for conventional steel hot-wall pressure vessels (e.g., autoclaves) are typically limited to a maximum temperature of about 400 Degrees Celsius and a maximum pressure of 0.2 GigaPascals (GPa). Fabrication of conventional pressure vessels from nickel-based superalloys allows for operation at a maximum temperature of about 550 degrees Celsius and a maximum pressure of about 0.5 GPa. Therefore, these conventional hot-wall pressure vessels are often inadequate for some processes, such as the growth of gallium nitride crystals in supercritical ammonia that often require pressures and temperatures that extend significantly above this range in order to achieve growth rates above about 2-4 microns per hour. In addition, nickel-based superalloys are very expensive and are difficult to machine, limiting the maximum practical size and greatly increasing the cost compared to traditional steel pressure vessels.

Attempts have been made to overcome the drawbacks of conventional pressure vessels. D'Evelyn et al., US patent application 2003/0140845A1, indicates a so-called zero-stroke high pressure apparatus adapted from the type of belt apparatus used for synthesis of diamond using high pressure and high temperature. Cemented tungsten carbide, however, is used as the die material, which is relatively expensive and is difficult to manufacture in large dimensions. In addition, the use of a hydraulic press to contain the apparatus increases the cost and further limits the maximum volume. Finally, the use of a pressure transmission medium surrounding the cap-