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Production Of Nanosize Particles by Gas Phase Synthesis Using MOCVD

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A system for the production of nanoparticles incorporating MOCVD has been assembled at the National High Magnetic Fields Laboratory. The system comprises: 1. a microwave plasma deposition system utilizing a powder delivery system; 2. a supercritical fluid system; and 3. three vaporizers, one of which includes a liquid delivery system. The use of the plasma system will aid in the development of novel composite materials. The supercritical fluid system will be used to make nanoparticles from metalorganic compounds or noble metals. The different vaporizer systems will make it possible to use volatile solid or liquid precursors for deposition onto the surface of the nanoparticles. This coating technique is aimed at producing composite and/or magnetoresistive materials. Evaluation of the manual system controls are currently being accomplished. Computer control designs are well underway and being tested. Any information gathered during the manual control testing will be implemented into the computer control design.

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Summary of Project Results

Procurement, Assembly, and Initial Testing

The purpose of this project was to construct a combination thin film technique system modified to produce large amounts of nanosize materials in a reproducible manner. Assembly of the first phase of the nanoparticle system has been accomplished (Figure 1). There were numerous delays in acquiring both the parts and the laboratory space for assembling the system. Of major concern were safety issues related to the operation of the system at the National High Magnetic Fields Laboratory. These issues greatly delayed installation of the cabinet for housing the mechanical parts of the system.

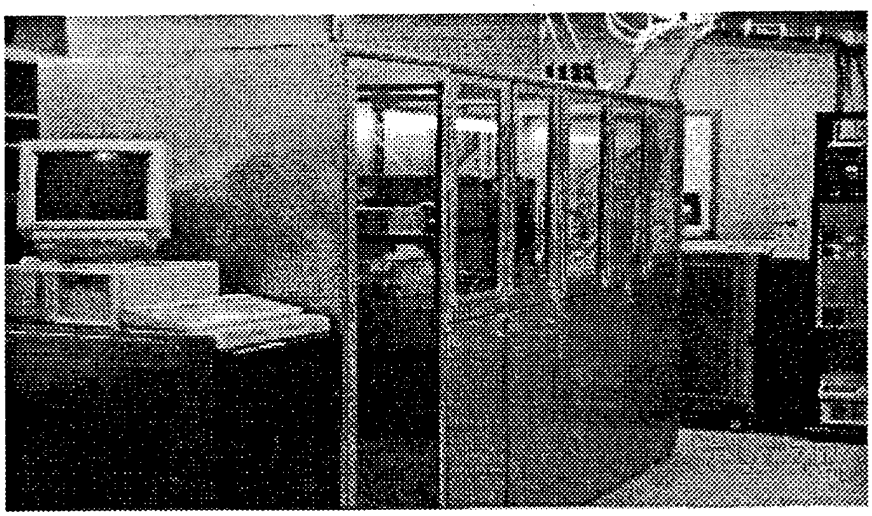

Figure 1: View of nanoparticle system as installed at the National High Magnetic Fields Laboratory.

Evaluations of the manual system controls are currently being accomplished. Any information gathered during this testing will be implemented into the computer control design. The computer control designs are well underway and being tested. An ultrasonic atomizer manufactured by Lechler, Inc. and an ionization chamber have been incorporated into the system.

System Description

The System consists of three vaporizers (manufactured in-house) one of which utilizes a liquid delivery system. These vaporizer systems make it possible to use volatile solid or liquid precursors for deposition of various metal or metal compounds onto the surface of the nanoparticles. The liquid delivery system utilizes a SciLog fluid metering pump with computer interface capability.

For production of the nanoparticles, a supercritical fluid system is incorporated. The system is manufactured by ISCO, Inc and includes a SFX 2-10 extractor and a 100DX series syringe pump. The supercritical fluid system gives the system the capability of producing nanoparticles from organometallic compounds or noble metals. The supercritical fluid pump is controlled by an ISCO series D pump controller and can be computer interfaced via a RS232 port.

A three-inch diameter stainless steel reactor tube heated by a Lindberg/Blue 5000-watt tube furnace will be utilized in the MOCVD process of the various nanoparticles. The furnace is capable of a maximum temperature of 1700 °C and is controlled by a Linberg/Blue programmable controller. The controller includes a RS232 data port for data transfer and computer control.

The nanoparticles will be collected in cold traps, of which are two stainless steel and one glass. Only two of the cold traps will be utilized at any one given time. The glass trap will be used in visual inspection for achieving optimum settings. One of the stainless steel traps will be later adapted to house a cascade impactor. The impactor will be used to separate the larger nanoclusters. The impactor to be used is manufactured by Anderson Samplers Incorporated and is on loan from Engineering Technologies, Inc., Orlando, Florida. The instrument is a multistage impactor and automatically separates the particles into eight fractions from 10.0 microns down to 400 nanometers in diameter.

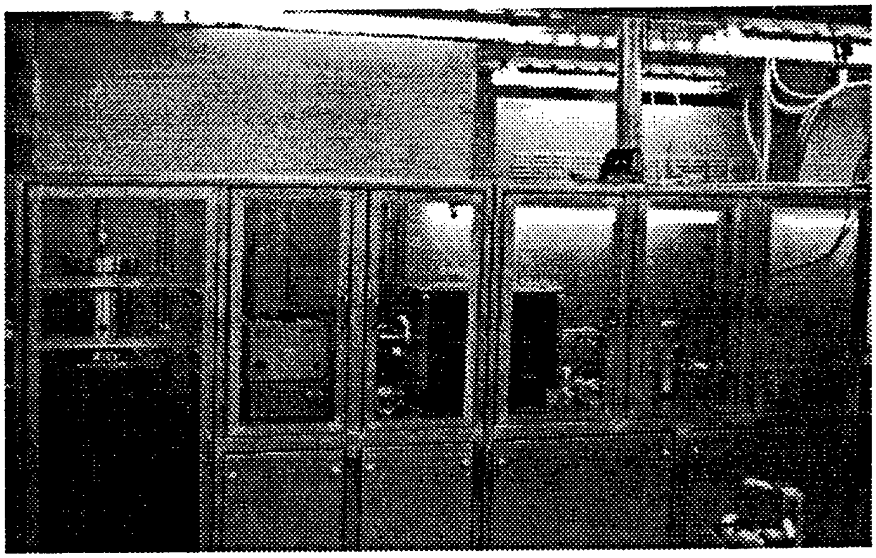

Figure 2: View of nanoparticle system, from left to right, through cabinet doors respectively: ionization (plasma) chamber, supercritical fluid system, reactor tube in furnace and vaporizers (lower corners two doors), bellows and throttle valves.

A pneumatic high vacuum stainless steel in-line bellows sealed valve will be used to seal off the vacuum when removing the nanoparticles. Key High Vacuum Products, Inc. manufacture the valve installed and will be manual and/or computer controlled.

For vacuum measurements MKS Baratron's are installed. These are a type 615A high accuracy sensor head for high temperature areas and a type 626A absolute pressure transducer for cooler areas. Pressure display along with signal conditioning will be maintained by a MKS type 270D high accuracy signal conditioner. For sensor selection a MKS type 274 three-channel sensor selector is used.

Vacuum pressure is controlled and regulated by a MKS type 651C pressure controller and a MKS type 653B exhaust throttle valve. All MKS instruments will be computer interfaced for monitoring and control.

Gas flows are measured and controlled by Brooks model 5850E flow controllers. Setpoints will be controlled and maintained by a Brooks model 0154E microprocessor-based control and read out unit. The computer through communication with the control unit will also control the mass flow controllers.

All organic and inorganic materials will be scrubbed out of the system to prevent vacuum pump and atmospheric contamination. The scrubber utilizes FCA activated carbon, manufactured by Calgon Carbon Corporation. The carbon is impregnated with copper and chromium and incased in a stainless steel vacuum tube. Heat will be applied to the activated carbon and vacuum tube by means of an Omega vacuum-formed ceramic fiber radiant heater. Temperatures for the scrubbing operation will be maintained at over 1000 °C.

Vacuum is maintained by a Trivac high-vacuum pump manufactured by Lebold Vacuum Products, Inc. The pump displaces 13.4 cubic feet per minute with an ultimate pressure of 1×10^{-4} torr. Operation and temperature monitoring will be handled via computer.

The system is housed in a 120 x 71 x 40 inch stainless steel cabinet. The cabinet was manufactured by Kelly Brothers Sheet Metal, Inc. Tallahassee, Florida and incorporates solid stainless steel removable panels on the lower section and hinged stainless steel framed doors with Plexiglas inserts on the front upper section. The cabinet is connected to in-house airflow for a negative pressure atmosphere.

All vaporizers along with the heated reactant and purge lines are monitored and controlled trough the use of Omega type K surface mounted thermocouples. Temperature set points are controlled and maintained with the use of Omega CN76000 microprocessor-based temperature/process controllers and solid state relays. The controllers will be computer interfaced via RS-485 communications.

A MOCVD system using microwave plasma with powder feed unit will be installed. The microwave plasma unit is the property of Dr. Dahmen. The purpose of this system is to generate powders for use in the CVD process to develop novel composite materials. The main components have been installed and will be completed after the nanoparticle system tests have been completed.

Computer Control

Computer control will be implemented by an IBM PC/AT. Hardware to be utilized includes: an AT-MIO-16E-2 multifunction analog and digital I/O with NI-DAQ DOS/ Windows driver software; from National Instruments their SCXI, -1001 twelve-slot chassis, -1102 thirty-two-channel thermocouple amplifier, -1124 six-channel isolated digital to analog converter module, -1162 thirty-two optically isolated digital input module, -1163 thirty-two channel optically isolated digital output module, -1160 sixteen-channel single pole double throw relay module, -1303 thirty-two channel isothermal terminal block, -1325 terminal block, -1324 terminal block, -1326 terminal block, an AT-485/2 two-port RS-485 interface board, a two-port RS-232 interface board, and a LabVIEW base package for Windows 95/NT.

All hardware modules necessary for software development have been installed and the Labview drivers configured. A major portion of the user interface has been developed. This includes controls for valve operation, indicators for piping and temperature readings. All thermocouple voltages can be read using the data acquisition hardware. The voltages are then converted in the software to the appropriate temperature in degrees centigrade and displayed at the appropriate position on the user interface. The user can open and close valves by selecting and clicking with the mouse. This will change the state of the valve. When the user changes a valve state: 1.) the software commands are sent to the relay hardware to open or close the solenoid valve on the system. 2.) the user interface is updated; color indicators depict whether the valve is open or closed and whether there is gas flow through the associated piping. A valve state table is being developed to ensure safe operation of the system. The state table includes valves that must be open of closed in conjunction with the user selected valve and whether it is safe for the user to open the selected valve under the system conditions.

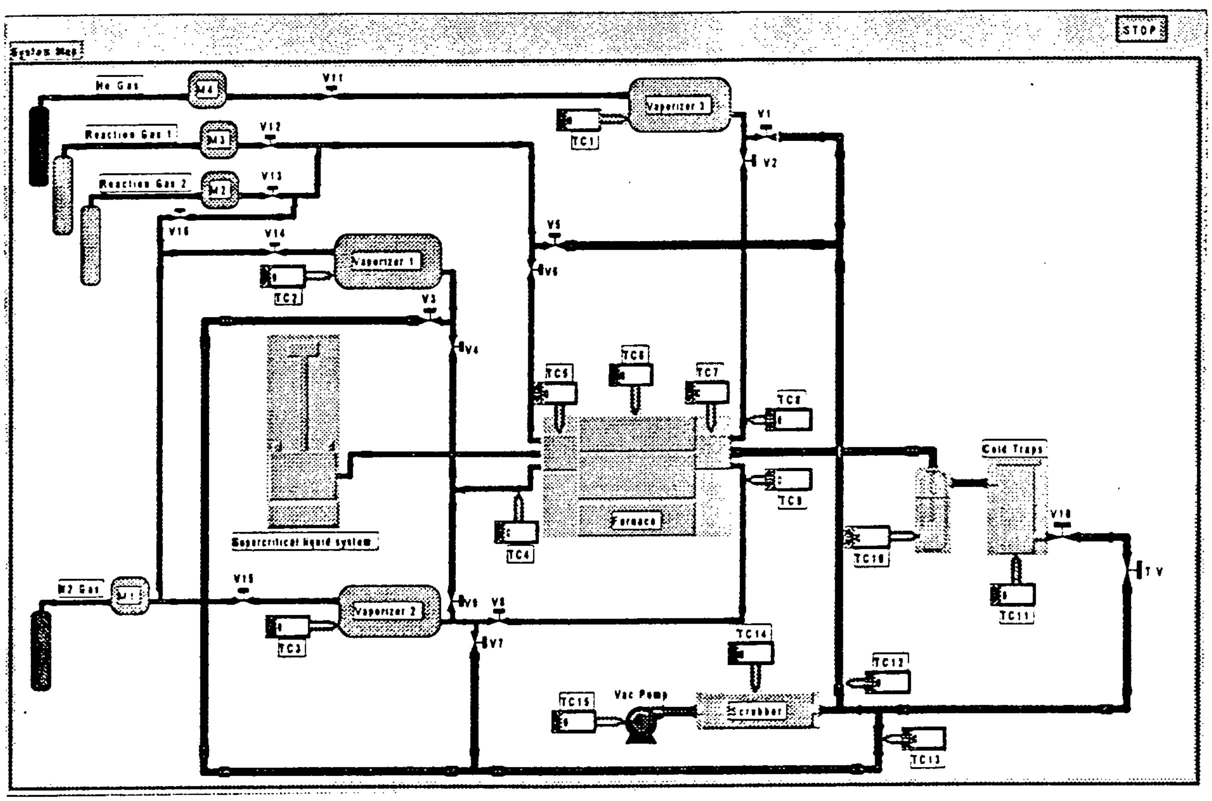

Figure 3: Printout of the LabView user interface for viewing valve, gas flow, and temperature readings. Valve can be opened or closed by pointing and clicking the desired valve with the computers mouse.

Personal Summary

Dr. Ming Li, a postdoc conducted initial procurement and expenditures, during the first year of receiving funding. His salary was paid by a matching fund from Florida State University; the matching fund was awarded for the successful funding of the project. Additional procurement and the assembly of the system were accomplished by James L. Guidry - a second year graduate student of Dr. Dahmen's who is now running test experiments and aiding Kurt Koetz, an Engineer of the Florida State University Physics Department, with the computer control system.

Future Plans

James L. Guidry will continue to work on the project in collaboration with Dr. Garmestani (College of Engineering - Florida State University and Florida Agricultural and Mechanical University.) The goal of this collaboration is to produce nanoparticles and composite materials. These particles and materials will be analyzed and characterized in facilities used by Dr. Gamestani and Dr. Dahmen.