HIGHER TECHNICAL INSTITUTE

MECHANICAL ENGINEERING DEPARTMENT

DIPLOMA PROJECT

DESIGN AND CONSTRUCTION OF A FIXED-BED THERMOCHEMICAL REACTOR

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Design and Construction Of a Fixed-Bed Thermochemical Reactor

By

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Project Report

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Abstract:

Nanostructured materials are a new class of synthetic materials with ultrafine microstructures, somewhat arbitrarily defined as structures smaller than 100 nanometers. These materials may be composed of metals, ceramics, polymers, and their composites. The materials can be in the form of powders, thin films, porous media, or dense structures.

The objective is to establish at H.T.I a Nanomaterials Research Center (**NRC**) with the target of being a leader in the rapidly emerging field of nanostructured materials and to achieve rapid growth and attractive industrial feedback from its proprietary position. The mission statement of the **NRC** is provided in the Appendices.

The breadth of the Center's technology places it in a position to achieve a strong proprietary position in both the manufacture and application of nanostructured powders. The Center's potential opportunity areas include the Spray Conversion Process for making nanostructured powders, the powder products of that process, processes for creating net-shaped parts from nanostructured powders, such as PIM, and nanostructured net-shape parts. **NRC** is focused on maximizing the commercial potential of its technology.

This project carries the title: "Design and Construction of a fixed-bed Thermochemical Reactor" and its major objective was to construct the essential experimental tool (Pilot Plant) for further activities in the future.

To My Father

Acknowledgements:

I would like to extend my sincere appreciation to the input provided by my supervisor ***Dr. Nicos Angastiniotis***. He was always involved in every phase of the project.

My colleague ***Michael Karaiskos*** was involved with the flow measurement characteristics of the process. His immense dedication and thoughtful feedback shares a considerable portion of this endeavor.

I am greatfull to ***Mr. Andreas Michaelides*** (Mechanical Engineer), who helped as overcome some difficulties came up during the design and construction of different components of the Pilot-Plant (e.g. End-cups, Consolidation mechanism, e.t.c).

I would also like to thank my father, ***Mr. Andreas Costa*** (***TYMPIOTIS METAL WORKS LTD***) who was always next to us, providing us his knowledge and experience on different issues with respect to the construction of the Pilot Plant.

Special thanks to ***Mr. Kaperakis*** (Chemical Engineer) and ***Mr. Chavales*** (Mechanical Engineer) of ***Linde Hellas, E.P.E, Ltd***, who willingly answered to some important questions we had on the regulators and on flame arresting devices (Linde products)

Thanks also to the Industry of "***Christakis Agathaggelou Ltd***" that provided us the "trolley" on which the Furnace is sitting on and also the "trolley" for the computer. They also were involved with the machining of various components of the Pilot Plant.

Finally, we would like to thank everyone who got involved in the design and the construction of the Pilot Plant.

Introduction:

The processing of materials from chemical precursors offers the potential for lower cost production of novel materials with homogenous ultrafine microstructures (nanostructures) and improved properties. The technology gained its initial prominence as the preferred route for synthesizing ceramic materials. Today, there is growing interest in the applicability of chemical processing technology to the production of metallic materials.

At H.T.I, we have been developing new capabilities for the synthesis and processing of nanostructured powders, starting from water soluble precursor compounds. After an extensive evaluation of synthesis routes, "Spray Conversion Processing" has emerged as the most versatile and reproducible. The new processing method consists of three coordinated steps:

(1) preparation and mixing of aqueous solutions of the precursor compounds to fix the composition of the starting solution;
(2) spray drying of the starting solution to form a chemically homogenous precursor powder;
(3) thermochemical conversion of the precursor powder to the desired nanostructured end-product powder.

The latter step may be performed in a *fixed-bed* ractor when the amount of powder being processed is small. However, for the thermochemical processing of large quantities of powder, it is advisable to use *fluid-bed* reactor, so as to ensure a uniform conversion rate for all

the particles in the bed. All three steps in the process are readily scaleable. An integrated manufacturing technology for the production of nanostructured composite powders is on its way here at H.T.I.

Spray drying is an essential step in the process when dealing with starting solutions that contain two or more precursor compounds. Rapid drying of aerosol droplets, accompanied by rapid precipitation of the solute, produces chemically homogeneous precursor powders, even from complex starting solutions. In other words, spray drying tends to suppress phase separation, which would normally occur during conventional crystallization of the solution mixtures. Typically, the spray dried precursor powders are spherical shells about 10-50 microns in diameter, and have amorphous or microcrystalline structures.

Thermochemical conversion of the precursor powder in a fluid-bed reactor is also an important step in the integrated process. This is because the local environment with respect to temperature and gas concentration in the fluid-bed reactor is the same for all parts of the bed, which insures uniform conversion of the precursor powder to the end-product powder. This is not the case in a fixed-bed reactor, where uniformity of gas percolation and temperature is difficult to maintain throughout the powder aggregate.

A considerable amount of research on nanostructured materials has been done using *Spray Conversion Processing* method.

In this project we have undertaken all the required steps in constructing the ***Pilot-Plant*** for the thermochemical conversion of precursor to the required end-product powder.

It is essential to state that the mixing of soluble salts and spray drying was the objective of previous project work at H.T.I (see reference 10).