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# Combinatorial screening of thin film materials: An overview

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**Abstract**

Over the past several decades, technological advancement has grown increasingly dependent on new and advanced materials. Accelerating the pace of new material discovery is thus critical to tackling global challenges in areas of energy, health, and security, for example. There is a pressing need to develop and utilize high throughput screening technologies for the development of new materials, as material discovery has fallen behind the product design cycles in many sectors of industry. This article describes techniques of high throughput combinatorial thin film material growth and characterization developed over the past several years. Although being adopted in selected industries, combinatorial screening technologies for *thin film materials* are still in their infancy. Caution must be exercised in selecting relevant combinatorial libraries and extrapolating from small-scale deposition techniques to industrially relevant processes. There are tremendous opportunities in the field of combinatorial discovery of thin film materials, as it enters its golden age along with the Materials Genome Initiative, which aims to change the pathway of materials discovery.


*Keywords:* High throughput screening technologies; Thin film materials; Materials discovery

## 1. Introduction

A great number of disruptive advances in technology over the past century resulted from the discovery of new materials. Furthermore, many of today's global challenges in energy, health, and security will also require the discovery of new classes of materials, including multi-component alloys and artificially structured materials or composites that have become known as *advanced* materials. It is becoming increasingly clear that new materials will be a key driving force behind a more competitive manufacturing sector and economic growth. In response to this challenge, the Materials Genome Initiative is a multi-stakeholder effort involving several funding agencies in the USA [1]. It aims to accelerate the pace of discovery of advanced materials, reducing the time required to bring new materials to market by at least 50% from the current 10–20 years.

While the Initiative is based on a strong computational and modeling approach, it is recognized that effective models of materials behavior can only be developed from accurate and extensive sets of data on materials properties. Indeed, the Initiative declares that "In the discovery stage it is crucial that researchers have access to the largest possible data set upon which to base their models, in order to provide a more complete picture of a material's characteristics [1]." It is important to understand how to best obtain such detailed data sets in the context of advanced materials that can be highly complex both in composition and structure.

In reality, the pace of new materials development remains slow in virtually all industry sectors. The time frame for bringing new materials to market is typically 10–20 years from laboratory discovery to first practical use. Fig. 1 shows examples of the time frame for a few widely used materials

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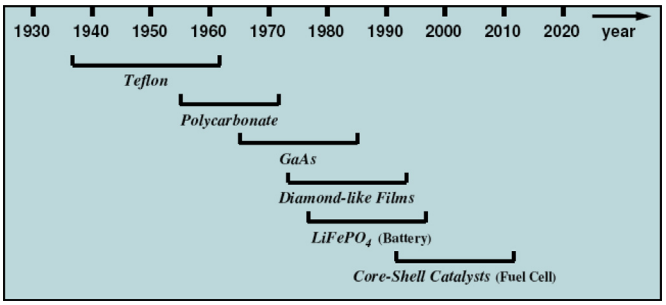


Fig. 1. Time frame of selected materials from initial research to market [2].

| | Number of Variations |
|---|---|
| **Base Material ($TiO_2$)** | |
| Co-dopant (improving absorption): | 50 |
| Co-dopant amount: | 5 |
| Co-catalyst (replacing Pt): | 30 |
| Co-catalyst amount: | 3 |
| **Material Synthesis Process** | |
| Precursor chemical selection: | 2 |
| Reaction temperature: | 4 |
| Reaction time: | 2 |
| **Total number of experiments:** | **360,000** |
| (~1000 years to complete if investigating 1 sample per day) | |

Fig. 2. Total number of experiments required to screen doped $TiO_2$ as industrial photocatalysts.

[2]. Such a lengthy time frame for materials to move from discovery to market is partly due to the time-consuming, trial-and-error style repetitive synthesis and characterization experiments that guide development. A frequently quoted example is that of Thomas Alva Edison who, it is said, worked through thousands of materials in searching for a light bulb filament more than a century ago, giving rise to the term "an Edisonian approach" to describe a linear process of materials development.

In his search for a filament, however, Edison was restricted to a relatively small number of materials, particularly carbonized natural fibers [3]. In contrast, modern advanced materials are often ternary, quaternary or yet higher order alloys. Furthermore, the properties of these materials are frequently modified by the adjustment of, for example, crystalinity, mesostructure and layering schemes. The total number of experiments needed to screen materials for modern day applications, therefore, can be orders of magnitude greater than a century ago. As an example, consider the discovery of the photocatalytic splitting of water on a $TiO_2$ electrode [4]. This ushered in a new era for heterogeneous photocatalysis based on semiconductor materials, which has been a subject of vigorous scientific research over the past 40 years [5,6]. In addition to material composition changes through elemental doping, however, variations in material synthesis also include different synthesis processes such as precursor chemical selection, reaction temperature and time. To illustrate the potential scale of the problem, consider a thought experiment to optimize a practical $TiO_2$-based industrial photocatalyst for a particular application. It is not unreasonable to expect to search 50 codopants (to optimize absorption) and 30 co-catalysts (to replace expensive platinum) at, for example, 5 and 3 different concentrations, respectively. In addition, precursor chemical selection can influence the end result, as can reaction temperature and reaction time. Adding 2 precursors, 4 reaction temperatures and 2 reaction times to the experimental matrix leads to 360,000 individual synthesis experiments as shown in Fig. 2 even for this simple optimization matrix. This is equivalent to about a thousand man-years of work if one different material is synthesized and characterized every day.

To address this limitation the Initiative includes high throughput combinatorial material synthesis technology that has already found applications in industry. In particular, it recognizes the need for further development of new high throughput technologies to characterize relevant material properties efficiently and quantitatively over a range of operating conditions and environments. Fig. 3 schematically illustrates the general procedures of high throughput material synthesis and characterization, integrated with theoretical material theory and modeling and material database development. Accelerating the fabrication and analysis loop is critical for reducing the time to market for new materials. The focus of this article is combinatorial thin film fabrication, first demonstrated [7] in 1995, which is among the most mature high throughput material synthesis technologies that have yielded new functional materials for a number of applications [8,9].

## 2. High throughput combinatorial thin film synthesis

Combinatorial synthesis of chemicals was developed in the mid-20th Century by Merrifield for the synthesis of peptide chains; work which formed the basis for his Nobel Prize in chemistry [10]. In the 1990s the synthesis technique was extended to commercial applications for drug discovery. In combinatorial chemistry, molecules are attached to a solid

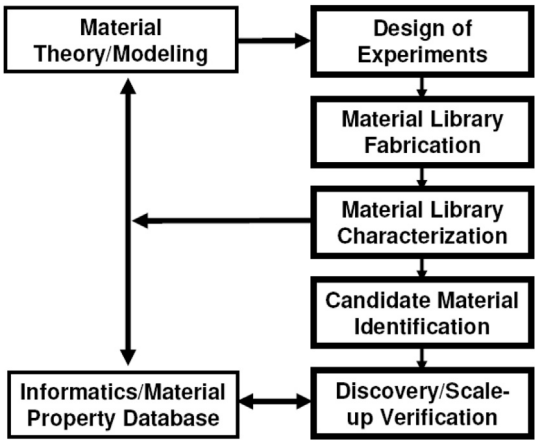


Fig. 3. General procedures of high throughput material synthesis and characterization, integrated with theoretical material theory and modeling, and material database development [8,9].