Combinatorial Materials Science: What's New Since Edison?

Eric J. Amis, Xiao-Dong Xiang, and Ji-Cheng Zhao, Guest Editors

Abstract

Combinatorial methods are high-efficiency methods to create large composition "libraries" of materials, for example, continuous composition variations, and test those compositions systematically in parallel for specific properties of interest, in contrast to the time-consuming one-composition-at-a-time approach. These methods have captured the attention of the materials industry with the promise of providing new discoveries "faster, better, and cheaper." However, in the academic community, combinatorial methods often meet with less enthusiasm, perhaps due to the perception of combinatorial methodology as an Edisonian approach to science. The facts are quite to the contrary. In addition to impressive successes arising from the application of combinatorial methods to materials discovery, results coming out of systematic high-throughput investigations of complex materials phenomena (which would be too time-consuming or expensive to undertake) provide data leading to improvement in theories and models of materials chemistry and physics. Indeed, combinatorial methods provide a new paradigm for advancing a central scientific goal—the fundamental understanding of structure–property relationships of materials behavior.

Keywords: *alloys, biomedical materials, ceramics, combinatorial methods, diffusion, electron microprobe, electronic materials, electronic properties, evanescent microwave probe, mechanical properties, nanoindentation, optical materials, phase equilibria, photoluminescence, polymers, structural materials, thin films.*

The advent of computers and automation equipment has dramatically accelerated the advance of both technology and science. Indeed, the ability to accomplish tasks a million, a billion, or even more times faster (and often, cheaper and better) is a driving force for such progress, for it greatly leverages the efforts of researchers and institutions across many disciplines. This process is exemplified in the unprecedented success of combinatorial and high-throughput approaches in the pharmaceutical and biotechnology industries. In these fields, automation of the fabrication of multivariate specimen arrays, screening and analysis techniques, and informatics has plainly hastened the development of important new drugs, drug variants, and genetic therapies, which accounts in part for the biotechnical revolution now in progress.

Given this triumph, it is a wonder that combinatorial techniques have not been more widely adopted in other scientific fields, particularly fundamental materials research, where, considering the case of formulations alone, the development and use of combinatorial techniques is a reasonable, if not obvious, next step. To be sure, the promise of "faster, cheaper, and better" materials discovery has captured the attention of many in the materials and chemical industries in the face of an increasingly competitive market. The response of the academic community, however, seems less enthusiastic. In part this reluctance has a philosophical basis.

Some have perceived combinatorial science as "merely" an engineering shortcut, a route to invention that circumvents true understanding (a criticism often leveled at Thomas Edison, despite his enormous success). In part, the academic reaction finds its foundation in the healthy skepticism with which scientists view all emerging fields, especially those, like combinatorial methods, that seem so promising. Accordingly, in this issue of *MRS Bulletin,* we strive to outline the current state of and recent advances in combinatorial and high-throughput methods applied to materials science through a collection of articles written by some leading researchers in the field. We hope that these articles will stimulate discussion regarding the significance of combinatorial techniques and their impact on the materials sciences in the 21st century. In addition, we trust that they will demonstrate how powerful combinatorial materials science methods have already become. As a means of further introducing this topic, a brief background on combinatorial materials science research is in order.

The ability to map out structure–property relationships is central to the goal of materials science. As materials scientists progress in this endeavor, considering more and more complex systems, the possibility of important discoveries increases, but so does the difficulty of the task. Phillips[1] estimated that at the end of the 1980s there were approximately 24,000 known inorganic phases. Of these, 16,000 were simple binary systems, and only 8000 were the more complex ternary phases. If 60 elements from the periodic table are considered, these could be combined to form roughly 34,000 ternary systems. Given that each of these systems could exhibit several phases (each with stoichiometry-dependent structure and properties), and that each system includes associated quaternary oxides and nitrides, it becomes increasingly clear that the number of understood complex systems is minuscule, compared with the total (and we have not even considered organic/polymeric systems!). To delineate the structure–property–processing relationships of each of these potentially useful systems would involve literally billions of experiments, a daunting task with "one-at-a-time" analysis and preparation techniques. And, while theoretical materials science has made great advances, it is expected (at least for the foreseeable future) that experimental exploration must bear most of this burden. It seems obvious that this challenge will not be met if we rely on traditional materials science techniques alone.

We should not underestimate the potential impact of combinatorial approaches on the fundamental understanding of materials physics and chemistry. The systematic mapping of complex systems can help reveal phenomena that may enrich and challenge our understanding of materials physics and behavior. The results coming out of combinatorial mapping (which would be too time-consuming or expensive to generate by conventional methods) can test and improve theoretical models and hypotheses. In the past, serendipitous observations made significant contributions to materials design and materials science. At the very least, combinatorial approaches can make such observations regular occurrences.

Although the recent resurgence of interest in combinatorial materials science may be rooted in a series of works published by Berkeley researchers in the last decade,[2,3] groundbreaking experiments in the field were first published in the 1960s. Indeed, many of the principles developed in this period (e.g., the use of gradient techniques) are being utilized and advanced today across a broad range of materials science. Take, for example, the 1965 paper of Kennedy et al.,[4] which described a ternary-alloy phase "library" produced using electron-beam coevaporation techniques and analyzed with electron diffraction. These authors succeeded in demonstrating a qualitative agreement between their phase diagram, produced by combinatorial techniques, and that determined by conventional methods. Although significant discrepancies remained, these were mainly due to the limitations of their deposition and analysis equipment. In 1967, Miller and Shirn[5] analyzed the Au-SiO_2 system using a cosputtering technique. By carefully aligning Au and SiO_2 targets, a film exhibiting a controlled composition gradient of Au/SiO_2 was deposited on the substrate. This gradient library was used to measure the electrical resistivity of the system over the full range of composition. Similar "composition-spread" methods were used to study transition-metal-alloy superconductors in the following years.[6–8]

These pioneering works spurred some enthusiasm for combinatorial materials science during the late 1960s and early 1970s. Hanak et al., for example,[6,8] made a substantial effort to find novel superconductors at the RCA Laboratories using gradient libraries. A similar technique was also used by Hartsough and Hammond[9] in a study of A15-structure superconducting V_3Al. In this work, the ability to form the phase *in situ* was critical, as the A15 structure was inferred to be unstable above 700°C, well below the temperature required for conventional metallurgical processing (over 1000°C). Geballe, Hammond, and co-workers used this technique in subsequent studies of binary and pseudobinary superconductors.[10]

Independently, Berlincourt, at the U.S. Office of Naval Research (as director of the Physical Science Division), proposed a national effort to conduct a search for high-temperature superconductors using gradient-deposition methods and automated screening techniques.[11] This proposal, however, was not adopted, and interest in combinatorial materials methods waned. While this could have been due to many issues, one certain factor is that the computers and analytical instruments available at the time did not have the speed, automation, or resolution required to make them effective tools for combinatorial materials studies. Today, of course, these capabilities exist. One can only imagine the impact of combinatorial methods had they been further pursued and developed during the intense search for high-temperature cuprate superconductors in the 1980s, an effort that spanned more than 10 years and involved thousands of scientists worldwide. Recently, van Dover et al. at Bell Laboratories continued to improve the codeposited composition-spread technique[12] and performed extensive studies of amorphous dielectrics, as discussed in the first article in this issue by Takeuchi et al. Likewise, the second article, by Sun and Jabbour, also presents a convincing case for the power of this methodology for materials discovery in functional optical, electronic, and photonic materials.

While the current combinatorial materials science renaissance is certainly inspired by the success of discovery efforts in the biotechnology sector, and employs seminal ideas from the field's early researchers, it is also a direct result of the enormous advances in instrumentation, computing resources, and the understanding of materials science in recent years. The articles collected in this issue of *MRS Bulletin* are also a testament to the "coming of age" of this aspect of combinatorial materials science.[13] In the article by Yoo and Tsui, examples are reviewed of detailed, reliable phase-diagram mapping (including structural and physical characterization) using sophisticated modern techniques such as molecular-beam epitaxy, micro-x-ray diffraction, and evanescent microwave impedance imaging. The article by Zhao et al. illustrates a diffusion multiple approach for rapid mapping of bulk phase diagrams and mechanical properties such as hardness and elastic modulus with an efficiency ~3 orders of magnitude higher than the conventional one-alloy-at-a-time approach. This approach evolves from the diffusion couple method, widely used by metallurgists for more than a century to study diffusion coefficients and phase diagrams, but takes advantage of advances in instrumented nanoindentation[14,15] that allow effective measurement of both elastic modulus and hardness from very small areas. Thus, for the first time, critical composition–property relations can be mapped using diffusion couples and diffusion multiples.[16,17] The final article of this issue, by Meredith et al., reviews some unique applications of the combinatorial and high-throughput approaches to polymer materials science. This work is especially focused on the development of quantitative measurements for the fundamental characterization of the effects of processing variables.[18,19] Polymer materials also require the development of special techniques for library preparation because the typical sputtering and vapor deposition methods are not applicable to polymers.[18,20] This article also demonstrates the application of cell growth and proliferation to provide a high-throughput analysis of potential biomaterials for tissue engineering.[21]

Because of limited space, we have chosen not to address applications of combinatorial methods to the development of catalysts in this issue. This important application area for combinatorial methods has generated substantial interest and investment from the chemical industry, and it has been the subject of several conferences, papers, and reviews in recent years.[22]

The articles in this issue emphasize the imperative that combinatorial and high-throughput methods be developed further. They offer the possibility of meeting the future needs of materials discovery in timely, cost-effective, and thoroughly systematic ways. We expect a lot from combinatorial materials science. We hope that the following articles will persuade you to invest your confidence and enthusiasm in this exciting new field as well.

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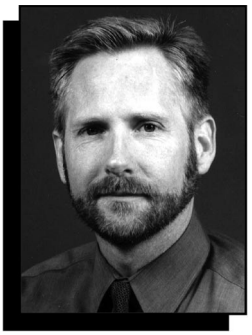

Eric J. Amis

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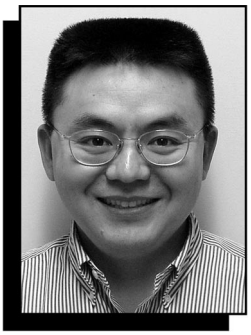

Ji-Cheng (J.-C.) Zhao

Eric J. Amis, Guest Editor for this issue of *MRS Bulletin*, has been chief of the Polymers Division of the National Institute of Standards and Technology (NIST) since May 1999. In 1998, he initiated a program that applies combinatorial and high-throughput methods to measurements in materials science and biomaterials, which led to the establishment of the NIST Combinatorial Methods Center.

Amis earned his PhD degree in chemistry from the University of Wisconsin and worked as a National Research Council postdoctoral associate at NIST's predecessor, the National Bureau of Standards. Before joining NIST in 1995 to lead the Polymer Blends and Processing Program, Amis spent 11 years as a faculty member in the Chemistry Department of the University of Southern California. Since 1992, he has been the editor of the *Journal of Polymer Science: Polymer Physics Edition.* He is also a fellow of the American Physical Society (APS) and a past chair of the APS Division of Polymer Physics. Amis has worked primarily in the areas of solution rheology combined with light, neutron, and x-ray scattering methods to investigate the physics of complex systems, such as biomembranes, polyelectrolytes, associating polymers, gels, polymer crystallization, and dendritic polymers. He has authored or coauthored more than 150 scientific papers.

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He received his PhD degree in physics from the University of Kentucky in 1989 and worked as a postdoctoral research fellow at the University of California, Berkeley, from 1989 to 1993. He then moved to Lawrence Berkeley National Laboratory and was a career staff scientist and principal investigator. Xiang has conducted extensive research in solid-state physics, materials research, and advanced instrumentation. He discovered a new class of high-T_c intercalated copper oxide superconductors and performed the first series of electrical transport measurements on single crystals of intercalated oxide superconductors and fulleride superconductors. This study yielded important information on superconducting mechanisms in these novel materials. Xiang has also designed and developed various high-sensitivity instruments, including an extremely sensitive displacement detector (with a sensitivity of 10^{-4} Å), a scanning evanescent microwave probe (resolution of <0.1 μm) for spatially resolved quantitative evaluation of electromagnetic impedance, and a highly sensitive spin-resonant microscope. He received the 1996 Discover Magazine Award for Technological Innovation, and in 2000 he received the R&D Award for his pioneering work in combinatorial materials science. He has authored or coauthored more than 70 scientific papers and holds over 15 patents.

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Zhao received his BS (1985) and MS degrees (1988) in materials from Central South University (CSU), China, and his PhD degree in materials from Lehigh University (1995) after teaching at CSU from 1988 to 1991. He has been honored with the Geisler Award from ASM International (Eastern N.Y. Chapter) and the Hull Award from the General Electric Corporate Research Center (now the General Electric Global Research Center). Zhao has 30 publications and holds six patents, with 20 more pending. He has also edited a book on materials design approaches and experiences.

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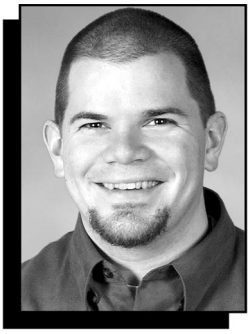

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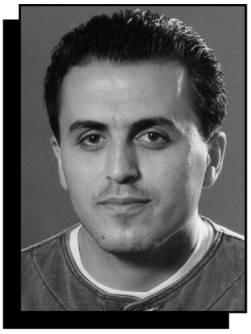

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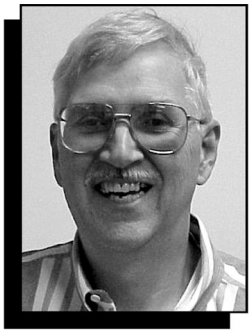

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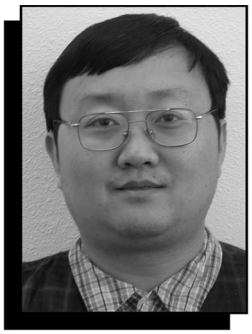

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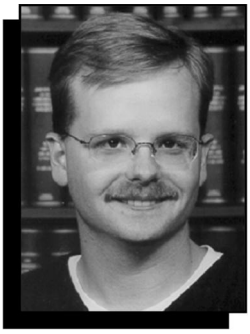

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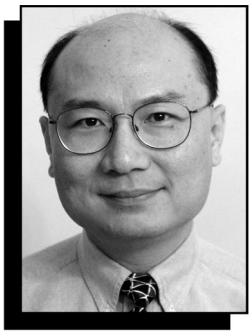

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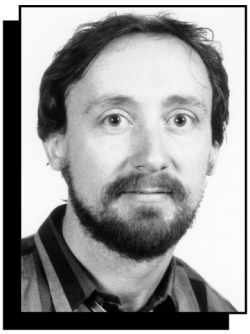

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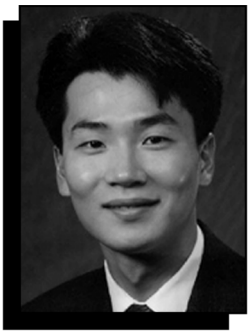

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research position in chemical kinetics at Kansas University and then shifted his interests to amorphous silicon and other thin-film electronic materials. His current research is focused on combinatorial nanotechnology for functional oxides. He founded the International Workshop on Oxide Electronics in 1995 and the Japan–U.S. workshop on Combinatorial Materials Science and Technology in 2000.

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