

# Molecules in the Solid State

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Guest Editors

## Abstract

The design and synthesis of solid-state materials constructed from molecules has emerged as an important frontier of materials research. Molecular materials promise an unparalleled opportunity for systematic manipulation of solid-state properties and functions by using molecular design principles and capitalizing on the versatility of organic synthesis. Furthermore, the use of molecular components may produce considerable economic benefits, whether by reducing fabrication cost or through increases in performance. The articles in this issue of *MRS Bulletin* cover recent discoveries and developments based on materials with properties and functions that hinge on the characteristics of their molecular constituents. These materials promise significant advances in several technologies of substantial commercial interest, including organic light-emitting diodes, nonlinear optics, gas separations, chiral separations, and molecular magnets.

## Introduction

Research during the last half of the 20th century led to the discovery and development of remarkable inorganic materials which spawned numerous technologies that collectively have become essential to modern society, as exemplified by silicon-based transistors, gallium arsenide lasers, ceramic piezoelectrics and thermoelectrics, and metallic and metal oxide magnetic storage media. Without these remarkable solids, progress in computers, electronics, optics, communication, energy storage, solar cells, machine tools, sensors, and more simply would not have been possible.

Although inorganic materials will continue to play a central role in such applications, materials based on molecular building blocks offer unparalleled versatility with respect to properties and function. This already is amply demonstrated by the ubiquity of polymeric materials—molecular building blocks linked by covalent bonds—that unquestionably play a key role in numerous commercial arenas. More recently, the coupling of functional organic molecules with inorganic components has prompted considerable interest in “hybrid” materials and devices, for example, semiconductor field-effect transistors decorated with molecular and biomolecular receptors.<sup>1</sup>

In the last decade, pronounced growth has occurred in functional solid-state materials that rely on the properties of uniquely tailored molecules embedded in the solid state either as isolated entities in composites or as single-phase materials containing discrete constituents—organic and organometallic molecules—held together by noncovalent forces. The ability of organic synthesis to produce functional molecules on demand promises substantial advances in materials design for existing technologies as well as emerging ones. As a group, molecular-based materials exhibit most of the properties typically associated with more conventional inorganic and elemental solids, from superconductivity to ferromagnetism to piezoelectricity to second harmonic generation. Like most inorganic materials, solid-state materials generated by the assembly of discrete molecules often are highly crystalline (aka “molecular crystals”). Unlike their inorganic counterparts, however, molecular-based materials tend to be mechanically soft and typically have melting or decomposition points ranging from 100–400°C, in contrast with typical ranges of 500–1500°C for inorganic materials. However, molecular-based materials usually are soluble in aqueous or organic solvents, providing access to solution-

based processing methods that introduce substantial economic advantages as well as prospects for fabricating devices with unusual (e.g., nonplanar) geometries.

## History and Development of Molecular Materials

The articles contained in this issue of *MRS Bulletin* address materials that derive their properties from molecules in the solid state. The recognition that molecules play a role in solid-state properties can be traced to some key historical benchmarks beginning in the latter part of the 19th century, a significant period for both the history and development of molecular materials. In 1848, Pasteur performed the first resolution of a chiral compound by physically separating crystals of sodium ammonium tartrate into left- and right-handed forms (a molecule is chiral if it cannot be superimposed on its mirror image; the two forms are enantiomers). Pasteur brilliantly observed that the crystals were different: the right- and left-handed forms were enantiomers and could not be superimposed, and thus were chiral. More than 150 years later, the impact of this discovery is still felt as numerous research efforts employ chiral reagents and catalysts for asymmetric synthesis, through which one particular enantiomer can be made with high selectivity. Ironically, molecular crystals with chiral environments are now being explored for asymmetric synthesis and selective separations of enantiomers, which clearly would have delighted Pasteur.

Early interest in molecular materials was sparked further by the development of alizarin and indigo dyes in Germany during the latter half of the 19th century, and the optical properties of molecular solids are as important today as they were then. In the 1960s, Schmidt and co-workers further stimulated interest in organic solid-state chemistry through their demonstration of stereospecific syntheses and polymerizations in the solid state by ultraviolet or  $\gamma$ -irradiation of crystalline molecular materials.<sup>2</sup> Schmidt's research established a connection between solid-state packing of different forms (i.e., polymorphs) of cinnamic acid and “topochemical” reactivity in the solid state. In a topochemical process, the fixed orientation of the reactants can lead to unusual stereospecific intermolecular reactions that cannot be accomplished in solution. Schmidt's work stimulated scientists to think about intermolecular interactions in crystals and provided the conceptual framework for the discipline of “crystal engineering.”

A particularly significant burst of activity in molecular crystals began in the

1960s with the emergence of organic electron-donor–electron-acceptor complexes, also known as charge-transfer complexes, followed by investigations of electronic transport in organic crystals. This spawned a flurry of activity in organic metals, superconductors, and semiconductors,<sup>3–10</sup> the latter reemerging recently in the form of new strategies for organic light-emitting diodes and field-effect transistors.<sup>11,12</sup> For example, single crystals of rubrene, a well-known poly-

acene molecule, exhibit hole mobilities in a field-effect transistor configuration that rival those measured for amorphous silicon (Figure 1).<sup>13,14</sup> Others have built on Schmidt's strategy of topochemical solid-state reactions to make polydiacetylenes—potentially useful as optical limiters, waveguides, and thermometric sensors—directly from crystals of discrete diacetylene molecules that are aligned properly in the solid state.<sup>15–17</sup> New materials based on infinite frameworks,

bounded only by their crystal dimensions, constructed from molecular “tinkertoys” held together by hydrogen bonding or metal-ligand coordination are emerging as promising candidates for molecular separations and gas storage.<sup>18,19</sup> More recently, the materials science of pharmaceutical compounds, which often consist of small-molecule crystals, has attracted considerable attention, owing to the importance of crystal form (i.e., polymorphs), phase stability, and mechanical properties in therapeutic formulations. The reader is directed to the article by Jones et al. in the November 2006 issue of *MRS Bulletin* for a comprehensive overview of this topic.<sup>20</sup>

### The Emerging Importance of Solid-State Structure

Collectively, these efforts and others have reinforced the notion that solid-state properties can be manipulated through careful design and selection of the molecular components. But they have also demonstrated that solid-state properties strongly depend on the molecular organization in the solid state and, in the case of electronic materials, the interactions between electronic states on adjacent molecules. Consequently, a key ingredient in materials design from molecules is the prediction and control of solid-state structure, which remains one of the foremost challenges in organic solid-state chemistry.

Advances in materials research invariably rely on a thorough understanding of solid-state structure and its relationship to solid-state properties. Indeed, the evolution of materials, from metals to alloys to ceramics to steels to specialty electronic materials, testifies to the inextricable linkage between solid-state structure and properties. As such, the emergence of organic solid-state chemistry as a discipline and the associated development of functional molecular materials can be linked to advances in solid-state structure determination, particularly single-crystal x-ray diffraction.

Although single-crystal x-ray diffraction was used initially to determine molecular structure alone, the strong connection between solid-state structure and properties in molecular crystals has increasingly directed attention toward analysis of molecular organization in the solid state and elucidation of the factors that govern solid-state packing. The Cambridge Structural Database, founded in 1965, is a compendium of more than 400,000 crystal structures, with more than 500,000 expected by 2010 (Figure 2).<sup>21</sup> Perhaps not surprisingly, the explosion in structural information is correlated

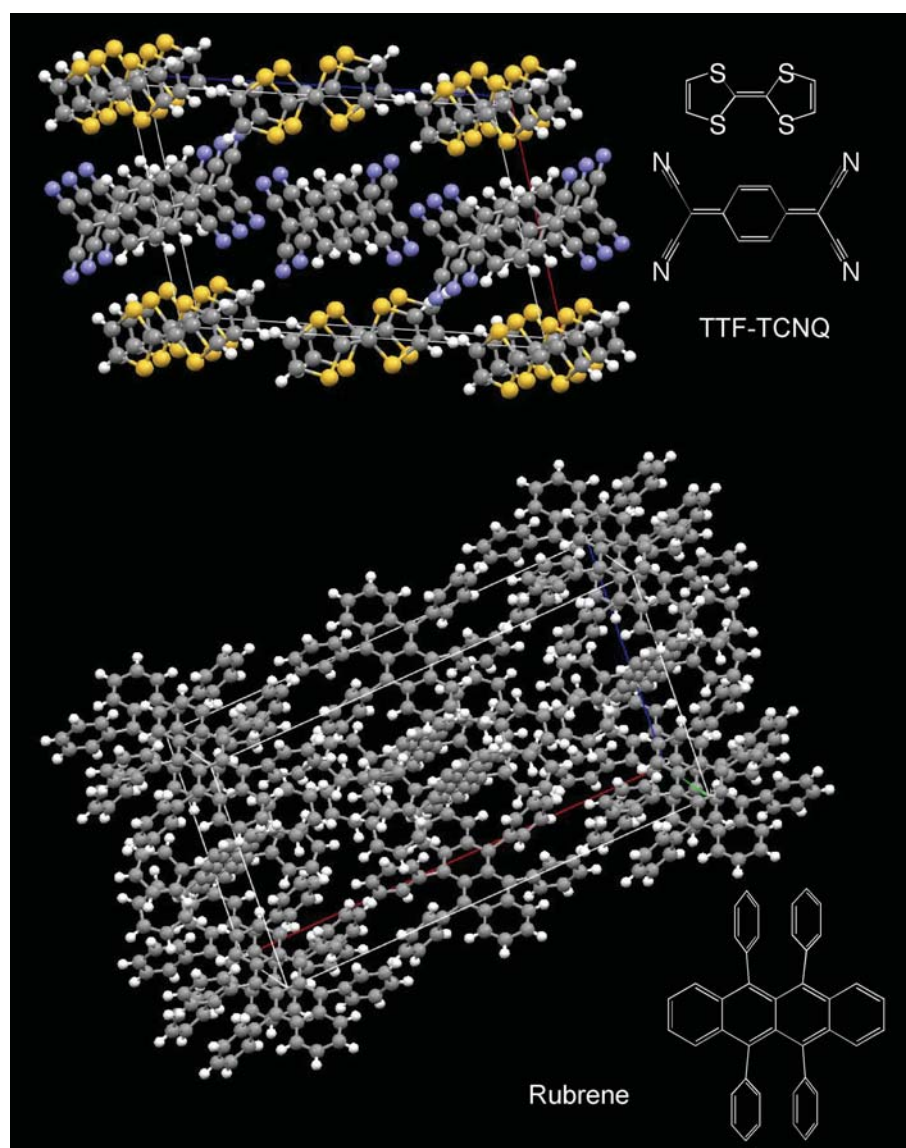


Figure 1. (top) Unit cell in a single crystal of tetrathiafulvalene-tetracyanoquinodimethane (TTF-TCNQ), the first “organic metal,” reported in 1974 (see Reference 3). Segregated stacks of TTF (with yellow sulfur atoms) and TCNQ molecules (with blue nitrogen atoms) result in the formation of an electronic band structure that endows the crystalline material with metallic conductivity. The TTF and TCNQ molecules are depicted at right. (bottom) Unit cell in a single crystal of rubrene, a polyacene that has recently been shown to exhibit hole mobilities near those of amorphous silicon (see References 12 and 13).

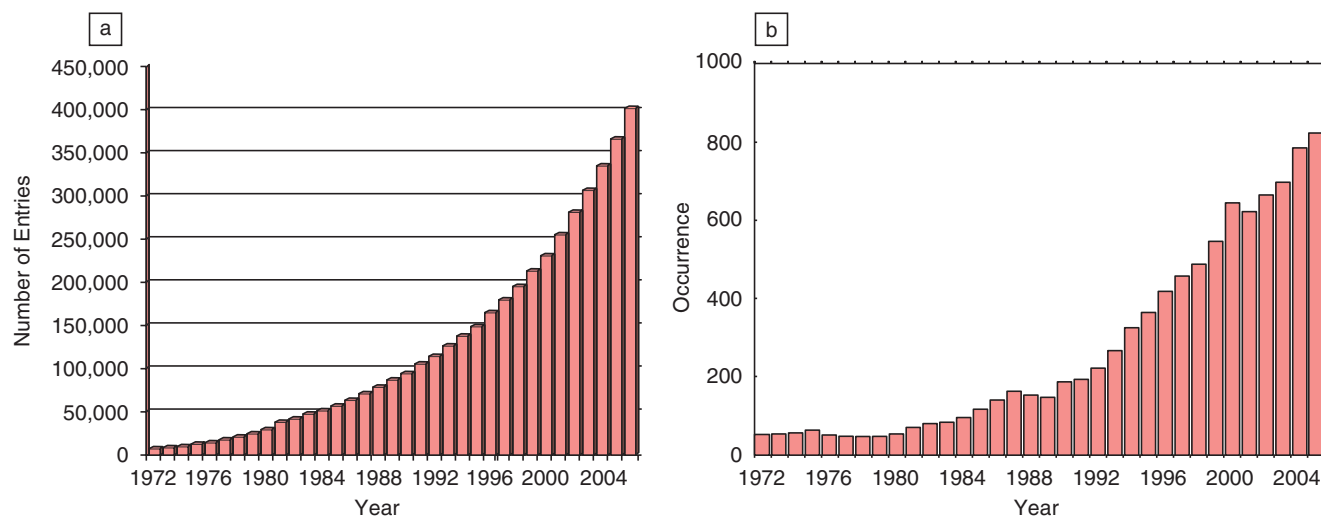


Figure 2. (a) Growth (1972–2006) in the number of entries to the Cambridge Structural Database. (Figure reproduced with permission of the Cambridge Crystallographic Data Center, [www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk).) (b) Growth (1972–2005) in the number of publications found using the search string “molecular materials + crystal” in SciFinder.

with the discovery and development of molecular materials, illustrating the relationship between synthesis, structure, and properties, a key tenet of materials research.

### In This Issue

Clearly, “molecules in the solid state” is a topic of enormous breadth, and this issue of *MRS Bulletin* can address only a small portion of the activities in the field. The articles in this issue were selected to illustrate the versatility and potential of molecular materials, with an emphasis on materials that are relevant to key technologies and have not been portrayed in previous issues of *MRS Bulletin*. Some of the articles describe efforts that are embryonic but promising, whereas others are farther along the path to commercial products.

The article by Tanaka and Kitagawa illustrates how crystalline metal coordination frameworks can be constructed (based on the structure-directing influence of the metal-ligand coordination sphere) with precisely prescribed pore sizes and environments, leading to new materials capable of storing methane, acetylene, and oxygen. The underlying physics related to the interactions between small molecules and networks is a key feature of these materials, which have the potential to impact several technologies, including fuel storage.

In his article, Lin considers molecular porous solids from a different point of view—their use and potential as asymmetric catalysts and as media for the separation of enantiomeric isomers. The

synthesis of chiral zeolites, porous materials that provide a chiral environment for either enantioselective separations or catalysis, can have a tremendous impact in the pharmaceutical industry, where enantiomeric purity is of paramount importance. As Lin notes, however, enantiopure chiral zeolites are difficult to obtain because of the high temperatures employed during their synthesis. The Lin group is actively pursuing the rational design of molecule-based chiral zeolites, which can be prepared under mild conditions, based on robust metal-organic extended frameworks. The ability to characterize precisely the solid-state structure of Kitagawa’s and Lin’s materials using single-crystal diffraction, combined with the ability to tune the solid-state structure and properties through judicious choice of the molecular building blocks, heralds a promising approach to technological obstacles that remain unresolved.

Miller’s article acknowledges that magnetic materials traditionally have been based on metals and metal oxides with three-dimensional network structures. However, magnetically ordered materials based on organic molecules possess spin-bearing organic radicals that contribute to both the magnetization and spin coupling that lead to magnetic ordering. An extremely broad range of behavior has been observed for these materials, including ferri- and ferromagnetism and antiferromagnetism; in these molecular materials, one can also observe spin Peierls transitions, spin density waves, and metamagnets.

The final two articles address important aspects of the optical properties of molecules in the solid state. Busby et al. discuss the preparation of electroluminescent devices through self-assembly of building blocks consisting of either molecular coordination compounds or functionalized nanoscale zeolite containers infused with organic dyes. The building blocks are interchangeable, providing an avenue to optimize energy transfer and tune the color of the emitted light. Carefully designed systems can produce white light, a key objective of solid-state lighting technology.

Marder et al. describe polymer formulations containing specially designed light-responsive molecules capable of two-photon absorption coupled to photocleavable groups that increase the solubility of the surrounding polymer matrix. The peculiar nonlinear properties of two-photon absorption enable excitation of the designer molecules with tight spatial confinement, in turn enabling the scribing of small feature sizes and the fabrication of complex three-dimensional objects that can impact photonics, materials processing, biology, and medicine.

Although space limitations prohibit us from capturing all the activities encompassing molecules in the solid state, we hope that the contributions in this issue of *MRS Bulletin* convince the reader of the technological potential of this ever-growing subject. Furthermore, we hope this issue will inspire readers to explore materials based on molecular constituents that will advance the development of new technologies.

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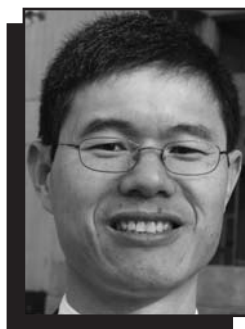
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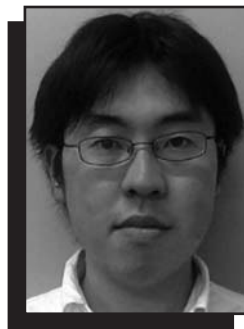


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