

Bioinspired Materials for Self-Cleaning and Self-Healing

Jeffrey P. Youngblood and Nancy R. Sottos,
Guest Editors

Abstract

Biological systems have the ability to sense, react, regulate, grow, regenerate, and heal. Recent advances in materials chemistry and micro- and nanoscale fabrication techniques have enabled biologically inspired materials systems that mimic many of these remarkable functions. This issue of *MRS Bulletin* highlights two promising classes of bioinspired materials systems: surfaces that can self-clean and polymers that can self-heal. Self-cleaning surfaces are based on the superhydrophobic effect, which causes water droplets to roll off with ease, carrying away dirt and debris. Design of these surfaces is inspired by the hydrophobic micro- and nanostructures of a lotus leaf. Self-healing materials are motivated by biological systems in which damage triggers a site-specific, autonomic healing response. Self-healing has been achieved using several different approaches for storing and triggering healing functionality in the polymer. In this issue, we examine the most successful strategies for self-cleaning and self-healing materials and discuss future research directions and opportunities for commercial applications.

Introduction

The extraordinary properties and functions of biological systems provide a new paradigm for the design and fabrication of engineering materials.^{1–3} Biologically inspired synthesis, hierarchical structuring, and stimuli-responsive materials chemistry have enabled materials systems with unprecedented function. Many exciting bioinspired materials concepts are currently under development, such as composite materials with nacre-like flaw tolerance,^{4,5} gecko-inspired reversible adhesives,^{6,7} and advanced photonic structures that mimic butterfly wings.⁸ This issue of *MRS Bulletin* focuses on two remarkable examples of materials with demonstrated bioinspired function: self-cleaning surfaces and self-healing polymers. Self-cleaning materials are based on the superhydrophobic effect whereby water drops show nearly spherical profiles (contact angles of $\sim 180^\circ$).^{9,10} Superhydrophobic surfaces are generally designed on principles at work in the lotus leaf where the surfaces have hydrophobic micro- and nanostructures that suspend

water droplets.^{11–13} Self-healing materials are inspired by living systems in which minor damage (e.g., a contusion or bruise) triggers an autonomic healing response. Successful healing relies on seamless integration of reactive chemical functionality into a polymer or polymer composite at the microscale, nanoscale, or molecular level.

Although these two functions are quite different, together they represent the wide range of autonomic responses achievable through bioinspired design. In this article, we separately introduce the key elements of self-cleaning and self-healing materials systems and identify the scientific challenges and successful strategies for continued advancement of these nascent fields.

Self-Cleaning Surfaces

Interest in self-cleaning surfaces has been rekindled because of the newfound ability to structure surfaces on the submicron scale over large planar areas relevant to macroscale wetting. These structured surfaces allow the so-called “lotus-leaf

effect,” whereby water droplets are shed easily from certain structures, carrying away dirt and other debris.^{11–13} Such bioinspired topographical methods toward self-cleaning are the focus of this review.

Basics of Wettability

Every material has an energy associated with its surface, and when a fluid droplet is in contact with this surface, the energies of the three-phase contact line balance to a minimum, forming a distinct angle of contact with the other surface that is described by Young's equation¹⁴

$$\cos \theta = (\gamma_{sv} - \gamma_{sl})/\gamma_{lv} \quad (1)$$

where γ_{sv} is the solid–vapor surface energy, γ_{lv} is the liquid–vapor surface energy, γ_{sl} is the solid–liquid interfacial energy, and θ is the angle of contact between the drop and the solid surface.

As lotus-like surfaces have structured surfaces, one might conclude that such structuring is necessary for self-cleaning. Indeed, surface roughness has a large effect on the contact angle. In the simplest case, a rough surface has more surface area underneath the liquid, and therefore, more surface energy must be taken into account. Wenzel¹⁵ stated this relationship in mathematical form

$$\cos \theta_A = r \cos \theta_T \quad (2)$$

where θ_A is the actual (measured) contact angle; θ_T is the thermodynamic angle as defined by Equation 1; and r is the roughness ratio, defined as the true surface area divided by the geometric area of integration.

As roughness increases, one must also consider that the fluid fails to penetrate the asperities because of the Laplace pressure.^{16,17} The surface becomes composite, with water sitting on both the surface and air. The situation is then similar to the case of chemically heterogeneous patchy surfaces for which Cassie¹⁸ derived an equation by geometrically averaging the constituents. In this case, the composite surface is simply a mixed surface where one constituent is air. Cassie and Baxter¹⁹ derived the following equation for this behavior assuming that $\theta = 180^\circ$ for the liquid–air interface

$$\cos \theta = f_1 \cos \theta_1 - f_2 \quad (3)$$

where θ_1 is the contact angle of the surface with water, f_1 is the fractional area of contact with the surface, and f_2 is the fractional contact area with air underneath the drop.

Although thermodynamics prescribes the contact angle, any physical or chemical nonuniformity causes the value to differ

Self-Cleaning Surfaces: An Industrial Perspective

Chuck Extrand

As an industrial researcher, I am frequently asked to update business leaders and executives at the company where I work on the progress of our projects. In 2002, during one of our technology update forums, we showed water bouncing off a superhydrophobic surface that we had created and then allowed several executives to try bouncing drops themselves. The initial response was a combination of amazement and excitement. Enthusiasm quickly waned when one of our executives innocently touched the surface with his finger, destroying its superhydrophobic nature. For me, this example captures the general state of bioinspired and self-cleaning surfaces: They have great potential, but many practical challenges still remain.

Much of the recent work has looked to nature for inspiration. However, simply mimicking nature's designs might not be effective. Natural surfaces generally have a number of advantages—they often have the ability to repair or renew themselves (e.g., plants) or are short-lived (e.g., insects). Most bioinspired and self-cleaning surfaces have been laboratory curiosities, yet there have been few commercial successes. Water-repellent fabrics are one of the prevalent examples in the marketplace. Why have self-cleaning surfaces not become ubiquitous? Most surfaces produced today cannot withstand high hydrostatic pressures and are easily damaged. If a self-cleaning surface fails, then liquids stick more tenuously than if the surface had not been treated at all.

How can researchers make an impact? By following one of two paths: pursuing fundamental research or creating practical surfaces. For the fundamental research path, the focus should not be the Cassie and Wenzel equations and theoretical high contact angles. The dogma and polemics that surround these fundamentally flawed constructs only fetter creativity and slow progress. Promising work by Gao et al. in this issue suggests one path for fundamental research.

The alternative path involves applied research into practical surfaces. Such surfaces must be self-cleaning in practice, durable, and easy to manufacture. Surfaces should prevent penetration of high-pressure liquids during immersion, flow, or spraying and then dispatch the liquids with minimal forces or pressures (from inclination, shaking, spinning, and so on). Any practical surface also must withstand touching or rubbing, as well as exposure to environmental contamination. The manufacturing process must be quick and versatile. The introductory article lists a toolbox of reported methods to make such materials. The process cannot take days and must be suitable for a broad array of surfaces beyond silicon wafers. If we can accomplish this, someday, our houses, cars, boats, and clothing might be covered with these highly engineered surfaces.

from that dictated by surface energetics.²⁰ Nonuniformities raise the contact angle of a drop advancing across a surface above the thermodynamic value, whereas the contact angle of a drop receding across a surface will be depressed. This contact angle hysteresis (the difference between the advancing angle and the receding angle) thus gives a measure of the degree of nonuniformity of the surface, with greater amounts of hysteresis indicating higher degrees of imperfection.

A drawback of the Wenzel and Cassie–Baxter constructions is their inability

to account for the advancing and receding contact angles. (For more information on other limitations, see the article in this issue by Geo et al.) Johnson and Dettre^{17,21–24} proposed a model in which the rough surface causes metastable local minimum states that trap the three-phase contact line. Their model correctly predicts both the advancing and receding contact angles and the hysteresis between them, which phenomenologically corresponds to observed behavior,^{22,25,26} as shown graphically in Figure 1a.

Bioinspired Structured Surface

In the design of self-cleaning surfaces, the ability of the surface to shed water is of paramount importance. The force required to move a drop across a surface, and therefore the angle at which a drop slides off the surface, is proportional to the contact angle hysteresis, according to^{27–30}

$$F \propto \gamma_{LV}(\cos \theta_{\text{rec}} - \cos \theta_{\text{adv}}) \quad (4)$$

Thus, the key to repellency is in reducing the hysteresis and not necessarily the actual surface energy. Surfaces that exhibit near-zero hysteresis of water and, therefore, have drops that move readily are thus termed superhydrophobic.

According to the plot of Johnson and Dettre²⁴ of the water contact angle on a surface with sinusoidal roughness (Figure 1a), the contact angle on the surface has low contact hysteresis above a critical roughness value. That value also corresponds to the transition between behavior caused by the increased surface area defined by the Wenzel equation and behavior of a composite surface as dictated by Cassie. Whereas the physical heterogeneity of the roughness acts to increase hysteresis, the composite nature of the surface lessens this effect, as air has no hysteresis. This region is the superhydrophobic region. A droplet in this region has almost no energetic barrier to motion and moves easily.^{26,31–33}

The superhydrophobic effect is the origin of self-cleaning.^{11,12,34,35} As the nearly spherical water droplets roll around, they encounter debris and other particulates. The debris is loosely bound to the surface because the structuring provides few contact points, so that the surface tension of the water “grabs” the material and localizes it to the surface of the drop. Eventually, the ease of motion allows the droplet to slide off the surface with very little resistance, thus carrying the debris off the surface as well.

In nature, insects use superhydrophobicity to great effect to accomplish such feats as walking on water. The water strider has hierarchical structure at the end of its legs (tarsi) with protrusions called setae, each at the tens-of-microns scale, with grooves at the hundreds-of-nanometers scale (Figure 1b).³⁶ These structures, coupled with inherent hydrophobicity, allow the water strider to attain extremely high fractions of air at the leg–water interface. Essentially walking on air, this insect can move freely about the surface of water, as the high contact angle overcomes gravity and the low hysteresis allows the leg to move onto and off of the water surface with ease.

Nature uses similar methods to attain self-cleaning structures (Figure 1b), such

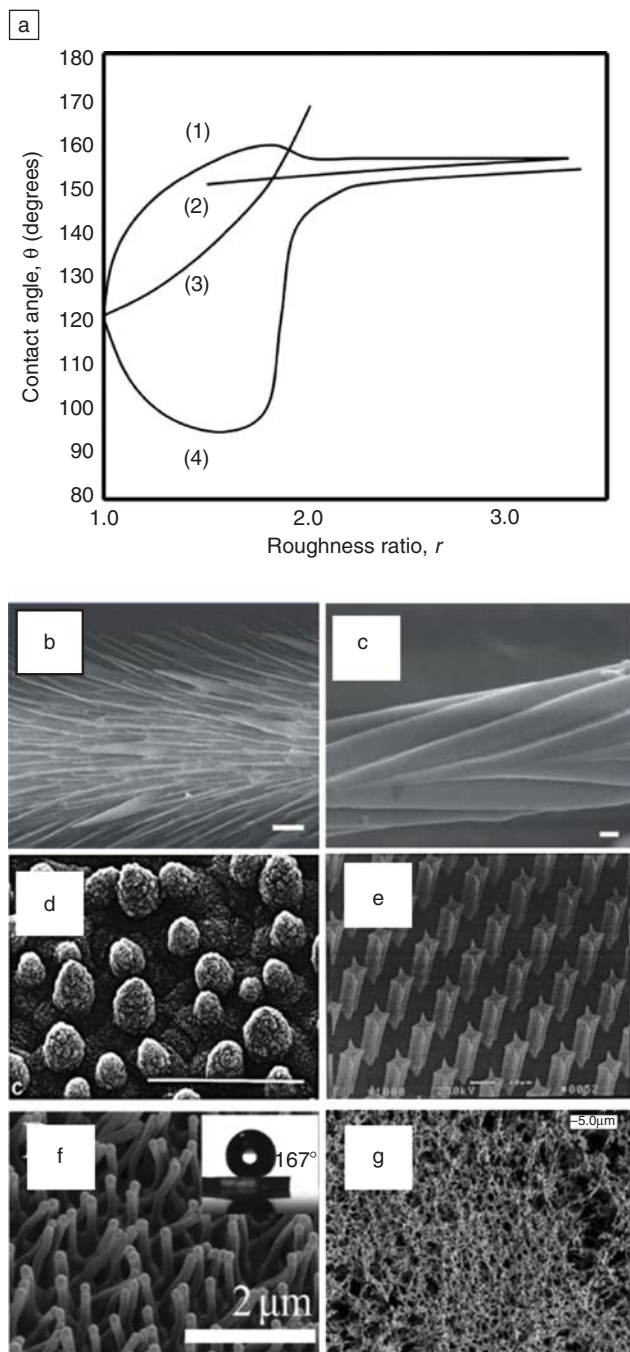


Figure 1. (a) Plots of contact angle versus roughness for (1) Wenzel's equation, (2) Cassie and Baxter's equation, and Johnson and Dettre's work for both the (3) advancing and (4) receding angles on a surface of sinusoidal roughness.²⁴ (b–d) Superhydrophobic structures from nature: (b) water strider leg microstructure,³⁶ (c) water strider leg nanogrooves,³⁶ and (d) lotus leaf.¹³ (e–g) Synthetic superhydrophobic structures: (e) photolithographed posts,³³ (f) templated polymers nanofibers,⁴² and (g) solvent structured surface.⁴⁸

as butterfly wings,³⁵ cicada wings,³⁷ and lotus leaves,^{11–13} the latter being a notable example and the reason superhydrophobicity is also termed the lotus-leaf effect. Much like the water strider, the lotus has

multiple-length-scale roughness on the surface of its leaves with protrusions of tens of microns that are themselves each bumpy at the scale of hundreds of nanometers.^{11–13} The structures of the leaf

create a contact angle greater than 150° with hysteresis of only a few degrees.³⁷ These values allow the water drops to slide off with ease, carrying dirt and debris with them—the essence of superhydrophobicity-derived self-cleaning.

Outlook

Heretofore, the aim of many superhydrophobicity and self-cleaning researchers has been the elucidation and physical explanation of the key features necessary to achieve lotus-like surfaces. Factors such as size scale,^{9,26} topology,^{31,33} and multi-scale structure^{38,39} are of utmost importance. Others have focused on a toolbox of methods for creating these materials. A variety of superhydrophobic surfaces obtained using dry methods such as plasma modification,^{26,40} laser etching,⁴¹ and templating⁴² and wet methods such as layer-by-layer deposition,⁴³ colloidal assembly,⁴⁴ electrospinning,^{45–47} and solvent evaporation⁴⁸ have been reported.

Going forward, the chief issue that must be addressed for these materials to realize their ultimate potential as superhydrophobic materials is a lack of mechanical integrity. Because the superhydrophobic effect is essentially topographical, any loss of structuring leads to loss of self-cleaning. Unfortunately, at the necessary size scales, robustness is generally unattainable. As self-cleaning surfaces will likely be used in systems where periodic mechanical cleaning (as in windows) or aggressive water flow (as in boat hulls) is necessary, the ability to resist abrasion is key. Use of stronger materials such as metals or ceramics will help mitigate this issue. However, an even better approach would be to combine self-cleaning properties with the ability to self-heal. For materials that achieved such a combination, the surface would eventually recover all superhydrophobic behavior even after being scratched, wiped with a rag, or slammed against a dock. Such self-healing materials are considered next.

Self-Healing Polymers

Structural polymers are used in applications ranging from adhesives to coatings to microelectronics to composite airplane wings, but they are highly susceptible to damage in the form of cracks. These cracks often form deep within the structure, where detection is difficult and repair is almost impossible. Regardless of the application, once cracks have formed within polymeric materials, the integrity of the structure is significantly compromised. The addition of self-healing functionality to polymers provides a novel solution to this long-standing problem

and represents the first step in the development of materials systems with greatly extended lifetimes.

In biological systems, chemical signals released at the site of fracture initiate a systemic response that transports repair agents to the site of injury and promotes healing (Figure 2a). The biological processes that control tissue response to injury and repair are extraordinarily complex, involving inflammation, wound closure, and matrix remodeling.⁴⁹ Coagulation and inflammation begin immediately when tissue is wounded. After about 24 hours, cell proliferation and matrix deposition begin to close the wound. During the final stage of healing (which can take several days), the extracellular matrix is synthesized and remodeled as the tissue regains strength and function. Ideally, synthetic reproduction of the healing process in a material requires an initial rapid response to mitigate further damage, efficient transport of reactive materials to the damage site, and structural regeneration to recover full performance.

Although no materials system yet accomplishes this complete set of autonomic healing processes, crack healing has been explored in a wide spectrum of materials.⁵⁰ The efficiency of crack healing for structural materials is defined as the ability to recover the mechanical integrity of the virgin (undamaged) material, including such properties as fracture toughness (K_{IC}), fracture energy (G_c), elastic stiffness (E), and strength (σ_{ult}). For some materials systems, healing is achieved autonomically (independently and automatically) without any external intervention, whereas others require additional energy (such as heat) to heal.

In polymers and polymer composites, two distinct approaches for self-healing have emerged. In the first, the crack-mending process is initiated by an external thermal-, photo-, mechanical- or chemical-induced stimulus. Successful crack healing has been achieved through both molecular diffusion and thermally reversible solid-state reactions. In the second approach, damage in the form of a crack triggers the release of healing agents stored in the material, as also occurs for fracture events in biological systems (Figure 2b). Both compartmentalized and continuous delivery strategies have been demonstrated. Site-specific crack healing is achieved autonomically in this approach, without any external intervention.

Thermally Induced Crack Mending

Crack healing has been achieved in a range of polymer systems through the use of heat and pressure to promote diffusion

of polymer chains and/or chemical changes (Figure 3). In many cross-linked polymers, healing occurs through chain interdiffusion if there is physical contact between the crack planes and sufficient viscoelastic deformation and wetting.^{51,52} An increase of the temperature above the glass transition, T_g , and application of pressure significantly enhance the mending process.⁵¹ The manual addition of solvent also promotes polymer healing by effectively lowering T_g .⁵³ For certain ionomers, self-healing is triggered by thermal energy transferred to the polymer during a projectile puncture event.⁵⁴

Thermally induced reversible polymerizations, in which the material contains a reversible or dynamic covalent bond, have also been exploited for crack healing. Chen and co-workers used a retro Diels–Alder strategy to prepare thermally repairable cross-linked polymers (Figure 3).^{55,56} With the application of heat

(120–150°C) and modest pressure, these polymers demonstrate high crack-healing efficiencies based on recovery of fracture toughness.^{55–57} More recently, a thermoreversible, self-healing rubber based on hydrogen bonding has been reported.⁵⁸ Details on the underlying chemistry of these and several new polymer systems are described in the article by Williams et al. in this issue.

Trigger and Release for Autonomic Healing

Polymers that self-heal with no external intervention rely on the quiescent, stable storage of liquid healing agents and an activator or catalyst within the material. The healing components must remain active and separated until damage occurs, without significantly impacting the inherent properties of the material. Effective storage and release has been achieved with healing-agent-filled microcapsules

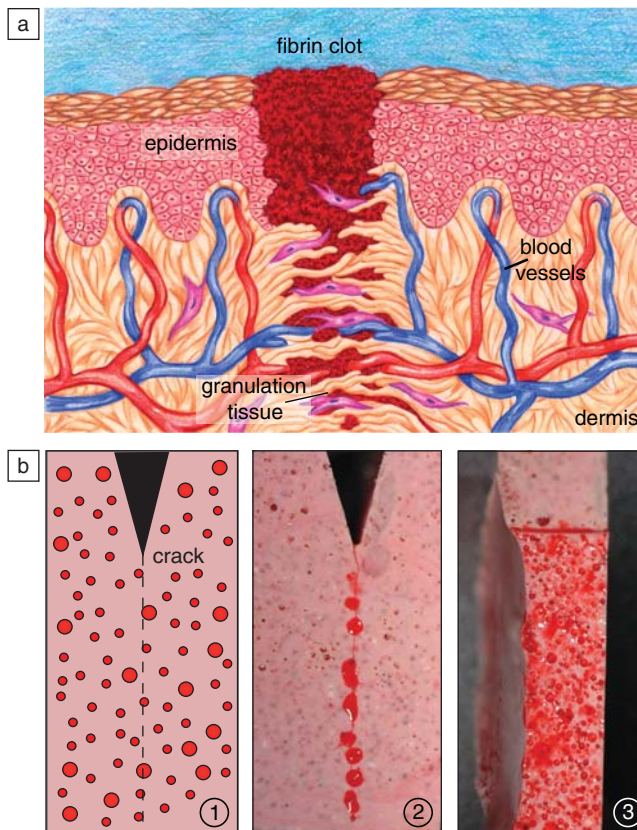


Figure 2. (a) Schematic of an intermediate stage of biological wound healing in skin. Tissue damage triggers bleeding, which is followed by the formation of a fibrin clot. Fibroblast cells migrate to the wound site enabling the creation of granulation tissue to fill the wound.⁴⁹ (b) Demonstration of bioinspired damage-triggered release of a microencapsulated healing agent in a polymer specimen: ① schematic of compartmentalized healing agent stored in a matrix; ② release of dyed healing agent into the crack plane, which leads to a synthetic clotting (polymerization) process to bond the crack faces; and ③ one-half of the fracture surface revealing ruptured capsules.

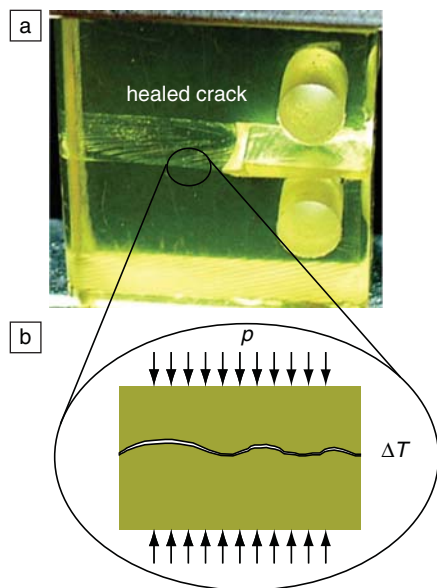


Figure 3. (a) Thermally repairable cross-linked polymer fracture specimen with healed crack. (Reproduced with permission from Reference 55. Copyright 2002 American Association for the Advancement of Science.) (b) Schematic of localized thermally induced crack-mending process. Applying pressure, p , normal to the crack faces and increasing the temperature repairs the fracture.

(Figure 4a),^{59–64} hollow glass fibers (Figure 4b),^{65–70} and phase-separated droplets.⁷¹

The interaction of a crack with the storage vessel plays a critical role in the successful design of these self-healing materials. Rupture of the shell wall is the mechanical trigger to the healing process, and without it, no healing occurs. Key parameters for successful rupture include the shell wall thickness, the stiffness and toughness of the shell wall material, the interfacial adhesion between the vesicle and the polymer matrix, and the inherent stiffness and toughness of the matrix material system.⁶²

White and co-workers^{59–62,72,73} first achieved high healing efficiencies in epoxy materials and epoxy-based composites with a healing reaction based on the ring-opening metathesis polymerization (ROMP) reaction of microencapsulated dicyclopentadiene (DCPD) with solid particles of Grubbs catalyst. Bond and co-workers^{67,69,70,74} have explored a two-part epoxy chemistry stored within hollow glass fibers to heal a structural fiber-reinforced epoxy-based composite. As described in the articles by White et al. and Bond et al. in this issue, several new

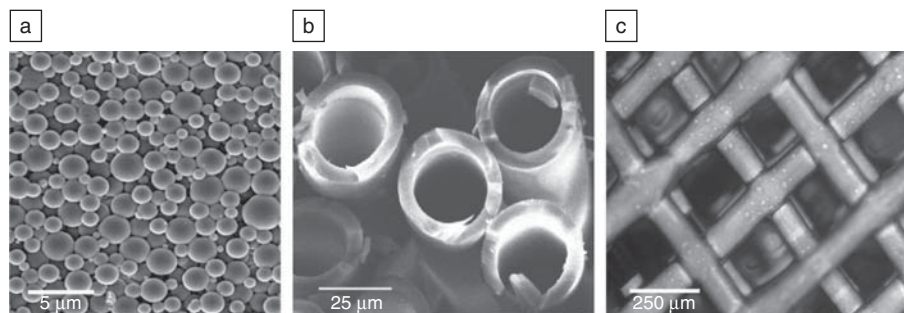


Figure 4. (a) Healing-agent-filled microcapsules produced by interfacial polymerization.⁸⁵ (b) Hollow glass fibers. (Reproduced with permission from Reference 91 by permission of Sage Publications Ltd.) (c) Three-dimensional network of microvascular channels embedded in an epoxy matrix. (Photo credit: D. Theriault.)

compartmentalized self-healing materials systems have emerged, ranging from elastomers^{71,75} to highly cross-linked polymer composites.^{64,76–79}

Although self-healing polymers composed of compartmentalized healing agents exhibit remarkable mechanical performance and regenerative ability, these materials generally are limited to autonomic repair of a single damage event in a given location. Once the capsules or fibers in a localized region are depleted of healing agent, further repair is precluded. A biologically inspired solution to this problem is the introduction of a circulatory system that replenishes and perfuses the host material with the chemical building blocks of healing (Figure 4c). In contrast to the compartmentalized approach, this strategy allows the supply of healing agent to be replenished indefinitely in a vascular network so that polymers with greatly extended lifetimes can be achieved. Using a microvascular coating/substrate architecture that mimics human skin, Toohey et al.⁸⁰ demonstrated repeated autonomic healing of a single crack in the coating. Williams et al.⁷⁸ have developed a vascularized composite sandwich structure that is capable of rebonding delamination damage. Recent analyses suggest ways to design and optimize vascularized networks for healing with minimized impact on structural performance;^{81–84} the challenge is to realize these structures.

Outlook

Research and development activities for self-healing polymers are expanding rapidly, with new concepts and materials systems under development in academic, government, and industrial laboratories worldwide. The ultimate goal for self-healing materials is complete synthetic reproduction of the biological healing processes described previously. Future

self-healing systems could incorporate fully autonomic circulatory networks capable of healing large damage volumes, or at the other end of the spectrum, they could rely on self-healing nanostructure such as nanocapsules⁸⁵ or migration of nanoparticles.^{86,87}

Recent developments in mechanochemically active polymers,⁸⁸ in which chemical changes are triggered in response to the local stress state, might result in a new class of polymers that are able to sense and repair damage. In addition, new cost-effective self-healing chemistries have been introduced (e.g., see Caruso et al.⁷⁶) that will facilitate the transition of self-healing polymers to commercial applications. Scratch-healing coatings are already generating significant interest for automotive, optics, and corrosion-resistant uses.^{89,90} Load-bearing applications such as self-healing adhesives and structural composites might require longer development times.

In This Issue

In this issue of *MRS Bulletin*, the first three articles introduce the topic of self-cleaning surfaces and explore current issues of superhydrophobicity. In the first article by Genzer et al., recent methods in the construction of materials with self-cleaning and anti-biofouling behavior are explored. Critically, the article delves into how the structures presented relate to natural self-cleaning structures. In the second article, Gao et al. explain how contact angle hysteresis depends on the physics of the three-phase contact line and can be treated in terms of activation energies between metastable states. The authors use these arguments to show how the presence of two different length scales is an important aspect of lotus-like behavior and shed light on the issue of why most natural self-cleaning surfaces

have such multiscale hierarchical structures. The third article, by Tuteja et al., describes how re-entrant curvature (a curving of the surface of the asperities away from the three-phase contact line as the water penetrates into the surface) is a dominant effect in superhydrophobic surfaces and that proper control can even lead to superoleophobic materials. The low-energy oils can be in the Cassie state, in which a rough surface under a liquid has a composite interface of liquid contact and air contact, and display unique wetting properties.

The second half of the issue switches focus to the growing field of self-healing polymers. The article by Williams et al. provides an overview of the chemistries used in self-healing materials, as well as those currently in development. Self-healing materials are divided into two conceptually distinct classes based on the underlying chemistry: autonomic and nonautonomic. The second article, by White et al., focuses on polymer systems that heal in an autonomic fashion, without the need for human intervention. Attention is directed toward describing the types of systems under development and how these remarkable materials function. The issue closes with the article by Bond et al., which describes the various self-healing technologies currently being developed for applications in fiber-reinforced polymeric composite materials. All three articles offer perspectives on future self-healing approaches.

Acknowledgements

N.R. Sottos gratefully acknowledges the support of the U.S. Air Force Office of Scientific Research (grant Nos. FA9550-06-1-0553 and FA9550-05-1-0346), the U.S. Army Research Office (grant No. W911NF-07-1-0409), and the National Science Foundation (grant Nos. CMS 05-27965 and CMS 02-18863), as well as the contributions of the Autonomic Materials Systems Group at the University of Illinois Beckman Institute. J.P. Youngblood acknowledges the support of the Purdue Research Foundation.

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Jeffrey P. Youngblood



Nancy R. Sottos



Christopher W. Bielawski



Ian P. Bond



Mary M. Caruso

Jeffrey P. Youngblood, Guest Editor for this issue of *MRS Bulletin*, can be reached at the School of Materials Engineering, Neil A. Armstrong Hall of Engineering, 701 West Stadium Ave., Purdue University, West Lafayette, IN 47907, USA; tel. 765-496-2294, and e-mail jpyoungb@purdue.edu.

Youngblood is an assistant professor of materials engineering at Purdue University. He did his undergraduate studies at Louisiana State University, majoring in chemistry and physics. Working in the laboratory of William Daly, Youngblood spent three years working on compatibilization, aging, and thermomechanical investigation of asphalt/polymer blends and the synthesis of liquid crystalline nonlinear optical polymers. In 2001,

Youngblood completed his PhD degree at the University of Massachusetts Amherst in the Department of Polymer Science and Engineering—under the tutelage of Thomas McCarthy—after having investigated the ultrahydrophobic (lotus) effect, developing general methods for chemical surface modification of polymers and synthesizing pendant siloxane block copolymers. Moving on to postdoctoral work at Cornell University's Materials Science and Engineering Department under the direction of Christopher Ober, Youngblood developed synthetic strategies for the development of coatings that prevent marine biofouling. In 2003, Youngblood accepted a position in the School of Materials Engineering at Purdue University. In recent

years, the Youngblood laboratory has investigated a variety of fields including the biocompatibilization, activity enhancement, and understanding of surface properties of polymeric quaternary biocides; electrospinning of carbide, nitride, and functional ceramics; processing of ultrahigh temperature materials; stimuli-responsive anomalous wetting and anti-fog materials; adhesives; techniques for modification of surfaces to control wettability; and, of course, self-cleaning surfaces.

Nancy R. Sottos, Guest Editor for this issue of *MRS Bulletin*, can be reached at the Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, 1304 W. Green St., Urbana, IL 61801, USA; e-mail n-sottos@uiuc.edu.

Sottos is the Donald B. Willet Professor of Engineering in the Departments of Materials Science and Engineering at the University of Illinois Urbana-Champaign (UIUC). Sottos started her faculty career at UIUC after earning her BS and PhD degrees in 1986 and 1991, respectively, in mechanical engineering from the University of Delaware. In addition to her position at UIUC, she also is a co-chair of the Molecular and Electronic Nanostructures Research Initiative at the Beckman Institute for Advanced Science and Technology and a University Scholar. Sottos' research group studies the mechanics of complex, heterogeneous materials, such as self-healing polymers, advanced composites, thin-film devices, and microelectronic packaging, specializing in

micro- and nanoscale characterization of deformation and failure in these material systems. Her work on self-healing polymers was recognized by *Scientific American's* SciAm 50 Award for research demonstrating outstanding technological leadership in 2007.

Christopher W. Bielawski is an assistant professor of chemistry at The University of Texas at Austin. He received his BS degree in chemistry in 1997 from the University of Illinois at Urbana-Champaign and his PhD degree in chemistry from the California Institute of Technology (Caltech) in 2003 under the mentorship of professor Robert H. Grubbs. After postdoctoral studies in the laboratories of professor David A. Tirrell at Caltech, Bielawski accepted his current position in 2004. His research interests are



Wonjae Choi

centered on the applications of N-heterocyclic carbenes in polymer chemistry and catalysis.

Ian P. Bond can be reached at the University of Bristol, Department of Aerospace Engineering, Queen's Building, University Walk, Bristol, BS8 1TR, UK; tel. 44-117-928-8662, fax 44-117-927-2771, and e-mail I.P.Bond@bristol.ac.uk.

Bond is a reader in aerospace materials at the University of Bristol, UK. After receiving his BS degree in materials science (1991) and his PhD degree (1995) from the University of Bath, Bond briefly worked in the aerospace industry before accepting a position at Bristol in 1997. He has published more than 50 papers in the field of composite materials with a particular emphasis on the development of self-healing. Bond also has been invited to present his work at numerous international workshops, seminars, and conferences—establishing him as a leading figure in this field.

Mary M. Caruso is an organic chemistry graduate student at the University of Illinois at Urbana-Champaign under the guidance of



Robert E. Cohen

J.S. Moore and S.R. White. She earned her BS degree in chemistry from Elon University in 2006 where she conducted undergraduate research under the direction of K.D. Sienarth. Caruso's graduate research includes developing new catalyst-free self-healing systems.

Wonjae Choi can be reached by e-mail at wonjaec@mit.edu.

Choi is a graduate student in the Department of Mechanical Engineering at the Massachusetts Institute of Technology. He received his BS and MS degrees at Seoul National University. Choi's major research interest is small-scale fluid dynamics including superhydro/oleophobicity of various surface textures.

Robert E. Cohen can be reached at the Department of Chemical Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA; e-mail recohen@mit.edu, and Web site <http://web.mit.edu/cohengroup>.

Cohen is the St. Laurent Professor of Chemical Engineering at the Massachusetts Institute of Technology (MIT) and directs the DuPont/MIT Alliance. Prior to joining the MIT faculty in 1973,



Daniel R. Dreyer

Cohen studied at Cornell University (BS degree), the California Institute of Technology (MS and PhD degrees), and was a post-doctoral researcher at Oxford University. He is the founding director of MIT's Program in Polymer Science and Technology and the architect of MIT's unique Doctoral Program in Chemical Engineering Practice. Based on patents produced in his laboratory, Cohen also co-founded MatTek Corporation in 1985. Cohen's publications reflect interests in polymer structure/property relations. He is a Fellow of the American Institute of Chemical Engineers and the American Physical Society.

Daniel R. Dreyer is an inorganic chemistry graduate student at The University of Texas at Austin studying under C.W. Bielawski. Dreyer obtained a BS degree in chemistry from Wheaton College in 2007 where he conducted research in confocal microscopy and lipid bilayers with Daniel Burden. Dreyer also worked with Mark Foster at The University of Akron on the characterization of plasma-polymerized thin films.

Chuck Extrand is a principal scientist and man-



Chuck Extrand

ager of the Entegris Research Group. He received his PhD degree in polymer engineering from The University of Akron and his BS degree in chemical engineering from the University of Minnesota. Prior to joining Entegris, Extrand worked in universities and national laboratories in France and Japan. His recent research activities include surface engineering and permeation resistance of polymers. In addition, Extrand holds nine U.S. patents and has published more than 60 papers in journals, books, and conference proceedings.

Alexander Y. Fadeev can be reached at the Department of Chemistry and Biochemistry, Seton Hall University, South Orange, NJ 07079, USA; tel. 973-275-2807, fax 973-761-9772, and e-mail fadeeval@shu.edu.

Fadeev is an associate professor at Seton Hall University. He received his degree in chemistry in 1986 and his PhD degree in physical chemistry in 1990 from Moscow State University. He stayed at the university as an assistant and associate professor until 1997 when he became a visiting scholar at the University of Massachusetts Amherst.



Alexander Y. Fadeev

Fadeev joined Seton Hall University in 1990 as an assistant professor. His major research interests involve covalent modification of metal oxide to impart functionality for control of properties including adsorption and wettability.

Lichao Gao can be reached at the Department of Polymer Science and Engineering, University of Massachusetts, Amherst, MA 01003, USA; tel. 413-577-1533, fax 413-577-1510, and e-mail lichao@mail.pse.umass.edu.

Gao has been a post-doctoral research Fellow at the University of Massachusetts Amherst since 2005. She received her BS degree in chemistry from Hebei University in 1999. In 2004, Gao's PhD degree research at Nankai University in Tianjin involved block copolymer self-assembly and aggregation of micelles. Her recent research includes fundamental wettability physics, surface property control, ionic liquids, liquid marbles, and semi-conducting nanocomposites.

Jan Genzer can be reached by email at Jan_Genzer@ncsu.edu.

Genzer is a professor of chemical and bio-



Lichao Gao

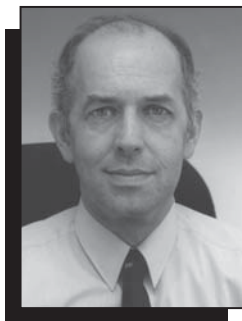
molecular engineering at North Carolina State University (NCSU). He earned his “Diploma-engineer” degree in chemical and materials engineering from the Institute of Chemical Technology in Prague, Czech Republic, in 1989, and his PhD degree in materials science and engineering from the University of Pennsylvania in 1996. Afterward, Genzer spent two and a half years as a postdoctoral Fellow at Cornell University and the University of California–Santa Barbara. In the fall of 1998, he joined the faculty of Chemical Engineering at NCSU as an assistant professor. In addition to his appointment at NCSU, Genzer is an adjunct professor at the Norwegian University of Science and Technology in Trondheim, Norway. His group at NCSU actively pursues experimental and computer simulation research to understand the behavior of polymers and oligomers at interfaces and in confined geometries, with particular emphasis on self-assembly, forced assembly, and combinatorial methods. Genzer is a member of the editorial boards of the *Annual Review of Materials Research*, *Polymer*, *Macromolecular*



Jan Genzer

Chemistry & Physics, *Macromolecular Theory & Simulations*, and *Journal of Dispersion Science and Technology*. Also, he is a Fellow of the American Physical Society (APS). Genzer’s honors include the Camille Dreyfus Teacher-Scholar Award, the Sigma Xi research award, the National Science Foundation (NSF) CAREER award, the John H. Dillon Award of the APS, the NSF Award for Special Creativity, and NCSU’s Outstanding Teacher award.

Abraham Marmor is a professor of chemical engineering at the Technion—Israel Institute of Technology. He received his DSc degree from the Technion in 1974. Marmor then spent two years as a postdoctoral researcher at the State University of New York at Buffalo. Later, he was a visiting associate professor at the University of Wisconsin—Madison and a visiting scientist at the IBM Almaden Research Center. Marmor has been working in the field of interfacial phenomena for about 30 years, has published extensively in this field and related research areas, and has consulted for major companies. Marmor also has participated in many



Abraham Marmor

international conferences and has been active in lecturing in universities and industrial sites in many countries. At the Technion, professor Marmor received awards for excellence in research and teaching. In addition, he was an editor of *Reviews in Chemical Engineering*, and was on the advisory committees of *Journal of Colloid and Interface Science* and *Journal of Adhesion Science and Technology*.

Thomas J. McCarthy can be reached at the Department of Polymer Science and Engineering, University of Massachusetts, Amherst, MA 01003, USA; tel. 413-577-1512, fax 413-577-1510, and e-mail tmccarthy@polysci.umass.edu.

McCarthy is a faculty member in the Polymer Science and Engineering Department at the University of Massachusetts Amherst. He received his BS degree in chemistry from the University of Massachusetts in 1978 and his PhD degree in organic chemistry from the Massachusetts Institute of Technology in 1982. McCarthy’s research has involved various aspects of polymer surface and interface science, metal and metal oxide surface chemistry,



Thomas J. McCarthy

polymers and supercritical fluids, and polymers and ionic liquids.

Gareth H. McKinley is the School of Engineering professor of teaching innovation within the Department of Mechanical Engineering at the Massachusetts Institute of Technology (MIT). He also is the director of the Program in Polymer Science and Technology at MIT, and head of the Hatsopoulos Microfluids Laboratory. He received his BA degree in 1985 and his MEng degree in 1986, as well as degrees from the University of Cambridge and his PhD degree in 1991 from the Chemical Engineering Department at MIT. He is a co-founder and member of the board of directors of Cambridge Polymer Group. McKinley’s research interests include extensional rheology of complex fluids, non-Newtonian fluid dynamics, microrheology, field-responsive fluids, super-hydrophobicity, and the development of nanocomposite materials.

Jeffrey S. Moore is the Murchison-Mallory Chair in Chemistry at the University of Illinois. He received his BS degree in chemistry in 1984 and his PhD degree in materials science in 1989—both



Gareth H. McKinley

from the University of Illinois. After postdoctoral studies at the California Institute of Technology and before returning to the University of Illinois in 1993, Moore began his independent career at the University of Michigan. Moore’s research now focuses on molecular self-assembly, macromolecular architecture, and self-healing polymers.

Michael F. Rubner is currently the TDK Professor of Polymer Materials Science and Engineering, within the Department of Materials Science and Engineering at the Massachusetts Institute of Technology (MIT), and the director of MIT’s National Science Foundation supported Center for Materials Science and Engineering. He received his undergraduate degree in chemistry from the University of Lowell, summa cum laude, in 1982. Rubner earned his PhD degree in polymer science from the Department of Materials Science and Engineering at MIT in 1986. While pursuing his undergraduate and graduate degrees, Rubner also worked as a full-time staff member in GTE Laboratories. His research interests include the molecular-level processing and electrical,



Jeffrey S. Moore

optical, and biomaterial property investigations of polymers and various nanomaterials.

Richard S. Trask can be reached at the University of Bristol, Department of Aerospace Engineering, Queen's Building, University Walk, Bristol, BS8 1TR, UK; tel. 44-117-337-7499, fax 44-117-927-2771, and e-mail R.S.Trask@bristol.ac.uk.

Trask is a research Fellow at the Department of Aerospace Engineering, University of Bristol, UK. He has also established himself as an expert in developing self-healing materials in a position at Bristol, after completing his PhD degree in 2004. After earning his BEng degree in materials science and engineering from the University of Bath in 1995, Trask was employed by the Defence Evaluation and Research Agency—now QinetiQ—culminating in a position as Technology Lead Air, Land, and Sea. After earn-



Michael F. Rubner

ing an MSc degree in advanced materials technology in 2000 from the University of Surrey, Trask became a research Fellow of the Royal National Lifeboat Institution at the University of Southampton in 2001, studying composite repair.

Anish Tuteja can be reached by e-mail at atuteja@mit.edu.

Tuteja is a postdoctoral research associate with the Department of Chemical Engineering at the Massachusetts Institute of Technology. He received his undergraduate degree in chemical engineering in 2001 from Panjab University, and his PhD degree in 2006 from the Department of Chemical Engineering and Materials Science at Michigan State University. His research interests include superhydrophobic and superoleophobic materials as well as the effects of nanoparticle addition on



Richard S. Trask

the properties of polymer melts.

Scott R. White is the Willet Professor of Engineering in the Department of Aerospace Engineering at the University of Illinois at Urbana-Champaign. White received a BS degree in mechanical engineering in 1985 from the University of Missouri-Rolla, an MS degree in mechanical engineering in 1987 from Washington University, St. Louis, Missouri, and a PhD degree in engineering science and mechanics in 1990 from The Pennsylvania State University. His research is focused on self-healing materials and microvascular materials systems.

Hugo R. Williams can be reached the University of Bristol, Department of Aerospace Engineering, Queen's Building, University Walk, Bristol.



Anish Tuteja



Hugo R. Williams

BS8 1TR, UK; tel. 44-117-331-7499, fax 44-117-927-2771, and e-mail Hugo.Williams@bristol.ac.uk.

Williams is a PhD degree student in the Department of Aerospace Engineering at the University of Bristol, UK. He acquired an MEng degree in aeronautical engineering at the University of Bristol in 2005 where he was the best student in his class. Williams' graduate studies are under the supervision of Ian P. Bond with his research focusing on the development of self-



Scott R. White



Kyle A. Williams

healing composite materials via the use of embedded vascular networks.

Kyle A. Williams is a graduate student at The University of Texas at Austin, working under the guidance of C.W. Bielawski. Williams earned a BS degree in chemistry in 2004 from Trinity University in San Antonio, Texas, where he worked in Nancy S. Mills' group. His graduate research focuses on the development of dynamic polymers. □



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