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FOCUS REVIEW

New developments in polymer brush fabrication: concepts and physical properties of dynamic polymer brushes

Hideaki Yokoyama[1]

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Abstract

In contrast to fabricating polymer brushes using conventional grafting-to and grafting-from techniques, we fabricated polymer brushes using a novel concept. This novel concept involves block copolymer segregation from the polymer region at the water interface. The resulting polymer brush is called a dynamic polymer brush because block copolymer segregation is a dynamic process that occurs at room temperature and is activated through contact with water. Dynamic polymer brushes undergo self-organization in the processes of self-assembly and self-healing (if destroyed). However, the analysis of dynamic polymer brushes is typically complicated because they are only detectable when in contact with water and change over time. Dynamic polymer brushes are not only a fascinating research topic but can also be used in many different fields, and the concept of dynamic polymer brushes is recognized as a core surface modification concept. The concept and physical characteristics of dynamic polymer brushes are discussed in this review.

What is a polymer brush?

Polymer chains have a coiled form in solution, and repulsive forces occur owing to the excluded volume effect. A polymer brush is a structure in which one end of a polymer chain is fixed to a solid surface/interface in a solvent [1]. Because polymer chains immobilized on a solid surface are strongly repelled but cannot be physically separated, they stretch in the vertical direction. These polymer chains stretch in a perpendicular direction toward the solid surface by repulsion, which is characteristic of polymer brushes.

Despite having a monolayered structure, polymer brushes have a thickness of several nanometers to several hundred nanometers because the polymer chain monolayer extends vertically from the solid surface and includes the solvent. By forming thin polymer brush layers, it is possible to decrease friction and abrasion resistance, reduce protein adsorption and cell adhesion, modulate wettability, and maintain catalytic activity without altering the bulk material or its physical properties.

Two primary conventional methods for fabricating polymer brushes are presented in the following. The grafting-to approach has been used extensively [2] and involves surface chemical reactions induced by terminal functional polymers or surface physical adsorption caused by block copolymers that immobilize polymer chains on a solid surface. Conversely, recent advancements in precision polymerization have led to the development of the grafting-from approach [3], in which an initiator is immobilized on a solid surface, resulting in polymer chain formation by polymerization. The grafting-to method is highly adaptable toward a variety of polymer species. However, it has the disadvantage of strong steric repulsion caused by repulsive interactions between polymer brush chains, which hinders the reaction/adsorption of new polymer chains to the solid surface, and the upper limit of the grafting density remains relatively low. The initiator of a small molecule is immobilized on the solid surface in the grafting-from procedure to increase the density. A less dense monomer is supplied to the surface during the subsequent chain-growth processes. Therefore, a high brush density is generally obtained using the grafting-from method. However, the chemical species of the brush chains that can be polymerized are limited because brush generation occurs through precise living polymerization. Using the grafting-from method, high-

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✉ Hideaki Yokoyama
hideaki@edu.k.u-tokyo.ac.jp

[1] Department of Advanced Materials Science, Graduate School of Frontier Sciences, The University of Tokyo, 603 Transdisciplinary Sciences Bldg, 5-1-5 Kashiwa-no-ha, Kashiwa, Chiba 277-8561, Japan

density brushes of 0.1 chains/nm^2 or higher with a narrow molecular weight distribution were realized. Notably, water-soluble polymer brushes are useful for biological and environmental applications [4–6].

What is a dynamic polymer brush?

In contrast to fabricating polymer brushes using conventional grafting-to and grafting-from techniques, we fabricated polymer brushes using a novel concept, which involves block copolymer segregation from the polymer region at the water interface. We call the resulting polymer brush a dynamic polymer brush because block copolymer segregation is a dynamic phenomenon that occurs at room temperature and is initiated through contact with water [7–14]. When the glass transition temperatures of polymers are lower than room temperature, a block copolymer freely diffuses in the system and spontaneously segregates to the interface when added to systems that contain an interface between two regions A and B, such as a homopolymer and air or homopolymers A and B, to reduce the interfacial energy. Macromolecular surfactants are amphiphilic block copolymers composed of both hydrophilic and hydrophobic building units. It is well known that amphiphilic block copolymers segregate in water/air and water/hydrophobic solids from the water region of the interface. One of the grafting-to procedures involves the adsorption of an aqueous solution onto a hydrophobic surface.

In contrast, the dynamic polymer brush method utilizes the phenomenon of water interface segregation from the polymer region of A-B amphiphilic block copolymers [1]. In the dynamic polymer brush method, a mixture of an elastomer composed of hydrophobic homopolymer A and an A-B amphiphilic block copolymer with a hydrophilic B block is used. In air, the surface of the mixture is more stable when hydrophobic homopolymer A, which has a lower surface energy in air, is exposed and the A-B amphiphilic block copolymer is not. However, when the mixture comes into contact with water, the A-B amphiphilic block copolymer segregates to the water interface, forming a brush that projects the hydrophilic B block into the water to decrease the interfacial energy. The use of an elastomer as the matrix and block components is essential for block copolymer segregation to the interface at room temperature. The hydrophilic B block extended into the water is considered a polymer brush, and because this brush dynamically formed by self-assembly at the water/elastomer interface of the block copolymer, it is called a dynamic polymer brush.

The fabrication of a dynamic polymer brush is much simpler than the standard approach because it involves the simple mixing of a small amount of copolymer with an elastomer matrix. Dynamic polymer brush systems can have various brush densities and degrees of elongation in response to external environmental changes. In contrast, the brush density is fixed for conventional polymer brushes, and only the elongation of the brush chain is variable. The dynamic polymer brush has a self-healing ability that causes the copolymer to resegregate in the matrix when the brush layer is lost, which is a major advantage compared to conventional brushes. Matrix homopolymers and block copolymers with low glass transition temperatures, which permit the diffusion of amphoteric block copolymers at room temperature, are necessary for the self-healing of brushes at ambient temperatures. We previously reported dynamic polymer brush systems using crosslinked polydimethylsiloxane (PDMS) or polystyrene-b-polyisoprene-b-polystyrene (SIS) as elastomers [7–14]. Although dynamic polymer brushes can be considered inverse grafting-to brushes, dynamic polymer brushes can have a higher brush density than conventional grafting-to brushes. A detailed discussion of the thermodynamics of dynamic polymer brushes is given in the following section.

Interfacial segregation of block copolymers and properties of dynamic polymer brushes

The brush density of a dynamic polymer brush system varies. The free energy of the brush chain is minimized by varying the brush density. Therefore, dynamic polymer brushes become dense when the thermodynamic driving force is large. Structural analysis of a dynamic polymer brush formed by a diblock copolymer system composed of polyethylene glycol (PEG) as the hydrophilic block and PDMS as the hydrophobic block was performed. Measuring the nanoscale structure of a water/elastomer interface in water is complicated. The only method to achieve this goal is neutron reflectivity (NR) in contrast-enhanced deuterated water, which allows a detailed analysis of dynamic polymer brush growth at the water/elastomer interface. The brush structure at the deuterated water/elastomer interface was analyzed, and the formation of a high-density brush (2.8 chains/nm^2) was reported (Fig. 1), which was the first report of the formation of a surprisingly high-density brush. Furthermore, with this dynamic polymer brush, the hydrophilic PEG chain was stretched to 80% of the chain length, indicating that a high-density, high-stretching brush was spontaneously formed [7]. A high-density, high-elongation brush structure was experimentally confirmed by NR measurements, but the fabrication of such a dense brush was unexpected because it involved an increase in the elastic energy required to stretch the polymer chain. Thus, we discuss the reason why dynamic polymer brushes can have

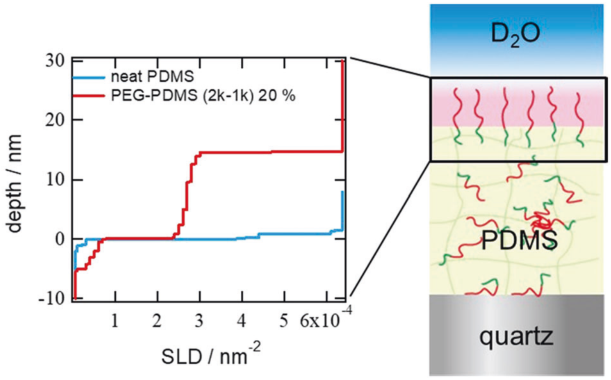


Fig. 1 Depth profiles of the scattering length density (SLD) of a dynamic polymer brush interface revealed by neutron reflectivity. Deuterated water (D_2O), which has the highest SLD value, was used to enhance the contrast of the brush. Reproduced from the data in reference [7]

high-density and high-elongation brush structures using simple theoretical free energy calculations.

By assuming a simple model in which the change in free energy owing to brush formation is represented by the sum of the hydration energy F_{int} of PEG and the extension energy F_{ent} of the polymer chain, the change in free energy owing to brush formation per unit area of the interface can be obtained. In this simplified model [7], it is considered that the hydration energy of PEG is the driving force that increases the brush density of PEG and balances the extension energy of the brush chain. The PEG hydration energy F_{int} can be calculated as a function of elongation R/R_{max} and brush density σ (chains/nm^2) using the experimental values of the hydration energy of PEG with a molecular weight of 2000. R and R_{max} represent the distance between the ends of the polymer chain (brush thickness) and the total contour length of PEG, respectively.

Regarding the elongation energy of the polymer chain, it is assumed that the elongation degree R/R_{max} and stress f of the polymer chain are described by the Langevin function L, including the effect of elongation.

$$\frac{R}{R_{\mathrm{max}}} = L\left(\frac{fb}{k_{\mathrm{B}}T}\right) = \coth\left(\frac{fb}{k_{\mathrm{B}}T}\right) - \left(\frac{fb}{k_{\mathrm{B}}T}\right)^{-1}, \tag{1}$$

where k_{B}, b, and T are the Boltzmann constant, segment length, and temperature, respectively.

The Langevin function shows that the stress changes linearly with the degree of elongation in the low-elongation region, representing linear elasticity. Thus, in this region, the polymer chain is considered to act as a spring according to Hooke's law. Furthermore, a rapid increase in stress occurs in the high-elongation region. This represents the stretching effect of the polymer chain with finite length R_{max}. The extension energy is obtained by integrating the inverse function of the Langevin function.

$$F_{\mathrm{ent}} = k_{\mathrm{B}}TN\left[\frac{R}{R_{\mathrm{max}}}L^{-1}\left(\frac{R}{R_{\mathrm{max}}}\right) - \ln\left\{\sinh\left(L^{-1}\left(\frac{R}{R_{\mathrm{max}}}\right)\right)\right\} + \ln\left\{L^{-1}\left(\frac{R}{R_{\mathrm{max}}}\right)\right\}\right] \tag{2}$$

The change in free energy per unit area is calculated as follows:

$$\Delta F = \sigma(F_{\mathrm{int}} + F_{\mathrm{ent}}). \tag{3}$$

Figure 2 shows the free energy mapping of the two types of copolymers used in the experiment based on Eq. (2).

Figure 2a shows the most stable state at an elongation of 72% and brush density of 1.8 chains/nm^2, and the high-density polymer brush measured by NR can be modeled. Figure 2b indicates an elongation of 61% and brush density of 1.5 chains/nm^2 for the longer PDMS anchor chain, which requires a larger extension energy to achieve the same increase in hydration energy. In the conventional grafting-to method, only the energy of the reaction or adsorption of the terminal group acts as a driving force. However, for dynamic polymer brushes, the relatively long hydrophilic chains in the dry state segregate at the water interface and acquire a much larger energy due to hydration energy. Thus, the conventional grafting-to brushes acquire energy through two-dimensional interactions of grafting points at the interface, whereas dynamic polymer brushes acquire energy through the three-dimensional volume interactions of entire hydrophilic chains with water. Therefore, dynamic polymer brushes have a remarkably high density.

The expected brush densities and elongations of brush chains in this thermodynamic calculation are reasonably close to the experimentally observed values but are slightly underestimated. This underestimation can be caused by the oversimplification of thermodynamic variables. Only the hydration energy and elastic energy of block copolymer chains are taken into account, whereas the osmotic pressure between the dynamic polymer brush and water is also important in determining the brush density and elongation. Although we did not calculate the contribution of osmotic pressure quantitatively, the osmotic pressure should increase the density and degree of elongation of brush chains.

Dynamic polymer brush formation dynamics and self-healing

The time required for brush formation is very important. Moreover, another characteristic property of dynamic polymer brushes is their self-healing property. When the surface is damaged, the brush is spontaneously reconstructed if the elastomer contains a sufficient amount of

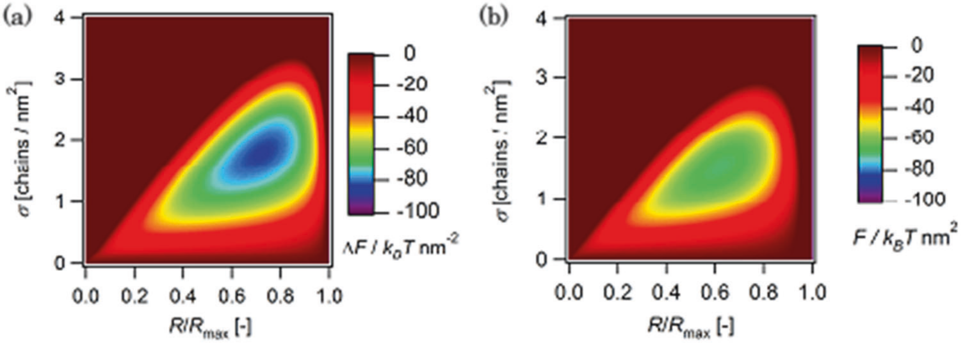


Fig. 2 Free energy maps in terms of the brush density σ and brush thickness R/R_{max} of **a** PEG-b-PDMS (molecular weight = 2100–1000) and **b** PEG-b-PDMS (molecular weight = 2100–5000). Reproduced from the data in reference [7]

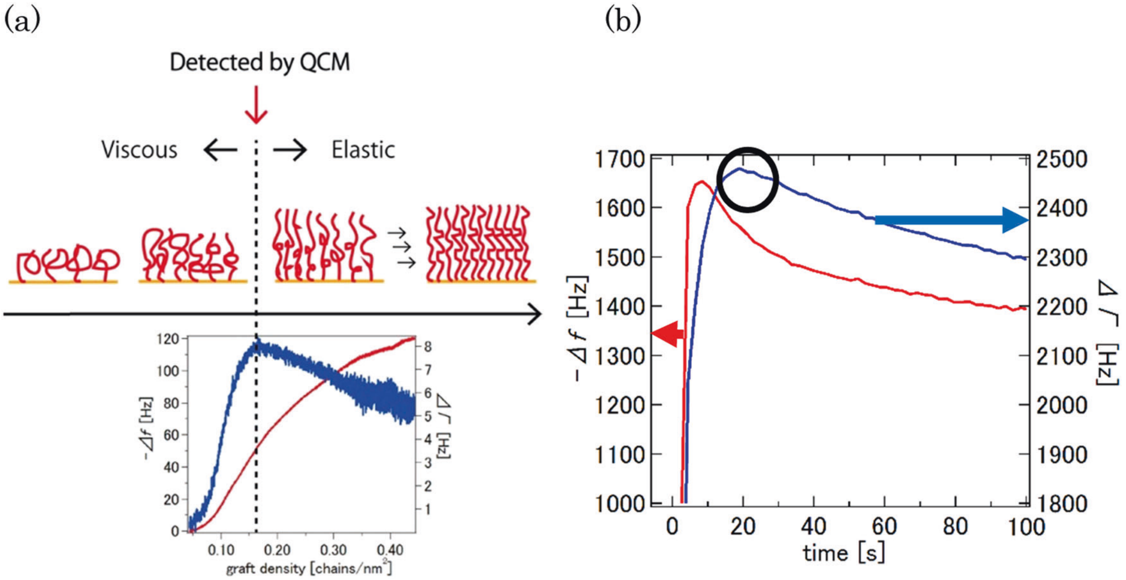


Fig. 3 **a** QCM calibration using the brush formation process of well-known grafting-to brushes to calibrate the frequency and structural changes of polymer brushes. The brush density dependence of the frequency change ($-\Delta f$) measured by QCM (red line) and the attenuation factor change $\Delta\Gamma$ (blue line) show a peak indicating the transition from a viscous liquid to an elastic solid. **b** Formation dynamics of a dynamic polymer brush composed of PEG-PDMS (Mn = 2100–5000) (ED2-5) as an amphiphilic diblock copolymer measured by a QCM. The $-\Delta f$ and $\Delta\Gamma$ values after soaking a film with 10 wt% ED2-5 added to the PDMS homopolymer are shown as a function of time. Reproduced from the data in reference [8, 9]

copolymer in the bulk. This is a very important property for the applications of dynamic polymer brushes.

As previously discussed, the equilibrium structure of the dynamic polymer brush can be quantitatively analyzed by the NR method, but the brush formation mechanism generally cannot be analyzed by NR. This is because the formation rate of dynamic polymer brushes is faster than the time scale of NR measurements. Quartz crystal microbalance (QCM) analysis was performed as an alternative method to detect the formation of dynamic polymer brushes with high time resolution; however, it is not a quantitative method [8, 9]. QCMs have generally been used to detect minute mass changes, but the QCM response becomes complicated when a flexible layer, such as a brush, is formed in a liquid. QCMs do not respond to the mass of the brush layer but rather to the many mechanical parameters, such as the mass, viscosity, elasticity, and thickness, of the brush layer. The dynamic polymer brush system is very complicated, with a four-layer structure comprising quartz, elastomer, brush, and water. Before elucidating the unknown dynamic polymer brush formation kinetics, QCM measurements of a brush formation process were calibrated using well-known grafting-to brushes. Using the QCM measurements of grafting-to brushes, the frequency and structural changes of the polymer brushes were calibrated [8]. Fig. 3a shows the brush density dependence of the frequency change -Δf obtained through QCM measurements and the attenuation factor change $\Delta\Gamma$, indicating the contribution of viscosity.

In the grafting-to method, -Δf increased monotonically, which was unexpected. The QCM responded to the change in mass of the added grafting-to brushes. Note that in the dynamic polymer brush system, the change in mass is zero because the block copolymer simply moves from inside to the water interface. Therefore, we avoided comparing the $-\Delta f$ values of grafting-to and dynamic polymer brushes. At a brush density of 0.17 chains/nm^2, $\Delta\Gamma$ exhibited a peak. A detailed viscoelastic analysis showed that this peak indicates a transition from a low-density mushroom or brush with a viscous response to a high-density brush with an elastic response [8]. This result was used to analyze the formation of dynamic polymer brushes, by which a $\Delta\Gamma$ peak was obtained from the QCM measurement, as shown in Fig. 3b [9].

Figure 3b shows the $-\Delta f$ and $\Delta\Gamma$ values after soaking a film with 10 wt% ED2-5 added to the PDMS homopolymer as a function of time. For the film with 10 wt% ED2-5, $-\Delta f$ and $\Delta\Gamma$ both exhibited a peak. In particular, the $\Delta\Gamma$ peak was similar to that in the calibration experiment using the grafting-to brush, corresponding to the transition from a viscous brush to an elastic brush. This transition occurred ~20 s after immersion in water. Therefore, the dynamic polymer brush formed a dense elastic brush-like hydrogel within ~20 s. The self-healing period was considered identical to the initial formation time, which was sufficiently short to achieve self-healing.

Prevention of adhesion and reduction of interfacial energy by dynamic polymer brushes

Interfacial energy is an essential physical criterion for defining the characteristics of interfaces. However, because many polymer surfaces adapt to liquids and show complex dynamics in terms of the contact angle, it is very difficult to evaluate the interfacial energy of polymer-liquid interfaces [10].

Rebuilding is activated only beneath the droplets in contact angle measurements, forming an uneven surface, as shown in Fig. 4. Thus, it is not possible to describe the interfacial energy using generic contact angle approaches because Young's equation can only be applied to and interfacial energy can only be defined for a uniform surface at equilibrium. As previously stated, a dynamic polymer brush has a complex reconstructive surface. The interfacial energy is expected to decrease significantly with brush development. We developed a novel approach for detecting the interfacial energy of a reconstructive elastomer surface by monitoring the deformation of ultrathin square elastomer films floating on water and analyzing the dynamic polymer brush interface, as shown in Fig. 5. Considering that the deformation is sufficiently small to be described by the neo-Hookean model, the relationship between the stress σ and elongation ratio λ can be described as $\sigma = 2E(\lambda - \lambda^{-5})/3$, where E is the Young's modulus of the elastomer. The forces acting on the elastomer thin films floating on water are the surface energies of water (γ_L) and the elastomer (γ_S) and the interfacial energy between the elastomer and water (γ_{SL}). Because the film thickness d is very small compared to the magnitude of the planar direction, the stress σ can be simply described as $\sigma = (\gamma_L - \gamma_S - \gamma_{SL})/d$. Because γ_L and γ_S are known, γ_{SL} can be evaluated from the change in λ.

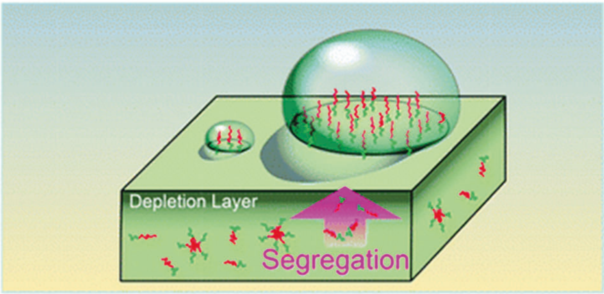


Fig. 4 Dynamic polymer brush formation under water droplets. Contact angle analysis cannot be performed precisely because of heterogeneous segregation. Reproduced from reference [10]

SIS with 14 wt% PS (SIS14) and poly(1,4-isoprene)-b-poly(ethylene glycol) (PI-b-PEG) were used as a hydrophobic elastomer and an amphiphilic block copolymer, respectively. The molecular weights of the two types of PI-b-PEG used were 6500-b-16500 g/mol (I6E16) and 18500-b-6500 g/mol (I18E6). For thin SIS14 films with I18E6 and shorter hydrophilic PEG chains, the interfacial energy decreased slightly but remained positive owing to a small increase in hydration energy induced by the formation of low-density and collapsed brushes (Fig. 6a). However, the interfacial energy decreased considerably to a large negative value for the thin SIS14 films containing I6E16 and longer PEG chains because of the significant increase in hydration energy induced by the formation of highly dense and stretched brushes (Fig. 6b). We successfully measured the interfacial energy of a reconstructive dynamic polymer brush, revealing its unique negative interfacial energy.

The highly hydrophilic nature of the dynamic PEG brush is indicated by its large negative interfacial tension at the water interface. These hydrophilic polymer brushes should have enhanced antifouling properties and resistance to hydrophobic substances. Preventing hydrophobic interactions in water is crucial for facilitating antifouling properties. Atomic force microscopy and a hydrophobic probe were used to analyze the hydrophobic interactions of a dynamic polymer brush in water, as shown in Fig. 7 [12]. The force-distance curve between the hydrophobic probe and elastomer thin film in water at approximately 25 °C was measured to determine the adhesion forces. The force required to remove the probe from the film surface is referred to as the adhesive force. According to Johnson–Kendall–Roberts theory, the adhesion force is defined as $F_{ad} = -(3/2)\pi RW$, where F_{ad} is the adhesion

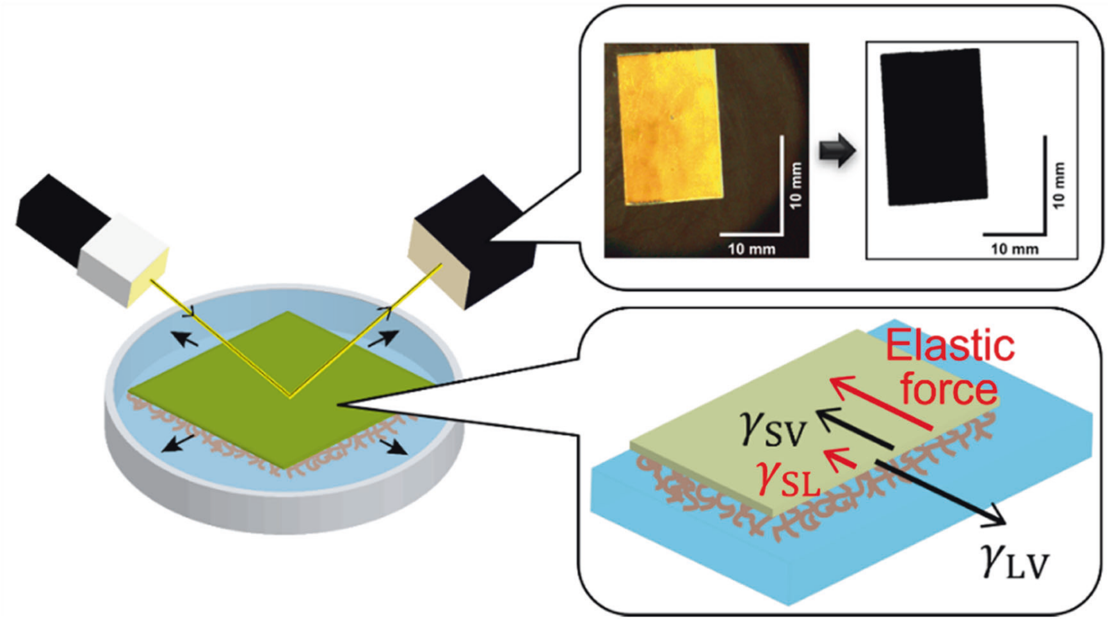


Fig. 5 A novel method for detecting the interfacial energy of a reconstructive elastomer surface by monitoring the deformation of ultrathin square elastomer films floating on water and analyzing the dynamic polymer brush interface. Reproduced from reference [11]

Fig. 6 SIS with 14 wt% PS (SIS14) and poly(1,4-isoprene)-*b*-poly(ethylene glycol) (PI-*b*-PEG) were used as a hydrophobic elastomer and an amphiphilic block copolymer, respectively. Two types of PI-*b*-PEG were used with molecular weights of 18500-*b*-6500 g/mol, I18E6 in **a**, and 6500-*b*-16500 g/mol, I6E16 in **b**. Reproduced from reference [11]

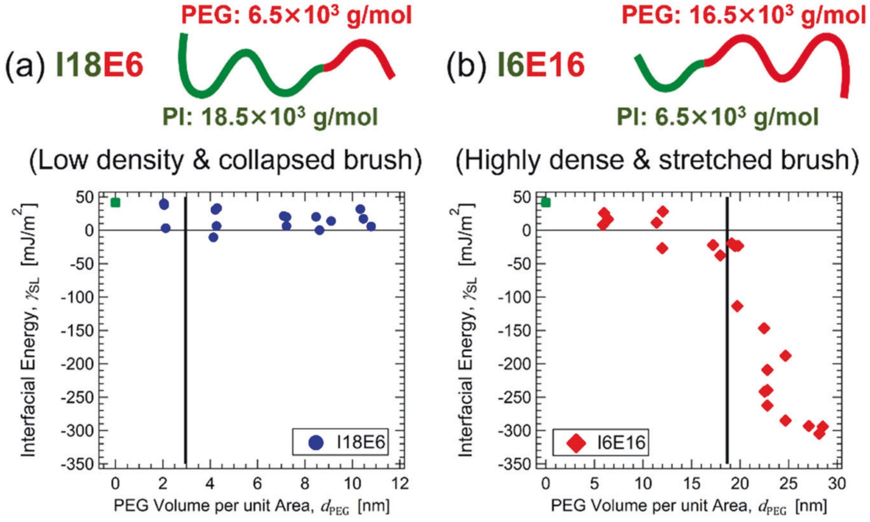
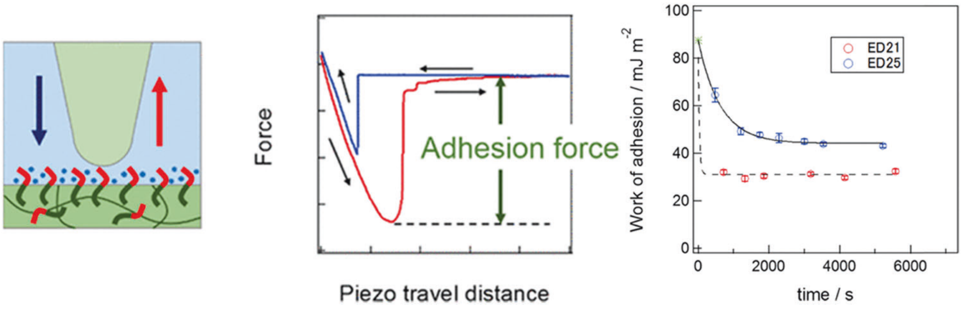


Fig. 7 Adhesion forces were calculated by measuring the force−distance curve of a hydrophobic probe and an elastomer thin film in water at approximately 25 °C. Reproduced from reference [12]



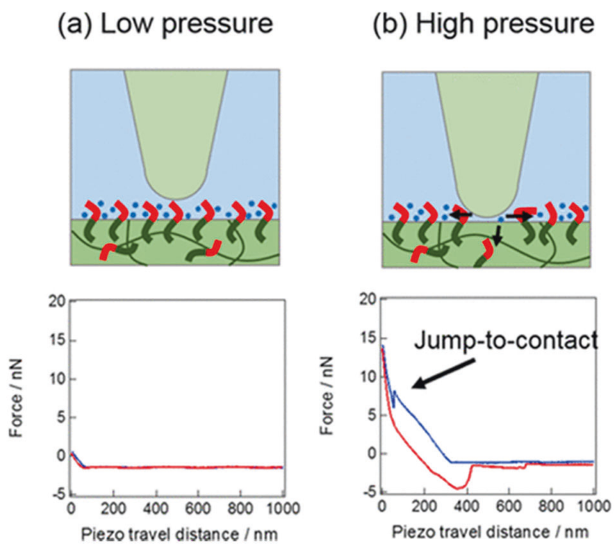


Fig. 8 Schematics of dynamic polymer brushes and examples of the typical force curves measured under **a** low pressure and **b** high pressure for a PDMS elastomer with ED21(PEG-b-PDMS, Mn = 2100–1000) with a graft density of 2.8 chains/nm^2. Reproduced from reference [12]

force, *R* is the radius of the probe, and *W* is the work of adhesion, which is influenced by the interfacial energies between the probe environment, sample film, and environment, as well as between the probe and sample film.

Reduced hydrophobic interactions were observed with the dynamic polymer brushes, which were more visible at higher graft densities. Additionally, the dynamic polymer brush exhibited a distinct transitional response to applied pressure. As shown in Fig. 8, the adhesion force was nearly negligible at low applied pressures and increased with increasing applied pressure. At low applied pressures, a hydrophobic substance did not adhere to the dynamic polymer brush; however, adhesion occurred at high applied pressures. Pressure-sensitive adhesion is one of the distinctive properties of a nonbound dynamic polymer brush and may indicate the reallocation or retraction of the block copolymer brush chains from the contact area induced by the applied pressure.

Summary

The concept of polymer brushes has been used extensively and is anticipated to be useful in a variety of applications. However, the structural analysis of polymer brushes is complicated because they exist in a very thin layer at the interface. Moreover, polymer brush research requires sophisticated equipment and analytical techniques. Therefore, there is limited understanding of their structure and physical properties. The analysis of the dynamic polymer brush, a new polymer brush that utilizes self-organization, is even more difficult because it is only detectable through contact with water and changes over time. In Japan, the joint use of neutron facilities has recently begun at the Japan Proton Accelerator Research Complex, and the quantitative analysis of dynamic polymer brushes has advanced significantly. Dynamic polymer brushes grow independently and self-heal if they become damaged. Dynamic polymer brushes decrease the interfacial tension of water to negative values. Typically, a negative interfacial tension destabilizes the interface, but the matrix of the crosslinked elastomer stabilizes the dynamic polymer brush. The superhydrophilic dynamic polymer brush with negative interfacial tension exhibits an exceptionally low hydrophobic attractive interaction in water and has superior antifouling properties. Additionally, the attractive hydrophobic interactions of the dynamic polymer brush produce novel pressure-dependent behavior, which may be due to the mobility of the block copolymer chains under high pressures. The dynamic polymer brush concept can be further extended to a variety of amphiphilic macromolecules, such as thermoresponsive polymer brushes [13] and polyrotaxane brushes [14]. In the future, these unique properties may be applied in a variety of fields and are accepted as a fundamental surface modification concept.

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Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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Hideaki Yokoyama received B.Eng. and M.Eng. degrees in Polymer Chemistry from Tokyo Institute of Technology, as well as M.S. and Ph.D. degrees in Materials Science and Engineering from Cornell University, Ithaca, NY, USA. Following his professional careers at Bridgestone Corporation and National Institute of Advanced Industrial Science and Technology, he joined his current position as Associate Professor in the Department of Advanced Materials Science, Graduate School of Frontier Sciences, The University of Tokyo, Japan. He has been investigating the self-assembly, diffusion, surface, and interface of block copolymers and their applications. He is a recipient of the Wiley Award and the Mitsubishi Chemical Award of the Society of Polymer Science, Japan.