



Repeatable adhesion by proton donor-acceptor interaction of polymer brushes



Hayato Yoshioka^a, Chiharu Izumi^a, Miki Shida^b, Kazuo Yamaguchi^b,
Motoyasu Kobayashi^{b,*}

^a Graduate School of Engineering, Kogakuin University, 2665-1 Nakano-cho, Hachioji, Tokyo 192-0015, Japan

^b School of Advanced Engineering, Kogakuin University, 2665-1 Nakano-cho, Hachioji, Tokyo 192-0015, Japan

ARTICLE INFO

Article history:

Received 21 February 2017

Received in revised form

8 May 2017

Accepted 14 May 2017

Available online 15 May 2017

Keywords:

Polymer brushes

Adhesion

Donor-acceptor interaction

Hydrogen bonding

Interface

Surface-initiated ATRP

ABSTRACT

Polymer brushes having proton donor and acceptor type functional groups were prepared on silicon wafers by surface-initiated atom transfer radical polymerization of 2-hydroxyethyl methacrylate (HEMA), 4-hydroxystyrene (4HS), 2-vinylpyridine (2VP), and 4-vinylpyridine (4VP), respectively. Two silicon wafers, one prepared with a poly(HEMA) brush and one with a poly(4VP) brush, were jointed in a 1-cm² contact area using 5- μ L of methanol under a pressure of 120 N at room temperature and successively air-dried to form hydrogen bonds at the brush interface, yielding a lap shear adhesion strength of 337 kPa. Furthermore, poly(4HS) and poly(4VP) brushes also adhered more firmly with 5- μ L of methanol to yield a lap shear adhesion strength of 843 kPa due to the phenolic OH group with a larger dipole moment compared to aliphatic OH. In addition, the adhered specimens smoothly debonded in methanol within 20 min at 298 K. The two separated substrates adhered again with a small amount of methanol. Repeatable adhesion and debonding was successfully achieved by proton donor and acceptor type polymer brushes.

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1. Introduction

A hydrogen bond exists between two electronegative atoms or groups, one of which has a hydrogen atom covalently bound to it [1]. The negatively charged molecules work as proton acceptors, and the molecules connected to the hydrogen are proton donors. The size of the hydrogen atom is so small that strong Coulombic forces effectively cause intermolecular interaction. Therefore, the energy of a hydrogen bond is higher than that of intermolecular bonds formed by van der Waals interaction including dipole-dipole, dipole-induced dipole, and dispersive forces. This interaction plays an important role in forming a higher-order structure in natural polymers, such as in the double helical structure of DNA and the protein folding process [2]. Hydrogen bonding of catecholic OH groups containing the adhesive protein plays an essential role in the adhesion of mussels on various inorganic and organic materials [3,4]. In addition, hydrogen bonding has been widely used for preparing highly ordered, self-assembling artificial supramolecular

polymers [5–8], and self-healing hydrogels [9–11].

The strength of a hydrogen bond is sensitive to environmental conditions, such as the pH, solvent quality, ionic strength in solution, and temperature. Sudre et al. investigated systematically the effect of pH and temperature on hydrogen bonding between poly(acrylic acid) and hydrogen-acceptor poly(*N,N*-dimethylacrylamide) by cloud point measurement of dilute solution using LCST behavior of interpolymer complex of these polymers [12]. The formation of interpolymer complexes based on hydrogen bonding as the main driving force have been studied by gravimetry [13], potentiometry [13], turbidimetry [14–16], viscosimetry [16–20], fluorescence study [19,20], light scattering [21], infrared [15], and NMR spectroscopies [22]. Therefore, hydrogen bonding is expected to be a useful reversible interaction for macroscopic adhesion. Creton and colleagues investigated the hydrogen bonding interaction between an acrylamide gel surface and pH-sensitive poly(acrylic acid) brushes on a planar surface under water in order to understand the effect of pH, contact time, and debonding velocity on adhesion [23]. This research was the fundamental application of a hydrogen bonded complex to macroscopic adhesion. Polymer brushes are surface-tethered polymers of which chain ends are immobilized on solid surfaces through covalent

* Corresponding author.

E-mail address: motokoba@cc.kogakuin.ac.jp (M. Kobayashi).

bonding, so that the brush chains cannot be released from the substrate even in a solution. Surface physicochemical properties are tunable by a functional group attached to polymer brushes. Therefore, polymer brushes provide a very useful technique and attractive interface for studying the effect of intermolecular interaction, such as electrostatic interaction, hydrogen bonding, and van der Waals interaction between functional molecules, on the macroscopic adhesive interaction between polymeric materials.

Adhesion by polymer brush interface, based mainly on electrostatic interaction, has been reported in a number of studies. Cohen Stuart et al. measured the electrostatic attractive interaction between oppositely charged polymer brushes in water by scanning probe microscopy (SPM) to investigate the effect of ionic strength in aqueous solution on the adhesive interaction between negatively charged and positively charged brushes [24]. LaSpina et al. investigated the pH-sensitive adhesion of poly(*N,N*-dimethylaminoethyl methacrylate) (Poly(DMAEMA)) brushes on a poly(methacrylic acid) (Poly(MAA)) gel [25]. Rafrari et al. directly measured adhesive and friction force between poly(DMAEMA) and poly(MAA) brushes in various pH aqueous solution by SPM using poly(MAA)-grafted cantilever [26]. Takahara and colleagues demonstrated repeatable adhesion utilizing the electrostatic attractive interaction between two substrates bearing oppositely charged polyelectrolyte brushes using a small amount of water and successive air drying [27]. Dipole-dipole interaction between sulfobetaine type zwitterionic polymer brushes has also been applied to unique repeatable adhesion [28] and adhesion between different materials [29,30].

The present study focused on hydrogen bonding as the interaction force between polymer brushes. We demonstrated repeatable adhesion by hydrogen bonding between two polymer brushes, one having a proton donor functional group and the other one with a proton acceptor group. It is well known that poly(4-hydroxystyrene) (poly(4HS)) and poly(vinylpyridine) form an interpolymer complex due to the strong attractive interaction between the proton donor phenolic OH group and proton acceptor nitrogen of pyridine groups [31]. Dai et al. also reported that poly(4-vinylpyridine) (poly(4VP)) shows a stronger interaction with poly(4HS) compared to that of poly(2-vinylpyridine) (poly(2VP)). Template polymerization using the strong interaction between poly(2VP) and methacrylic acid has also been reported previously [32]. Therefore, in the present study, poly(4VP) and poly(2VP) brushes were prepared on silicon wafer as proton acceptor substrates. On the other hand, poly(4HS) and poly(2-hydroxyethyl methacrylate) (poly(HEMA)) were used for proton donor type polymer brushes, as shown in Fig. 1. Then, two substrates bearing proton donor and acceptor type polymer brushes were jointed with a small amount of water or methanol. The adhesion strengths of phenolic OH of poly(4HS) and aliphatic OH of poly(HEMA) were compared by performing a lap shear adhesion test of the jointed substrates bearing proton donor and acceptor polymer brushes. In addition, the effect of solvent quality on the swollen brush structure and adhesion strength are discussed in this report.

2. Experimental

2.1. Materials

Copper (I) bromide (CuBr, Wako Pure Chemicals, 99.9%) was purified through successive washes with acetic acid and ethanol and then dried under vacuum. Ethyl 2-bromoisobutylate (RBr, Tokyo Chemical Industry (TCI), 98%) was dried and distilled over CaH₂ before use. Commercially available copper (II) bromide (CuBr₂, Wako Pure Chemicals, 99%), calcium hydride (CaH₂, Nacalai Tesque, Tokyo), sodium hydroxide (NaOH, Kishida Chemical Co.), 2,2'-bipyridyl (bpy, Wako Pure Chemicals, 99.5%), 2-propanol (TCI,

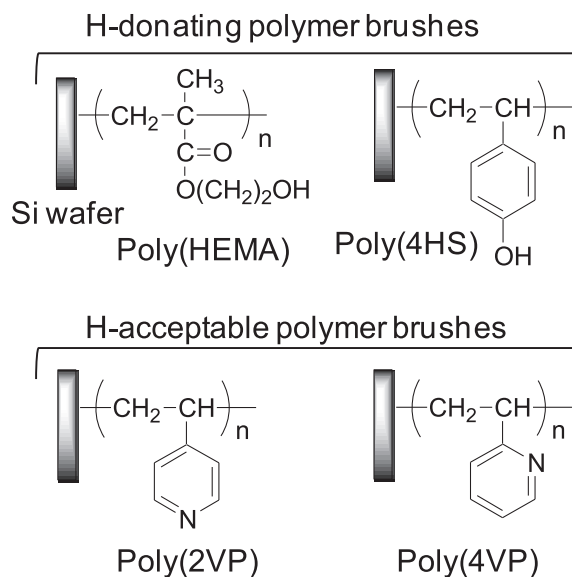


Fig. 1. Chemical structure of the polymer brushes.

99.5%), toluene, dehydrated (Kanto Chemical Co., 99.5%), formic acid (Wako Pure Chemicals, 97%), formaldehyde solution (Yoneyama Chemicals, 35%), hydrochloric acid (HCl, Wako Pure Chemicals, 35.0–37.0%), hydrogen peroxide (H₂O₂, Kanto Chemical Co., 34.5%), sulfuric acid (H₂SO₄, Kanto Chemical Co., 95.0%), tris(2-aminoethyl)amine (TCI, 98.0%), 5-hexen-1-ol (TCI, 95%), 2-bromo-2-methylpropionyl bromide (Aldrich, 98%), tin(II) 2-ethylhexanoate (Wako Pure Chemicals, 90.0%), 2-hydroxyethyl methacrylate (HEMA, Wako Pure Chemicals, 95.0%) and 1-chloroethylbenzene (RCl, Wako Pure Chemicals, 98%) were used without additional purification. Triethylamine was purchased from Kanto Chemical Co. and purified by distillation with CaH₂. Tris(2-dimethylaminoethyl)amine (Me₆TREN) was synthesized from tris(2-aminoethyl)amine and formic acid in formaldehyde solution and purified by distillation with CaH₂ under reduced pressure. 5'-Hexenyl 2-bromoisobutylate was synthesized from 5-hexen-1-ol and 2-bromo-2-methylpropionyl bromide, and purified by distillation with CaH₂ under reduced pressure. The surface initiator, (2-bromo-2-methyl)propionyloxy hexyltrimethoxysilane (BHM) was synthesized by hydrosilylation of 5'-hexenyl 2-bromoisobutylate treated with trimethoxysilane in the presence of a Karstedt catalyst. The BHM monolayer was immobilized on silicon wafer (10 × 40 × 0.5 mm³) in dry toluene at 298 K for 4 h. Deionized water was purified using the Direct-Q UV3 system (Merck Millipore, Inc.). Copper(I) chloride (CuCl, Wako Pure Chemicals) was purified by washing with acetic acid and ethanol and then dried under vacuum. *N,N,N',N'*-Pentamethyldiethylenetriamine (PMDETA) was purchased from Aldrich and used without further purification. Anisole, methanol, tetrahydrofuran (THF) and sodium hydrogen carbonate (NaHCO₃) were purchased from Wako Pure Chemicals and used as received. *N,N*-dimethylformamide (DMF, Kanto Chemical Co., 99.7%) was used as received. 4-Acetoxy styrene (4AS, Aldrich, 96%), 2-vinylpyridine (2VP, Aldrich, 97%) and 4-vinylpyridine (4VP, Wako Pure Chemicals, 90.0%) were purified by distillation with CaH₂ under reduced pressure. Water for the contact angle measurement was purified with the Direct-Q UV3 system (Merck Millipore, Inc.). The silicon (111) wafer (diameter = 100 ± 0.5 mm², thickness = 500 ± 25 μm, Matsuzaki Seisakusho Co., Ltd.) was cleaned by washing with piranha solution (H₂SO₄/H₂O₂ = 7/3, v/v) at 373 K for 1 h.

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