

Polymer Nanowire Arrays With High Thermal Conductivity and Superhydrophobicity Fabricated by a Nano-Molding Technique

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High thermal conductivity is helpful for thermal control and management, and superhydrophobicity can benefit fluid friction reduction and liquid droplet control in micro-/nanodevices. We report on a nano-molding technique that can prepare polyethylene nanowire arrays with high thermal conductivity (more than 10 W/m-K) and superhydrophobicity (contact angle >150°). The thermal conductivities of the fabricated high-density polyethylene nanowire arrays with diameters of 100 nm and 200 nm, measured by a laser flash method, are about 2 orders of magnitude higher than their bulk counterparts. The estimated thermal conductivity of a single high-density polyethylene nanowire is as high as 26.5 W/m-K at room temperature, while the thermal conductivity of low-density polyethylene nanowire is a little smaller. The self-organized surfaces of polymer nanowire arrays are found to have micro-to-nanoscale hierarchical nanostructures, and have superhydrophobicity of greater than 150° contact angles for water. We also measure the wettability of organic liquids, including glycerin, ethanol, paraffin liquid, and methyl silicone oil. We find glycerin gives hydrophobic wettability, but the others give hydrophilic wettabilities. This technique is promising for fabrication due to the advantages of simple fabrication, high quality, low cost, and mass production.

INTRODUCTION

Polymer nanostructures have drawn much attention in recent years for their potential applications in the fields of electronics, mechanical, biomedical, and fluidic micro-/nanodevices due to their unique properties [1–3]. Interfacial materials made from

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polymer nanostructures were tried to control the solid–solid interfacial thermal conductance [4–6]. However, that polymers generally have very low thermal conductivity of 0.1–1 W/m-K and the thermal conductivities of nanostructures are further limited by boundary phonon scattering does not match the requirement for the ultrahigh-density heat dissipation in most nanodevices yet [7]. Filling polymers with additives with high thermal conductivity, such as metallic nanoparticles or carbon nanotubes, and constructing heat transport networks are typical ways to increase their thermal transport [8, 9]. The enhancement of the thermal conductivity of such nanocomposites, however, remains much lower than theoretical expectations because of the high thermal contact resistance at polymer–additive interfaces [10, 11] and the decrease of the additives’ thermal conductivity affected by surroundings [12, 13]. Therefore, increasing the intrinsic thermal conductivity of polymer nanostructures is highly desired.

Polymers will exhibit significant anisotropy in thermal conductivity when the polymer chains are partially oriented [14]; that is, the thermal conductivity can be enhanced along the orientation direction. Fundamentally, molecular dynamics simulations showed that a single chain or aligned chains might have extremely high thermal conductivity, ~ 350 W/m-K or even divergent with chain length [15], since the heat transports along individual polymer chains are in a ballistic way [16, 17]. This has also been experimentally confirmed recently by a femtosecond laser pulse technique [18]. Under normal conditions, the random orientation of polymer chains with very weak couplings between them in amorphous polymers or amorphous-phase semicrystalline polymers shortens the mean free path of phonons, and results in the low thermal conductivity. If the polymer chains are aligned in a certain direction by spinning [19] or draw [20–22], the ballistic transport of heat will be increased and thus the thermal conductivity along this direction is greatly enhanced [23]. Drawn or spun polymer bulk structures were found to be able to give thermal conductivities of up to 50 W/m-K [19–22].

Shen et al. first applied this idea to fabricate gel-spun and ultra-drawn polyethylene (PE) nanofibers with very high thermal conductivity, ~ 104 W/m-K [24]. This was attributed to the restructuring of the polymer chains by stretching that improved the nanofiber quality from a random orientation toward an ideal single crystalline fiber. It should be mentioned that there is still great challenge in uniform and massive production for ultra-drawn nanowires. Cao et al. then developed an improved wetting template technique to fabricate high-density polyethylene (HDPE) nanowire arrays, which have also high thermal conductivity over 20 W/m-K at room temperature [25]. The nano-molding technique is quite promising because of its advantages in simple but massive, high-quality and low-cost production. We know the studies about the enhancement of the thermal conductivity of polymer nanostructures remain very limited thus far. This article reinforces the measured thermal properties of low-density polyethylene (LDPE) nanowire arrays, as well as the superhydrophobicity of the nanowire array surfaces.

Superhydrophobic surfaces are also highly desired in the fields of micro-/nanoscale heat transfer and fluid flow. As for the heat transfer, the small tension of a superhydrophobic surface may greatly decrease the interaction between a liquid droplet and the solid so that the superhydrophobic surface can supply anti-icing or anti-dewing properties [26, 27], control the solid-liquid thermal resistance [28–30], and produce a very high boiling heat transfer coefficient [31]. In addition, liquid flow and droplet control in micro- and nanochannels are widely used in recently advanced micro-/nanoelectromechanical systems (MEMS/NEMS) and biochip systems [32–34]. Surface effects substantially dominate the fluid flow due to the high surface-to-volume ratio in such micro- and nanoscale devices. Recently quite a few literatures have been published to show that liquids flowing over a solid surface do slip and the no-slip boundary condition is merely an approximation at macroscopic scale. The velocity slip of liquid flows at a solid surface has been measured experimentally and simulated by molecular dy-

namics simulations as reviewed in reference [34]. Wettability of a surface is shown to be one of the dominant factors. A hydrophobic surface can effectively enhance the velocity slip, and consequently decrease the flow friction [34–38].

This was originally inspired by the unique property of superhydrophobicity of lotus leaves [39] and water-strider legs [40], and specially called the “lotus effect,” and now researchers try to produce superhydrophobic surfaces through nanostructuring them. Traditional ways mainly include creating rough structures on a hydrophobic surface and modifying a rough surface by materials with low surface free energy. Various processes have been developed to fabricate micro-/nano-patterns with superhydrophobicity based on silicon, metal, glass, carbon nanotubes, polymer, or organic coatings [41, 42]. Among these technologies, the micro-/nano-molding method has advantages of simple fabrication, high quality, low cost, and mass production [43, 44], especially for micro-/nanoscale fluidic and heat transfer devices. Most previous studies about surface wettability were toward water, though various inorganic and organic liquids are often used in micro-/nanofluidics devices [45–47]. Here we report on the wettability of water, glycerin, ethanol, paraffin liquid, and methyl silicone oil over the PE nanowire array surfaces.

In this article we develop a nano-molding technique to fabricate HDPE and LDPE nanowire arrays with diameters of 100 nm and 200 nm. High thermal conductivity and superhydrophobicity of the nanowire arrays are found through experimental measurements. The thermal conductivity of the as-prepared nanowire arrays is measured by a laser flash method. The arrays have high thermal conductivity of about two orders higher than that of the bulk material. The underlying mechanisms for the high orientation of polymer chains are also demonstrated. The surface shows superhydrophobicity for water and glycerin, but increased hydrophilicity for ethanol, paraffin liquid, and methyl silicone oil, which is attributed to the self-organized and hierarchical surface nanostructures. Theoretical analyses based on the Cassie model are also presented.

FABRICATION OF PE NANOWIRE ARRAYS

The nanoporous template wetting technique, originally developed by Steinhart et al. [48], is now improved to enhance the polymer infiltration into the nanopores by a high-frequency fluid pulsation strategy, as schematically shown in Figure 1. The porous anodic alumina (PAA) templates with pore diameters of 100 nm and 200 nm are purchased from Whatman, Inc. The PAA templates are free-standing disks with a diameter of 13 mm, and their pores are all through-hole. The PE films with thickness of about 300 μm are obtained from Qilu Petroleum and Chemical Co. of China. More details about the fabrication system can be found in our previous articles [25, 49, 50]. First, the PAA templates are treated with solvents of different polarities including ethanol, acetone, chloroform, and hexane. Second, the PE film is placed on the top of a template with a good contact.

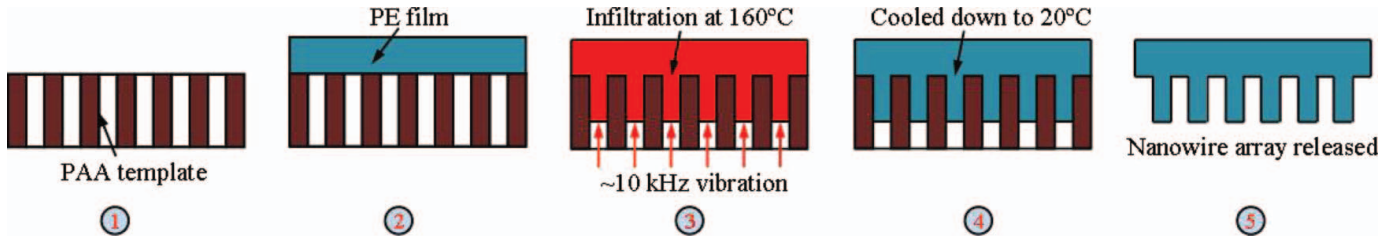


Figure 1 Fabrication procedure of the nano-injection molding technique. (1) Template; (2) sample including PE film and template; (3) infiltration; (4) solidification; (5) nanowire array releasing. (Color figure available online.)

Third, the sample is placed into an oven, and the chamber is then heated to 160°C to excite the infiltration of the PE melts into the nanopores of the template. During the infiltration process, a vibration with a frequency about ~10 kHz induced by a piezoelectric transducer is imposed. The vibration does help the polymer chains to be more oriented due to the oscillatory shear rates [51]. This process generally takes about 1 h to produce PE nanowire arrays with about 50 μm thickness. After that, the sample is taken out of the hot chamber and cooled to ambient temperature. The PE nanowire arrays are finally released by removing the template in NaOH aqueous solution and being rinsed with deionized water and ethanol and being dried at 30°C in vacuum in sequence.

The cross-section and top-view images, characterized by scanning electron microscopy (SEM, Leica Stereoscan 440) and field-emission scanning electron microscopy (FE-SEM, JEOL JSM-6335F), of the as-fabricated HDPE nanowire array are shown in Figure 2, in which the nanowire array with diameter of 200 nm is taken as a case study. The nanowires are of high quality, such as well-defined, straight, smooth in surface, and uniform in diameter, thanks to the good PAA templates. We do energy-dispersive x-ray spectroscopy analysis to make sure there is no residue alumina left in the film. The whole sample for thermal conductivity measurements consists of two layers with inhomogeneous properties. One is the substrate layer that solidifies from the redundant PE melts outside of the templates. The other is the nanowire array layer. By measuring the SEM images, we can obtain the thicknesses of the overall sample L_0 , the substrate layer L_s , and the nanowire array layer L_{nw} ($L_0 = L_s + L_{nw}$). One of the most important points is that there is no thermal contact resistance between the two layers, which enables the accurate extraction of thermal conductivity of the nanowire array from measuring the thermal conductivity of the whole sample.

THERMAL CONDUCTIVITY

A laser flash technique is employed to measure the thermal conductivity of the PE nanowire arrays. The samples are placed in a vacuum chamber with a pressure of about 0.1 Pa to reduce the heat losses. A nanosecond laser pulse with a pulse width of 6 ns and a wavelength of 1064 nm from a Q-switched Nd:YAG laser (Continuum Surelite I-10) is used to irradiate the sub-

strate layer's surface. The surface is made opaque by depositing ~2 μm thick Au and ~2 μm thick graphite in sequence. The temperature rise of the rear surface, that is, the nanowire array's surface, will give a corresponding infrared emission variation, which is detected by a liquid-nitrogen-cooled photovoltaic type of mercury cadmium telluride detector. From the emission variation, we can extract the thermal conductivity of the nanowire arrays. More details about the measurement mechanism and setup can be found in references [25] and [52].

The initial temperature is uniform for the thermally insulated double-layer samples. When a laser pulse of radiant energy is instantaneously and uniformly flashed onto the front surface of the samples, the temperature of the rear surface will be raised. Theoretically, the temperature rise at the rear surface with

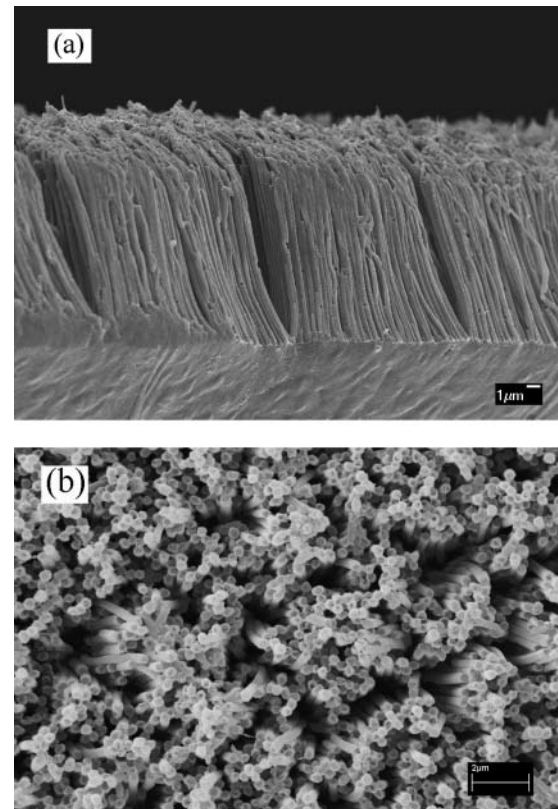


Figure 2 (a) Cross-section and (b) top-view SEM images of the nanowire array.

ignoring all heat losses follows

$$T(t) = \frac{Q}{\rho_0 c_{p0} L_0} \left[1 + 2 \sum_1^{\infty} (-1)^n \exp\left(\frac{-n^2 \pi^2}{L_0^2} \alpha_0 t\right) \right] \quad (1)$$

where Q is the absorbed energy per unit area, α_0 is the overall thermal diffusivity, ρ_0 is the overall density, c_{p0} is the overall specific heat, and L_0 is the overall thickness, keeping in mind that the double-layer sample is inhomogeneous in thermal properties. Divided by the maximum temperature rise $T_{\max} = Q/(\rho_0 c_{p0} L_0)$, the rescaled temperature rise is only related to the thermal diffusivity and thickness:

$$\theta(t) = 1 + 2 \sum_1^{\infty} (-1)^n \exp\left(\frac{-n^2 \pi^2}{L_0^2} \alpha_0 t\right) \quad (2)$$

By detecting the normalized temperature rise, we can obtain the overall thermal diffusivity of the whole sample, and then the thermal conductivity of the nanowire array can be extracted. The measured temperature signal is shown in Figure 3, and the curve agrees with the theoretical formula very well. For double-layer films, the overall apparent thermal conductivity, which regularly depends on individual thermal conductivities of the two layers, is able to be accurately measured by the laser flash method with the overall parameters properly defined. The overall density and specific heat of the whole sample are, respectively, defined as

$$\rho_0 = \frac{\rho[L_s + L_{nw}\varphi]}{L_0} \quad (3a)$$

$$c_{p0} = \frac{c_p[L_s + L_{nw}\varphi]}{L_0} \quad (3b)$$

where ρ is the density of PE, c_p is the specific heat of PE, L_s is the thickness of the substrate layer, L_{nw} is the thickness of the nanowire array layer, and φ is the porosity, ratio of the pore-to-total volumes, of the PAA templates. We assume that

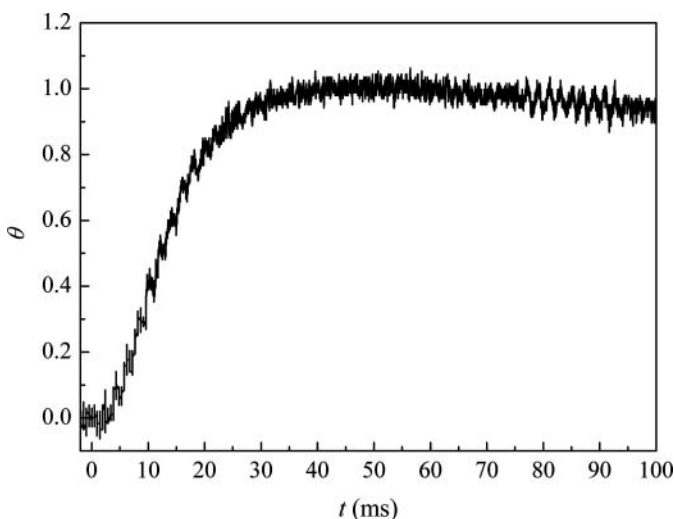


Figure 3 Typical measured normalized temperature rise of the nanowire array's surface varying along with time.

density and specific heat of the nanofibers are the same as those of the substrate since both the parameters are not sensitive to the crystallinity at room temperature. The porosities of the templates with pore diameters of 100 nm and 200 nm are 0.572 and 0.424, respectively. For handling unexpected heat losses, we use Degiovanni's expression to calculate the overall thermal diffusivity,

$$\alpha_0 = \frac{L_0^2}{t_{5/6}} \left[0.8498 - 1.8451 \frac{t_{1/3}}{t_{5/6}} + 1.0315 \left(\frac{t_{1/3}}{t_{5/6}} \right)^2 \right] \quad (4)$$

where $t_{1/3}$ and $t_{5/6}$ refer to times for the temperature rises to one-third and five-sixths of the maximum value, respectively. The overall thermal conductivity is given by $\lambda_0 = \alpha_0 \rho_0 c_{p0}$. We can obtain

$$\lambda_{nw} = \frac{L_{nw} \lambda_s \lambda_0}{L_0 \lambda_s - L_s \lambda_0} \quad (5)$$

The thermal conductivity of the substrate layer, λ_s , is obtained by measuring a PE film with thickness of about 300 μm ; λ_s is about 0.5 W/m-K for HDPE and 0.1 W/m-K for LDPE.

The measured thermal conductivities of the as-fabricated PE nanowire arrays are on the order of 10 W/m-K as shown in Figure 4. The thermal conductivity of the 100-nm-diameter HDPE array at room temperature is 14.8 W/m-K, about 30 times that of the bulk counterpart (~ 0.5 W/m-K). The 200-nm-diameter HDPE nanowire array has a thermal conductivity of 8.7 W/m-K. The well-aligned spun-cast polymer films were reported to have an effective thermal conductivity of only 0.20 W/m-K. With the temperature increasing from room temperature to 80°C, the thermal conductivity of the HDPE nanowire array with diameter of 200 nm increases to 13.8 W/m-K, and the value of the array with 100 nm diameter increases to 21.1 W/m-K. The thermal conductivity of the 200-nm-diameter LDPE nanowire arrays is 2.2 W/m-K at room temperature, and 3.8 W/m-K at a temperature of 80°C. First, it should be noted that the thermal conductivity of the 100-nm-diameter HDPE nanowire array is somewhat

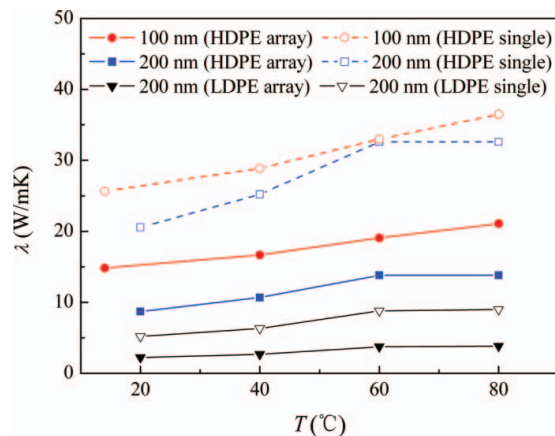


Figure 4 The measured thermal conductivity of HDPE and LDPE nanowire arrays and the estimated thermal conductivity of individual nanowires. (Color figure available online.)

higher than that of the 200-nm-HDPE diameter array, which implies that the polymer chains of the as-prepared nanowires with smaller diameter may be more oriented. This tendency is opposite to the traditional size effects caused by boundary phonon scattering but is in agreement with thermal transport in low-dimensional nanostructures, which is indicative of the domination of the ballistic thermal transport instead of the diffusive way. Second, the thermal conductivity of the HDPE nanowire arrays is smaller than that of the LDPE nanowire arrays. This may be due to the lower crystallinity in the LDPE. Due to the high thermal conductivity of the PE nanowire arrays and their simple but massive production, the as-fabricated nanowire arrays are promising for interfacial materials.

The thermal conductivities of individual nanowires are also estimated from dividing the measured values by the template porosities while ignoring inevitable phonon scattering between the nanowires, also as shown in Figure 4. This means that it gives only the lowest bound predictions of thermal conductivities. At room temperature, the estimated thermal conductivities are 26.5 W/m-K and 20.5 W/m-K for individual HDPE nanowires with 100 nm and 200 nm diameters, respectively. At 80°C, the estimated values reach 36.5 W/m-K and 32.6 W/m-K, which are nearly 2 orders of magnitude higher than that of HDPE bulk structure. For the LDPE nanowire, the estimated thermal conductivity is 5.2 W/m-K at room temperature, and 9.0 W/m-K at 80°C. The modern nanocomposites have thermal conductivity as high as more than 100 W/m-K. Despite this, we believe that there is still room for the enhancement of the intrinsic thermal conductivity of the polymer nanowire arrays regarding further improvement of the fabrication.

As demonstrated in our previous article [25], this enhancement may be attributed to the high chain orientation of the HDPE nanowire arrays. Several factors should be possibly taken into account, such as oscillatory shear rate, translocation motion, and nanoconfinement. The crystallinity of polymer nanofibers, which may dominate the final orientation of polymer nanofibers, was reported to depend on many factors, such as pore size and cooling rate. Hopefully, the integrative effects from shear rate, vibrational perturbation, translocation, nanoconfinement, and crystallization should play important roles in the high orientation of the PE nanowires fabricated by the present nano-molding technique.

WETTABILITY

During the releasing process of the nanowire arrays, the solvent evaporates and dries gradually, and the nanowires are much easier to form into bundles. The construction of the bundles is random in direction and orientation, that is, self-organized. From the top-view SEM images shown in Figure 2b, we can see that the nanowire array surface has a micro-to-nanoscale hierarchical structure. For the lotus leaf, the micro- and nanoscale hierarchical structures, like fractal topology, on the surface contribute to its superhydrophobicity.

A surface with multiscale character was found to be better for its hydrophobicity, as well hydrophilicity, enhancement. Therefore, the present self-organized hierarchical structures are greatly helpful for enhancing the surface wettability.

The wettabilities of the flat and nanostructured PE surfaces are characterized by a high-speed contact angle measuring system (OCAH 200, Dataphysics, Germany) at room temperature (20°C). We use water, glycerin, ethanol, paraffin liquid, and methyl silicone oil droplets to characterize the wettability of the flat film and nanostructured sample with nanowire arrays in 200 nm diameter. For water and glycerin, the PE native flat surface shows slightly hydrophobic with contact angles of about 102.2° and 94.4°, respectively, as shown in Figures 5a and 5c, while the nanowire array surface shows much higher hydrophobicity with contact angles of about 151.8° and 146.4° for water and glycerin, respectively, as shown in Figures 5b and 5d. First, we can see that the surface nanostructures greatly enhance the hydrophobicity for water and glycerin. The present nano-molding technique, having advantages of simple, massive production and high quality, can prepare superhydrophobic polymer surfaces. The technique is promising for developing micro-/nanoscale fluidic and heat transfer devices. It should be pointed out that there may be still room to optimize the hydrophobicity of the polymer nanowire array surfaces through varying the nanowire diameter, length, releasing parameters, and so on. Second, it is very interesting that, as an organic liquid, glycerin shows large hydrophobicity over the PE film. But for other organic liquids measured in our research, namely, ethanol, paraffin liquid, and methyl silicone oil, the contact angles are much smaller, as shown in Figures 5e–5i. Over the flat native PE film, ethanol, paraffin and methyl silicone oil have contact angles of 32°, 12.5°, and 12.9°, respectively. However, over the nanowire array surface, they have contact angles of 16.9°, 5.4°, and 7.6°, respectively. Thus, the surface wettability deserves to be checked if organic liquids are operated in micro-/nanodevices.

Traditionally, the wettability of a rough surface can be characterized by the well-known Cassie and Wenzel models. The interaction between a droplet and a rough surface is weakened by patches of air beneath the micro-to-nanoscale hierarchical nanostructures. The Cassie model for rough surfaces is

$$\theta_n = \cos^{-1}(f \cos \theta_f + f - 1) \quad (6)$$

where θ_n and θ_f are, respectively, the contact angles of the nanowire array and flat surfaces, f is the area fraction of the liquids contacting with the surface. For the present nanowire array surface, we have $\theta_n = 151.8^\circ$, $\theta_f = 102.2^\circ$ for water and $\theta_n = 146.4^\circ$, $\theta_f = 94.4^\circ$ for glycerin, so the area fraction of the present self-organized surface is only 15–20%. The Wenzel model gives the hydrophilic contact angle over rough surfaces as

$$\theta_n = \cos^{-1}(r \cos \theta_f) \quad (7)$$

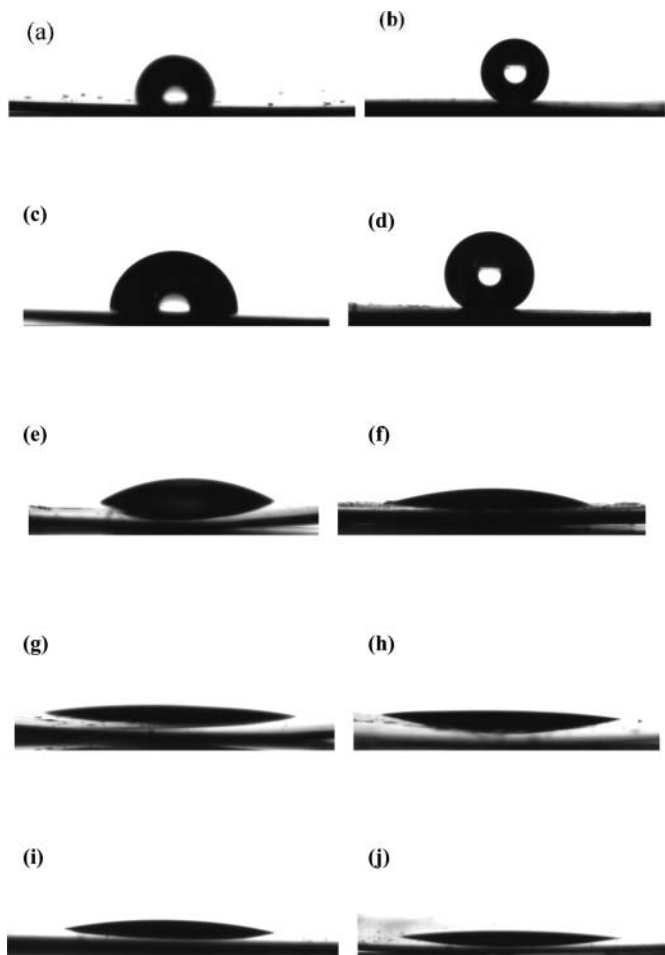


Figure 5 The measured contact angles: (a) 102.2° for water over flat surface; (b) 151.8° for water over nanostructured surface; (c) 94.4° for glycerin over flat surface; (d) 146.4° for glycerin over nanostructured surface; (e) 32° for ethanol over flat surface; (f) 16.9° for ethanol over nanostructured surface; (g) 12.5° for paraffin over flat surface; (h) 5.4° for paraffin over nanostructured surface; (i) 12.9° for methyl silicone oil over flat surface; and (j) 7.6° for methyl silicone oil over nanostructured surface.

Here, r is the roughness ratio defined as the ratio of true area of the solid surface to the apparent area. From the measured contact angles already described, the roughness ratios for ethanol, paraffin, and methyl silicone oil are 1.13, 1.02, and 1.02, respectively. We can see that the roughness ratio depends on the liquid species greatly. Finally, it should be pointed out that the PE film surfaces are patterned as well as roughened by the nanowire arrays, and the flow friction in real microfluidic devices will depend on the incorporative effects of slippage [34–38], sticky liquid–gas menisci [53], roughness-induced energy dissipation [36], and so forth.

CONCLUSIONS

We develop a nano-molding technique that can prepare polyethylene nanowire arrays with high thermal conductivity

(more than 10 W/m-K) and superhydrophobicity (contact angle $> 150^\circ$). The thermal conductivities of the as-prepared nanowire arrays are measured by a laser flash method. It is found that the thermal conductivities are in the order of 10 W/m-K, nearly 2 orders of magnitude higher than their bulk structure. The estimated thermal conductivity of individual HDPE nanowires can reach 26.5 W/m-K at room temperature. In particular, we find that the as-prepared nanowires with smaller diameters have higher thermal conductivities, which is indicative of the domination of ballistic thermal transport. The thermal conductivity of LDPE nanowires is a little smaller than that of HDPE nanowires. The integrative effects of shear rate, vibrational perturbation, translocation, nanoconfinement, and crystallization may be responsible for the high orientation of the HDPE nanowires. The surfaces of polymer nanowire arrays are found to have micro-to-nanoscale hierarchical structures, and have superhydrophobicity of $> 150^\circ$ contact angles for water. For organic liquids, we find that glycerin gives hydrophobic, but ethanol, paraffin liquid, and methyl silicone oil give hydrophilic wettabilities. Based on the Cassie model, the area fraction of the present self-organized surface is only about 15%. The present superhydrophobic surfaces with high thermal conductivity may be quite promising for developing micro-/nanoscale fluidic and heat transfer devices due to their simple but massive production with high quality. The orientation mechanisms of the as-fabricated polymer nanowires, the reliability and stability, and also the applications to thermal interface materials and microfluidics devices are hopefully investigated in the future.

NOMENCLATURE

| | |
|--------------------|---|
| c_p | specific heat of PE, J/(kg-K) |
| c_{p0} | overall specific heat, J/(kg-K) |
| f | area fraction of the liquids contacting with the surface, dimensionless |
| L_0 | thicknesses of the overall sample, m |
| L_s | thicknesses of the substrate layer, m |
| L_{nw} | thicknesses of nanowire array layer, m |
| Q | absorbed energy per unit area, J/m ² |
| r | roughness ratio, dimensionless |
| t | time, s |
| $t_{1/3}, t_{5/6}$ | times for the temperature rise to one-third and five-sixths of the maximum value, s |
| T | temperature, K |
| T_{max} | maximum temperature rise, K |

Greek Symbols

| | |
|------------|--|
| α_0 | overall thermal diffusivity, m ² /s |
| θ | rescaled temperature rise, dimensionless |
| θ_n | contact angle of the nanowire array, ° |
| θ_f | contact angle of the flat surfaces, ° |

- λ_0 overall thermal conductivity, W/(m-K)
 λ_s thermal conductivity of the substrate layer, W/(m-K)
 λ_{nw} thermal conductivity of nanowire array layer, W/(m-K)
 ρ_0 overall density, kg/m³
 ρ density of PE, kg/m³
 φ porosity, ratio of the pore-to-total volumes, of the PAA templates, dimensionless

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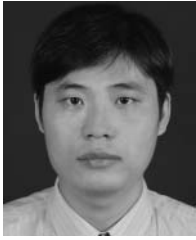
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