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APPARENT CONTACT ANGLES AND TIME OF THE THREE PHASE CONTACT FORMATION BY THE BUBBLE COLLIDING WITH TEFLON SURFACES OF DIFFERENT ROUGHNESS

The paper presents results and analysis of influence of hydrophobic surface roughness on apparent contact angle values (equilibrium conditions) and time of the bubble attachment (dynamic conditions) to hydrophobic solid surfaces (Teflon) of different roughness. The surface roughness of Teflon plates was modified in a mechanical way using abrasive papers and/or diamond paste of different grid numbers. Measurements of contact angles were carried out by the sessile drop technique, while the time of three phase contact (TPC) formation and the bubble attachment were determined in a course of the bubble collisions with Teflon plates, using a high speed camera (1182 Hz). It was found that the surface roughness is an important parameter affecting both quantities determined. With increasing surface roughness the static contact angle was increasing, while the time needed for TPC formation and the bubble attachment was significantly shortened, from ca. 80 to 3 ms. Air entrapped inside surface scratches seems to be a reason of these effects. With increasing roughness a larger amount of air can be entrapped inside the scratches. This hypothesis is confirmed by measurements of the diameters of contact perimeter of the attached bubble, where it was found that the perimeter increases with the surface roughness.

Key words: *hydrophobic solids, Teflon, surface roughness, three phase contact, bubble collision, bubble attachment, time of attachment, sessile drop, contact angle*

INTRODUCTION

The hydrophobic/hydrophilic characteristic of solids is known to play a key role in many processes such as: wetting, flotation, enhanced oil recovery, cleaning technologies, superhydrophobicity, liquid spreading, plants protection, etc. Wettability is quantified in terms of the contact angle (θ) values, and it is generally, but rather

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arbitrarily assumed that $\theta < 90°$ indicates that the solid is partially wetted by a liquid (for example water). Surfaces characterized by the contact angle of water smaller than 90° are usually termed hydrophilic, and if the contact angle $\theta > 90°$ they are called hydrophobic. On the other hand, if the contact angle of water is higher than zero, then the work of spreading, W_S, is negative.

$$W_s = W_A + W_C \tag{1}$$

where: W_A is the work of adhesion and W_C is the work of cohesion. The works of adhesion and cohesion are related to solution surface tension as follows:

$$W_A = \sigma_{LV}(1 + \cos\theta) \tag{2}$$

$$W_C = 2\sigma_{LV} \tag{3}$$

Hence:

$$W_S = \sigma_{LV}(1 + \cos\theta) - 2\sigma_{LV} = \sigma_{LV}(\cos\theta - 1) \tag{4}$$

Negative value of W_S is the necessary condition for the flotation process to occur. So, theoretically at any contact angle higher then zero flotation of a mineral can proceed (Leja, 1982; Drzymala, 2001) and therefore in flotation the surfaces having contact angles below 90° are termed as weakly hydrophobic (Leja, 1982), while that of $\theta > 90°$ are called strongly hydrophobic. Recently it was also showed (Chibowski and Hołysz, 1999/2000) in laboratory flotation experiments (Halimond's tube) that for a 50% floatability of a mineral, the negative work of spreading should amount to minimum -20mJ/m². It means that for an efficient flotation the contact angle of water has to be at least 48° and more. Solids showing the contact angles of an order 140° are termed as the superhydrophobic ones and are of rapidly increasing importance in many applications (contamination prevention, enhanced lubricity and durability of materials, biocompatibility and many others). Roughness modification of a hydrophobic solid surface is one of ways for obtaining the superhydrophobic surfaces.

The first approach to characterize the equilibrium in a solid/liquid/vapor system was introduced by Young (1805) and his equation describes the mechanical balance at the line of the three phase contact (TPC) on an ideal (smooth, homogeneous, rigid and insoluble) solid (see Fig.1A) as:

$$\sigma_{SV} = \sigma_{SL} + \sigma_{LV}\cos\theta \tag{5}$$

where: σ_{LV} is the liquid/vapor surface energy, σ_{SL} is the solid/liquid surface energy, and σ_{SV} is the solid/ vapor surface energy. The fundamental problem associated with the equilibrium contact angle is related to the structure and topography of the solid

surface, as the real solids are rough. Surface roughness can affect strongly wettability and values of the apparent contact angles. To characterize the non-geometrical ideality of a surface, roughness parameter r was introduced and defined as:
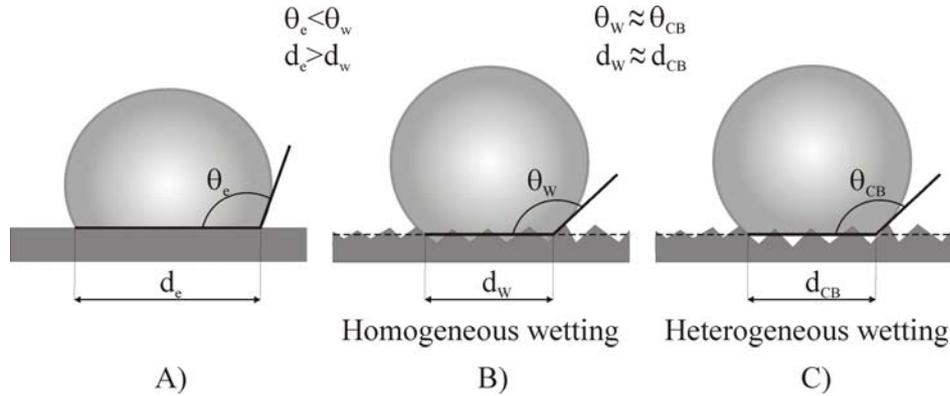
Fig. 1. Schematic of the effect of surface roughness of the hydrophobic solid on contact angle measurements by the sessile drop method

$$r = \frac{A_{real}}{A_{geometrical}} \tag{6}$$

where: (A_{real}) and ($A_{geometrical}$) are the geometrical and real areas of the surface. For $r>1$ Young equation (5) was modified by Wenzel (1936). The Wenzel equation:

$$\cos\theta_W = r \cdot \cos\theta \tag{7}$$

is a generalization of the Young equation where: θ_W is the Wenzel contact angle. The Wenzel equation refers to so-called homogeneous wetting regime (Marmur, 2003; 2004), i.e. when the liquid completely penetrates scratches, grooves and cavities (Fig. 1B). Another situation, when air is entrapped inside the grooves underneath the liquid, is termed the heterogeneous wetting (see Fig.1C), and is described by the Cassie-Baxter equation (Cassie and Baxter, (1944):

$$\cos\theta_{CB} = r_f f \cos\theta + f - 1 \tag{8}$$

where: θ_{CS} is Cassie-Baxter contact angle, f is a fraction of the projected area that is wet by a liquid and r_f is the roughness ratio of the wet area. When $f=1$ then r_f is equal r and Cassie-Baxter equation (8) turns into Wenzel equation (7). As recently discussed by Extrand (2004) and Sedev et al. (2004), the phenomenon of higher values of contact angle on modified (roughened, micro patterned, machined or etched (Veeramasuneni et al. (1997); Oner and McCarthy (2000); Nakajima et al. (2001))

hydrophobic surfaces is caused by inhibition of the liquid spreading into grooves, scratches and/or cavities on the rough surface. Moreover, a spreading of such drop can be "arrested" by the edges of the grooves.

Differences in wettability are the factor governing possibility of separation of various components of ores during flotation. When air bubble collides with hydrophobic mineral particle inside a flotation chamber, then a thin liquid film (TLF) separating them ruptures and the particle can be attached. A stable bubble-grain aggregate formed follows up to the froth layer where grains of useful component are concentrated. Grains of the waste material (hydrophilic in their nature) do not undergo attachment to the air bubbles and settle to the bottom of the flotation tank.

During a bubble collision, which is dynamic and complex phenomenon, with hydrophobic particle following steps must take place (Leja, 1982; Nguen and Schulze, 2004): i) the film drainage until its critical thickness of rupture is obtained, ii) the film ruptures and the "nuclei" of the TPC is formed and iii) TPC expansion until perimeter ensuring the bubble-grain aggregate stability is created. Time needed for the drainage, rupture of the TLF and the bubble-particle attachment is called time of the TPC formation (t_{TPC}). Magnitude of the t_{TPC} depends strongly on hydrophilic/hydrophobic properties (Schulze et al., 2001; Stöckelheuber et al., 2001) and surface roughness (Krasowska and Malysa, 2005; Krasowska and Malysa, 2006), as well.

This paper reports results on influence of roughness of the hydrophobic solid surface on the apparent contact angle values (static conditions) and the time of the three phase contact formation by the colliding bubble (dynamic conditions). The results are discussed in terms both of the surface roughness and presence of submicroscopic gas bubbles at the hydrophobic rough surfaces being in contact with water.

EXPERIMENTAL

The contact angle measurements were carried out using Digidrop apparatus of GBX (France). To measure the contact angle hysteresis, 6 µl doubly distilled water droplet was settled on the Teflon surface and the advancing contact angle was measured. Then, 2 µl of water was sucked-off and the receding contact angle was measured. Both temperature ($22^\circ \pm 2^\circ$) and humidity (50%) were constant.

The set-up for monitoring the bubble collision with Teflon plates consists of the following parts: i) a square glass column filled with four-fold distilled water (conductivity 0.2-0.4 µS), ii) capillary of inner diameter of 0.075 mm mounted at the bottom of the column, iii) syringe pump for high precision gas supplying, iv) high-speed camera for recording the bubble collisions with the Teflon plates, v) PC with image analysis software. For monitoring and recording the bubble collisions with studied surfaces the high-speed camera (SpeedCam 512+) was mounted at the same level as the Teflon plate. All movies recorded during bubble collision with the Teflon plates were transformed into *.bmp files and carefully analyzed using the Sigma Scan

Pro 5.0 software. All other details of the experimental procedure were described elsewhere (Krasowska et al., 2003; Malysa et al., 2005). Teflon plates were mounted horizontally inside the column at the distance ca. 300 mm from the capillary orifice, just beneath the water surface. The diameter of the rising bubble was constant (d_b = 1.5 mm).

| A) | B) |
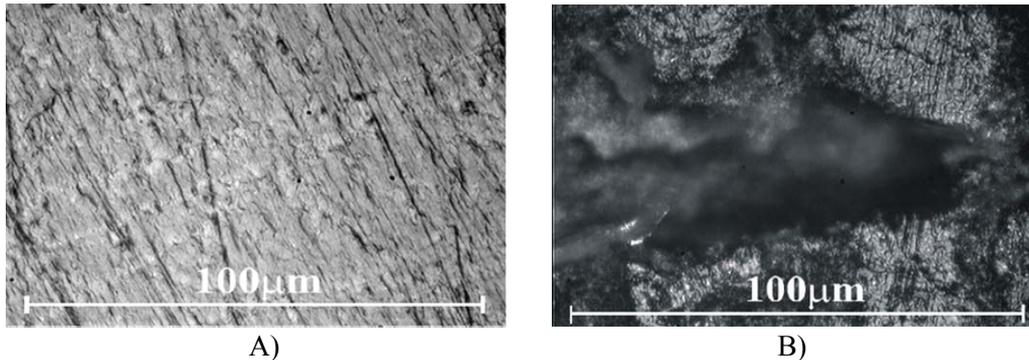
Fig. 2. Microscopic photos of the Teflon plates surfaces. A) - "Teflon I", B) – "Teflon V"

Five plates (20x20x3mm) were prepared from the same piece of Teflon, but their surface roughness was modified in a mechanical way using abrasive papers of different grid numbers. Figure 2 presents the microscopic photos of the smoothest (Fig. 2A) and the roughest (Fig. 2B) surfaces of the Teflon plates used in the experiments. First plate, called "Teflon I", had the surface modified using the abrasive paper No. 2400 and then, the diamond grinding DP-Paste ¼ μm. The second one, called "Teflon II" was tailored by using the abrasive paper No. 2400, the third one – "Teflon III" – was a Teflon plate as received from the manufacturer shop. The fourth plate, called "Teflon IV", was roughened with the abrasive paper No. 220, and the "Teflon V" was roughened using the abrasive paper No. 100. Cavities dimensions determined from the microscopic photos are listed in Table 1. As can be seen there the scratches dimensions at the "Teflon I" and "Teflon II" plates were below 1 m, for "Teflon III" – ca. 30 – 60 μm, "Teflon IV" – ca. 50-80 μm, "Teflon V" – ca. 80-100 μm.

For the contact angle measurements the plates were washed in methanol and three times in water from a Milli-Q 185 system. Then, they were boiled in the distilled water to remove air which could be entrapped between scratches, cavities etc at the Teflon surface. Next, they were put for 15 min to ultrasonic bath, rinsed with doubly distilled water and dried at 100°C. For the dynamic bubble collision measurements all Teflon plates were cleaned with a chromic mixture to avoid organic contaminants, which would strongly affect mobility of the rising bubble because of their adsorption at the bubble surface. Then, all plates were carefully washed-out with four-fold distilled water and boiled in four-time distilled water.

The experiments were carried out at room temperature (22 ± 2^0C).

RESULTS AND DISCUSSION

Variation of the advancing and receding contact angles values as a function of Teflon surface roughness are presented in Fig.3 and in Table 1. As can be seen there the contact angle values depend on the surface roughness. For the smoothest "Teflon I" and "Teflon II" the advancing contact angles were the smallest and did not reach 100°. With increasing the Teflon surface roughness an increase in values of the measured contact angles was observed. For the roughest "Teflon V" surface the advancing contact angle was over 128 degree. As the surface modified in mechanical way is not homogenous and size of the scratches, grooves, cavities varied in a quite wide range (see Table 1), therefore some scatter in the measured values was observed. To avoid inappropriate data interpretation every contact angle measurement was repeated 20-30 times on each Teflon plate. The obtained results clearly indicate that there is a correlation between roughness of the hydrophobic (Teflon) surface and the contact angle values.
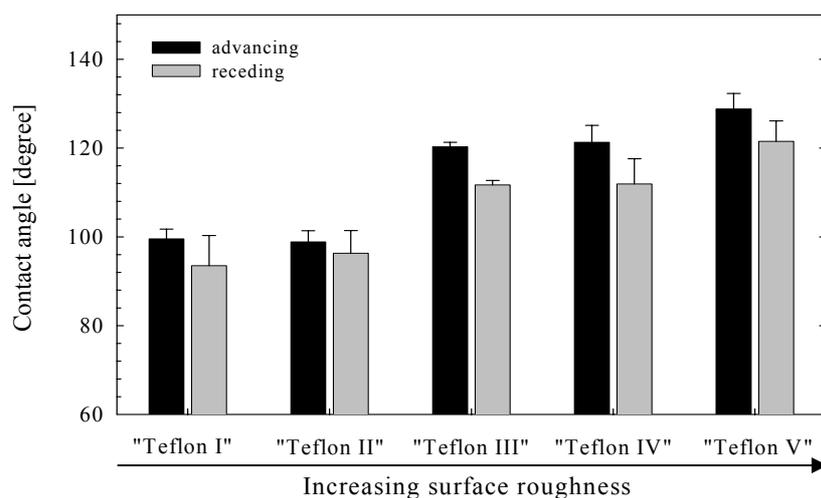
Fig. 3. Diagram of advancing and receding contact angles on Teflon surfaces of different roughness

Table 1 Advancing and receding contact angles, and roughness of the Teflon plates surfaces

Plate	Surface roughness	Contact angle	
		Advancing	Receding
Teflon I	Below 1 μm	99.6 ± 2.2	93.5 ± 6.8
Teflon II	Below 1 μm	98.9 ± 2.5	96.3 ± 5.1
Teflon III	30 – 60 μm	120.3 ± 1.0	111.7 ± 1.0
Teflon IV	50-80 μm	121.3 ± 3.8	111.9 ± 5.7
Teflon V	80-100 μm	128.8 ± 3.5	121.5 ± 4.6
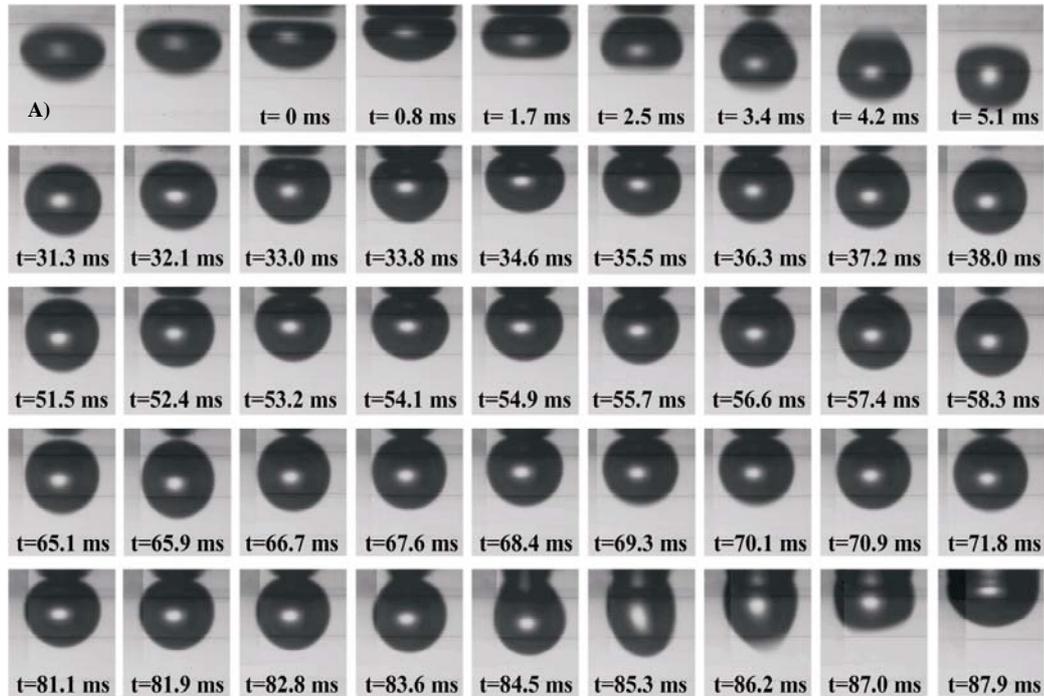
Fig. 4. Sequences of photos of the bubble colliding with "Teflon I" (A) and "Teflon V" (B) plates in distilled water

Figures 4A and 4B present two sets of photos illustrating sequence of the bubble collisions with the smoothest "Teflon I" (Fig. 4A, contact angle ca. 100°) and the roughest "Teflon V" (Fig. 4B, contact angle ca. 130°) surfaces. It is commonly assumed that when the solid surface is hydrophobic then the bubble colliding with such solid should be attached at once. Data presented in Fig.4 show clearly how surface roughness strongly affects the time of the TPC formation (t_{TPC}) and bubble attachment. It can be immediately noted that in the case of the smoothest (roughness below 1 μm) "Teflon I" surface (Fig. 4A) the attachment did not occur during the first collision but the bubble bounced a few times, despite that the surface was highly hydrophobic (contact angle ca. 100°). In the case of much more rough "Teflon V" surfaces (cavities of 80-100 μm) the bubble was attached during the first collision – there was no bouncing (see Fig. 4B). Thus, with increasing roughness of the Teflon surface the t_{TPC} was significantly shortened. As can be noted in Fig.4 the time needed for formation of the first "spot" of the TPC contact (i.e. the moment when rupture of the wetting film (WF) occurred) was t_{TPC} = 83.6 ms in the case of the smoothest surface ("Teflon I"), while for the rough "Teflon V" surface it was of an order of 2.5 ms only, i.e., 20 - 25 times shorter. However it should be stressed that the zero time on the time scale of the TPC formation and the bubble attachment had to be settled. To

avoid any arbitrariness as the zero time, called $t_{TPC} = 0$ ms it was chosen as the moment (first frames of the recordings) at which the horizontal diameter of the colliding bubble started to increase. This means that the bubble was already in contact with the solid, what stopped its upwards motion.
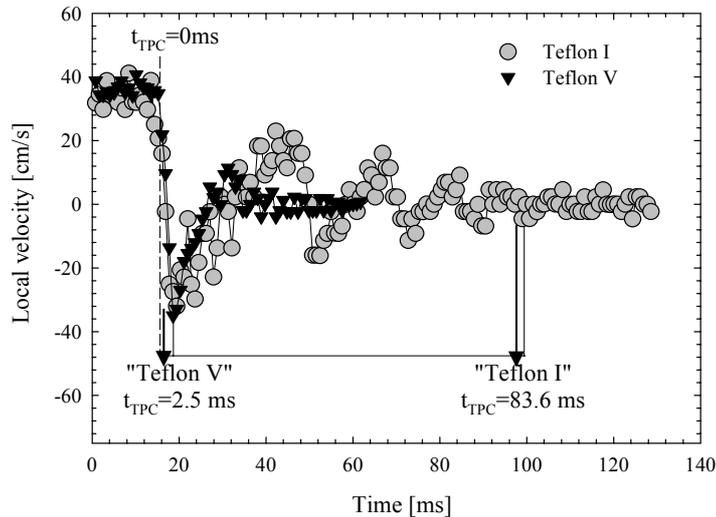
Fig. 5. Variations of the bubble local velocity during collisions ("approach-bounce" cycles) with "Teflon I" and "Teflon V" surfaces in distilled water

Quantitative data on variations of the bubble velocity during collisions with the "Teflon I" and "Teflon V" plates are presented in Fig 5. As seen there prior to the first collision the local velocity of the bubble was constant and equal to ca. 35cm/s. During the first collision the bubble was attached to the "Teflon V" surface ($t_{TPC} = 2.5$ ms). In the case of the smoothest "Teflon I" surface the bubble bounced and five "approach-bouncing" cycles can be clearly noted (see Fig. 5) prior to the TPC formation ($t_{TPC} = 83.6$ ms) and the bubble attachment. Due to energy dissipation the bubble velocity was decreasing in every subsequent cycle. Values of the t_{TPC} refer to the moment of the first "spot" of the TPC formation (see Fig. 4). As can be seen in Fig. 4 the diameter of the TPC is increasing after the t_{TPC}, but after next few milliseconds a constant perimeter was always achieved.

Contact angles can be determined using sessile drop technique or in a reverse system, i.e. by the captive bubble method. In both techniques, values of the contact angle are measured through the liquid phase. In the sessile drop method the drop settled on a highly hydrophobic surface remains almost spherical. The higher value of the contact angle is the more spherical shape of the bubble can be observed. Thus, the length of perimeter and/or the diameter of the three phase contact (TPC) are the parameters related to the contact angle values. With increasing contact angle (hydrophobicity) the diameter of TPC perimeter will decrease. The opposite is true in the case of the captive bubble method, i.e., in the case of the attached bubble.

Table 2. Photos and the three phase contact (TPC) diameters of the sessile drop and the bubble attached to different Teflon plates

Plate	Sessile drop	Sessile drop - TPC diameter [mm]	Bubble attached	Bubble attached - TPC diameter [mm]
Teflon I		2.64		1.71
Teflon II		2.67		1.72
Teflon III		2.15		1.76
Teflon IV		2.05		2.04
Teflon V		1.88		2.29
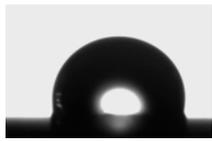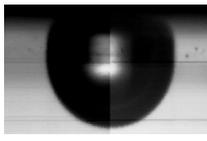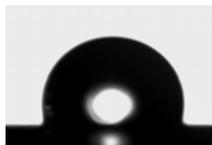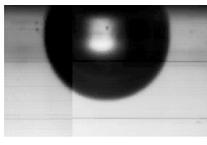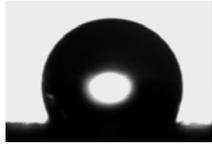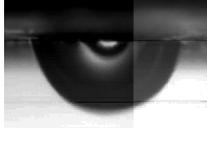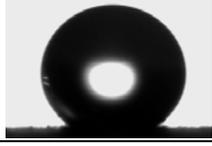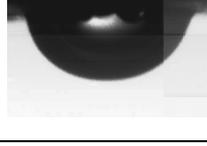
Table 2 presents photos of the sessile drops and bubbles attached to the Teflon surfaces. As can be noticed there both TPC diameters (the sessile drop and the bubble attached to Teflon surface) depend on the surface roughness. Figure 6 presents the diameters of the TPC formed at equilibrium (contact angle measurements) and dynamic conditions (bubble attachment to the Teflon surface) as a function of the Teflon surface roughness. For the sessile drop method the diameter of the TPC becomes smaller with increasing Teflon surface roughness (see Fig. 6 and Table 2). The diameter varies from 2.64-2.67 mm for the smoothest "Teflon I" and "Teflon II" surfaces to 1.88 mm for roughest "Teflon V". Opposite relationship can be observed for the TPC diameter of the bubble attached to the Teflon surfaces studied. Here, the

diameter of the TPC increases within the range with increasing the surface roughness (see Fig. 6 and Table 2). The smallest value of the TPC diameter was determined for "Teflon I" (1.71 mm) and the largest one for "Teflon V" (2.29 mm). Let us discuss reasons of the variations with Teflon surface roughness both the sessile drop and the attached bubble diameters. The rougher surface is the bigger scratches, grooves and gaps are on the surface. When a drop of water is settled on such surface a motion of the edge of the TPC is "restricted" by the scratches and cavities (Marmur (2004)).
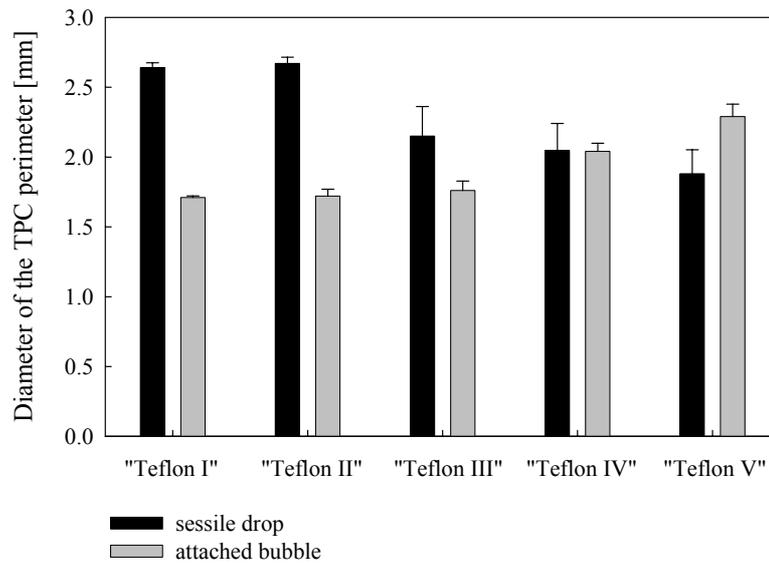
Fig. 6. Diameters of the three phase contact (TPC) perimeter for Teflon plates of different roughness

Therefore, water can not spread further over the solid surface and the diameter of the TPC becomes smaller with the increase in the surface roughness (drop remains more spherical). In the reverse system (i.e. bubble at the hydrophobic Teflon surface) the increase of the attached bubble diameter with surface roughness is related, in our opinion, to the number and size of submicroscopic bubbles adhered to the hydrophobic Teflon surface. It was reported by Ryan and Hemmingsen (1998) and Snoswell et al (2003) that during immersion of dry hydrophobic surface into solution there can be some microscopic air bubble entrapped in the grooves, scratches and gaps. In our previous papers (Malysa et al., 2005; Krasowska and Malysa, 2006) it was also showed that the presence of a micro-bubble at the hydrophobic solid surface can be one of most important parameters facilitating attachment of the colliding bubble. Because higher roughness is the bigger cavities are and more gas is entrapped. Therefore, the needed perimeter of the TPC for the bubble attachment is faster formed

there. The results presented in this paper again support this hypothesis. In the experiments the bubble size was always the same (d_b = 1.5 mm). Therefore, the fact that the diameter of the three phase contact of the attached bubble varied with the surface roughness (Fig. 6 and Table 2) shows that the volumes of air adhered to the Teflon plates were different. The largest diameters of the attached bubbles were found for the roughest surfaces and this is a strong evidence that this is due to larger volumes of the gas entrapped in larger cavities of the rough surface.
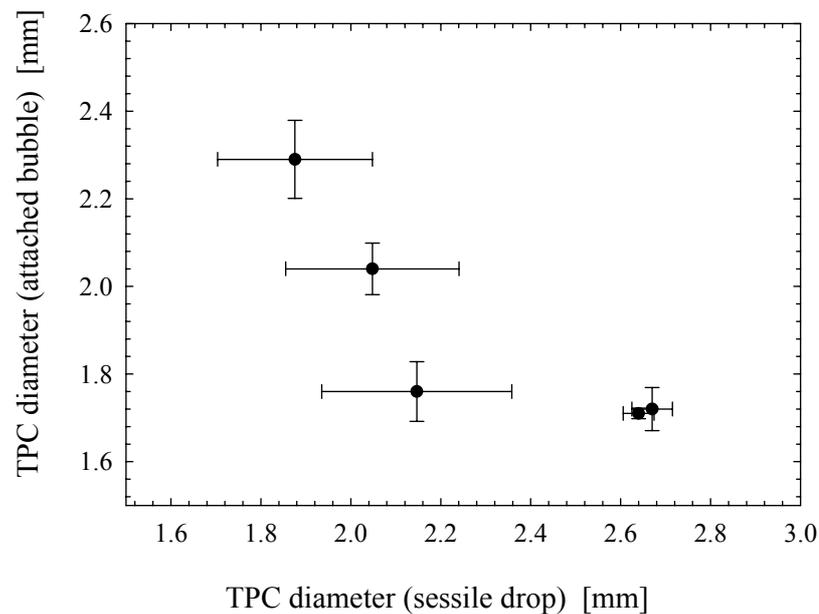
Fig. 7. Three phase contact (TPC) diameter of the attached bubble versus the TPC diameter of the sessile drop

The relationship between diameters of the TPC perimeters formed under dynamic (attached bubbles) and static (sessile drops) conditions is presented in Figure 7. It is clearly seen there that both magnitudes are inversely proportional. The contact angles are always measured through the liquid phase, i.e. smaller diameter of the sessile drop and larger diameter of the attached bubble indicate that the contact angle was larger. When the water drop is settled on the hydrophobic solid, cohesive forces between water molecules are stronger that interaction with the solid and the water tends to form a drop rather than to spread over the surface. In the case where the air bubble attached to the hydrophobic solid, such a system tends to minimize the area of the hydrophobic surface contacted with water and this is why the bubble seems to "spread" at the surface thus increasing the TPC diameter.

CONCLUS IONS

Roughness of hydrophobic Teflon surface affects both the static contact angle and the kinetics of the bubble attachment. With increasing roughness, the static contact angles at the Teflon surfaces were increasing, while the time needed for the TPC formation and attachment of the colliding bubble was decreasing (from ca. 80 to 3 ms). An appropriate modification of surfaces of the hydrophobic solids (for example through roughening) makes obtaining the superhydrophobic surfaces, i.e., the surfaces showing contact angle (for water) of 150° and larger (up to almost 180°) possible.

Air entrapped inside the scratches and cavities of Teflon surface is responsible for both the increase in contact angle and facilitation of the bubble attachment. This hypothesis is confirmed by measurements of the diameters of three phase contact perimeters of the sessile drops and the attached bubbles. In the case of sessile drop, the TPC diameter was decreasing with increasing the surface roughness because the scratches and grooves caused an energetic barrier for the water drop to spread. An opposite effect was observed for the bubble attached to the Teflon surfaces studied. The diameter of the TPC perimeter of the attached bubble was increasing with the Teflon surface roughness. As higher roughness means that more air was entrapped inside the surface cavities, therefore the diameter of the attached bubble increased.

ACKNOWLEDGEMENTS

LIST OF SYMBOLS AND ABBREVIATIONS

ABBREVIATIONS

TLF	Thin liquid film
TPC	Three phase contact
WF	Wetting film

SYMBOLS

$A_{geometrical}$	Geometrical area of the surface
A_{real}	Real area of the surface
d_b	Bubble diameter
f	Fraction of the projected area that is wet by a liquid
r	Roughness ratio
r_f	Roughness ratio of the wet area
t_{TPC}	Time of the TPC formation
W_A	Work of adhesion
W_C	Work of cohesion

W_S	Work of spreading
σ_{LV}	Liquid/vapor surface energy
σ_{SL}	Solid/liquid surface energy
σ_{SV}	Solid/vapor surface energy
θ	Contact angle
θ_{CS}	Cassie-Baxter contact angle
θ	Equilibrium (Young) contact angle
θ_W	Wenzel contact angle

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W pracy przedstawiono wyniki i analizę wpływu szorstkości powierzchni hydrofobowej na wielkości wstępujących kątów zwilżania (warunki równowagowe) oraz na czas potrzebny do przyczepienia bańki (warunki dynamiczne) do hydrofobowej powierzchni ciała stałego (teflon). Szorstkość powierzchni płytek teflonowych była modyfikowana mechanicznie przy użyciu papieru ściernego o różnym

uziarnieniu oraz pasty diamentowej. Pomiary kątów zwilżania wykonano metodą "siedzącej" kropli (sessile drop) a czas powstawania kontaktu trójfazowego (TPC) i przyczepienia bańki był wyznaczany przy zastosowaniu szybkiej kamery (1182 Hz). Wykazano, że szorstkość powierzchni jest parametrem mającym olbrzymi wpływ na obie badane wielkości. Ze wzrostem szorstkości powierzchni wzrastały wartości kąta zwilżania, a czas potrzebny do utworzenia TPC i przyczepienia bańki ulegał znacznemu skróceniu, od ok. 80 ms do 3 ms. Ponieważ ze wzrostem szorstkości zwiększa się ilość powietrza "uwięzionego" wewnątrz nierówności powierzchniowych dlatego wydaje się, że jest to czynnik decydujący o zmianach wielkości kąta zwilżania i wartości czasu potrzebnego do utworzenia TPC. Potwierdzeniem poprawności tej hipotezy są także przedstawione w pracy wyniki pomiarów średnic perymetru przyczepionej bańki. W pomiarach tych wykazano, że ze wzrostem szorstkości wzrasta średnica perymetru bańki przyczepionej do powierzchni teflonu.