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Rubber adhesion below the glass transition temperature: Role of frozen-in elastic deformation

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Abstract – We have studied how the adhesion between rubber and a flat countersurface depends on temperature. When the two solids are separated at room temperature negligible adhesion is detected, which is due to the elastic deformation energy stored in the rubber, which is given back during pull-off and help to break the adhesive bonds. When the system is cooled down below the glass transition temperature, the elastic deformation imposed on the system at room temperature is “frozen-in” and the stored-up elastic energy is not given back during separation at the low temperature. This results in a huge increase in the pull-off force. This study is crucial for many applications involving rubber at low temperatures, *e.g.*, rubber seals for cryogenic or space applications.

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Introduction. – Most surfaces of solid objects have surface roughness on many length scales [1], which has a crucial influence on the contact between elastic solids [2–11]. Surface roughness is the main reason why adhesion (manifested as a pull-off force) is usually not observed at the macroscopic length scale [12–27]. Solids can interact and form bonds only when separated at atomistic distances (nanometer or less), and in order to make contact the solids must deform at the interface. For elastically “hard” solids this results in a very small contact area which reduces the adhesion. More importantly, the elastic deformation of the solids at the interface (as a result of the adhesive forces) leads to stored-up elastic energy. During separation this elastic energy is given back, and helps to break the atomic bonds, and results in the vanishing pull-off force observed in most cases. Thus, adhesion is only observed for either extremely smooth surfaces (as in wafer bonding [28]), or when at least one of the solids is elastically very soft [29,30].

At room temperature rubber-like materials are soft with Young’s modulus of the order of a few MPa for most types of engineering rubber, such as rubber for seals, tires or wiper blades, and even much less for the weakly

cross-linked rubber used, *e.g.*, for pressure-sensitive adhesives (which can have Young’s modulus as low as 1 kPa) [30]. However, when rubber materials are cooled below the glass transition temperature Young’s modulus typically increases by a factor of ~ 1000 (and sometimes much more) to several GPa. Thus, if a rubber object is moved in contact with another solid at low temperature no adhesion force will be detected in most cases, even if the adhesion would be strong at room temperature. However, here we will show that if a rubber object is squeezed into contact with a counter object at room temperature and then cooled down below the glass transition temperature, an extremely strong adhesive bond can form. This results from the frozen-in elastic deformation field (see fig. 1), and from the high effective Young’s modulus in the glassy state. As will be discussed below, these phenomena have important technological implications, and if not properly accounted for, can result in expensive or disastrous consequences, such as the Challenger catastrophe [31].

Experimental. – We have performed two types of experiments (schematically shown in fig. 2(a) and (b))

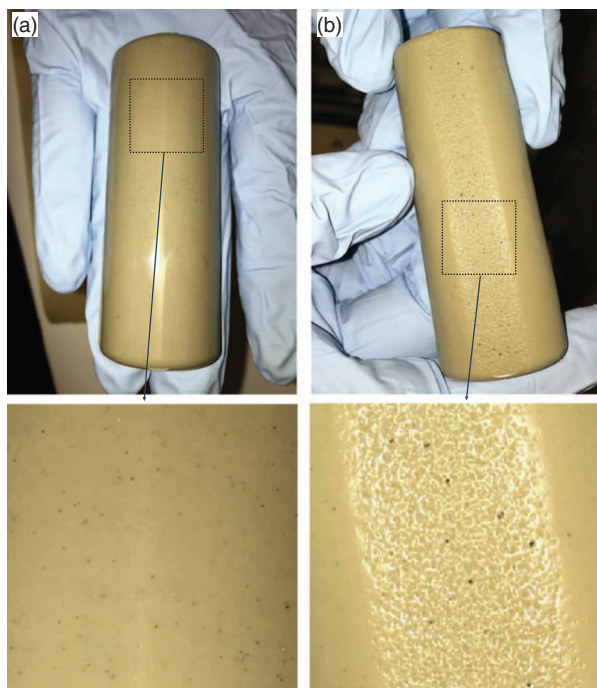


Fig. 1: (Colour online) Demonstration of frozen-in elastic deformation. Picture of a rubber half-cylinder at (a) room temperature and (b) at $T = -40^\circ\text{C}$, after the cylinder was squeezed at room temperature against a sandpaper surface, and then cooled down. Note in (b) the rectangular (nominal flat) strip of frozen-in rubber deformation (Hertz-like contact). The magnification picture (right) shows that the (nominal flat) contact area has frozen-in surface roughness inherited from the surface roughness of the sandpaper. The frozen-in deformation of the rubber cylinder stays unchanged as long as the rubber is kept at $T = -40^\circ\text{C}$, but disappears when the rubber sample is kept for some time at a temperature above the rubber glass transition temperature, *e.g.*, at room temperature.

to study how the adhesion between rubber and a countersurface depends on the temperature. We have used unfilled hydrogenated nitrile butadiene rubber (HNBR). The material composition and the processing details were reported elsewhere [32,33]. The first set of experiments used a rubber half-cylinder (radius $R = 1.5$ cm, length $L = 7$ cm) confined between two flat, smooth, and parallel Polymethylmethacrylat (PMMA) surfaces at a separation about 10%–20% smaller than the radius of the rubber cylinder. This resulted in a rectangular region of (apparent) Hertz-like contact with the width $2a \approx 1$ cm (see fig. 1). The system was kept at room temperature for about 12 hours before being put in a cold room with temperature -40°C . After staying in the cold room for about 12 hours we removed the rubber-PMMA contact and observed frozen-in elastic deformation (see fig. 1(b)). Similar observations were made in [33] for button-shaped HNBR specimens exposed to temperatures below the glass transition temperature T_g .

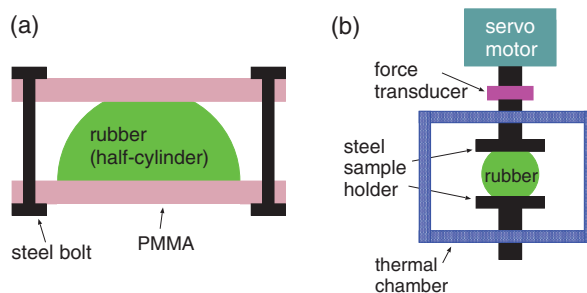


Fig. 2: (Colour online) Schematics of the two set-ups used for studying adhesion at low temperatures.

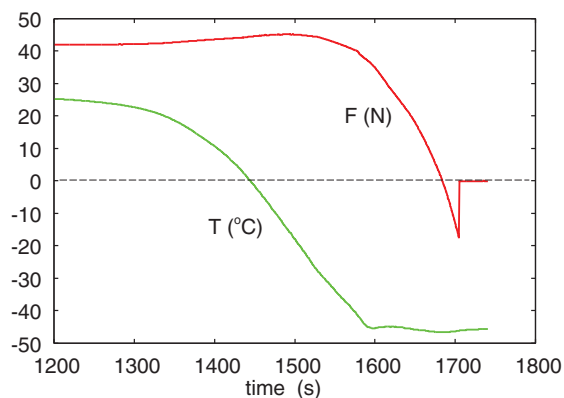


Fig. 3: (Colour online) The rubber O-ring with smooth surface initially compressed 10% and cooled down. The compressive force acting on the steel plate (red line) and the temperature (green line) as a function of time is depicted. Note that the temperature plot indicates the temperature inside the chamber, but not the rubber O-ring internal temperature.

For the second set of adhesion experiments we have used a Dynamic Mechanical Analysis (DMA) apparatus (Netzsch-Gabo Eplexor 150 machine with a thermal chamber and a 150 N force transducer). The machine was operated in the compression mode. The compression holders were cleaned with ethanol prior to each experiment. For the experiment, cylindrical specimens of 2.4–2.8 cm length were cut from rubber O-rings having a nominal cross-section diameter of $2R \approx 0.53$ cm.

The cylindrical specimens were compressed in the radial direction by the DMA machine and let relax for 1000 s under the constant deformation. Then the chamber was cooled down to -50°C keeping the displacement constant. The force, clamp separation and the chamber temperature were recorded during the experiment. At some temperature, below the glass transition point of the rubber, the compressive force gradually decreased down to zero (due to the frozen-in deformation field and the thermal contraction of the material; see below), and even to negative values due to the adhesive strength of the rubber-steel contact; see fig. 3. Once the tensile stress in the rubber near the interface reached the rubber-steel bond strength,

Table 1: The change in the force (pull-off force) during the abrupt detachment, induced by the thermal contraction of the rubber at low temperature (LT), and due to pull-off (at speed $v_z = 140 \mu\text{m/s}$) at room temperature (RT). In the first case the system is prepared at room temperature and then cooled down to -50°C in $\approx 300\text{s}$ (see fig. 3). The pull-off force was measured for 10% and 20% compression of the rubber O-ring, and for all surfaces cleaned by ethanol. We show results for smooth rubber and roughened rubber surface.

Surface condition	10% compression	20% compression
Smooth, LT	$20.4 \pm 3.0 \text{ N}$	$31.4 \pm 8.8 \text{ N}$
Rough, LT	$4.8 \pm 0.2 \text{ N}$	–
Smooth, RT	$0.9 \pm 0.2 \text{ N}$	–
Rough, RT	$0.015 \pm 0.015 \text{ N}$	–

breakage of the adhesive bonds (opening crack propagation) occurred, and the force vanished abruptly. We performed several experiments with different compression of the rubber O-ring, and for smooth and rough rubber surfaces.

We also performed some pull-off experiments with the system at room temperature. In this case we obtained the pull-off force (at the pull-off speed $v_z = 140 \mu\text{m/s}$): $0.9 \pm 0.2 \text{ N}$ for the smooth, clean rubber O-ring and $0.015 \pm 0.015 \text{ N}$ for the roughened rubber O-ring. These values should be compared to the 20.4 N and 4.8 N observed during (the thermal contraction-induced) pull-off when the system temperature is below the rubber glass transition temperature; see table 1 for a summary.

Adhesion in a room temperature contact. – The adhesive interaction between an elastic cylinder and a flat surface is a straightforward extension of the classical theory of the interaction between a spherical body and a flat surface known as Jonsen-Kendel-Roberts (JKR) theory [34]. If the applied pull-off force is orthogonal to the flat contact region, the pull-off force per unit length is given by [35–37]

$$f_c = -\frac{3\pi E_0^* a_c^2}{4R},$$

where the half-width of the the rectangular contact region at the pull-off instability is given by

$$a_c = \left(\frac{2wR^2}{\pi E_0^*} \right)^{1/3},$$

and the effective low-frequency Young modulus $E_0^* = E_0/(1-\nu^2)$, where E_0 and ν are the rubber low-frequency Young modulus and Poisson ratio, respectively. For HNBR rubber against smooth glass surface, for an opening crack at the pull-off velocity $\approx 1 \mu\text{m/s}$ we have measured the work of adhesion $w \approx 0.1 \text{ J/m}^2$ [21], which is influenced by both the viscoelastic properties and the surface roughness of the rubber object [21–24,38–40]. However, the pull-off velocity in our experiments is ~ 100 times higher and theory predicts for this case a ~ 10 times higher

work of adhesion because of the viscoelastic enhancement factor (see refs. [41–46]). Assuming $w \approx 1 \text{ J/m}^2$ for HNBR rubber against the steel surface (at the pull-off speed $\approx 100 \mu\text{m/s}$) we get $a_c \approx 0.1 \text{ mm}$ and the pull-off force $F_c/L = f_c = 40 \text{ N/m}$ or for our $L = 2.6 \text{ cm}$ long rubber cylinder, about $F_c \approx 1 \text{ N}$. This low pull-off force is close to our measured pull-off force at room temperature ($0.9 \pm 0.2 \text{ N}$).

Adhesion in a cooled contact. – For the system which was cooled below the rubber glass transition temperature we observed very strong adhesion (see above). This can be understood qualitatively as follows. First note that the adhesion observed at room temperature is so small as a result of the elastic energy stored at the interface during the compression of the rubber cylinder. That is, during removal of the steel (or PMMA) sheet, the elastic energy stored at the interface helps to break the adhesive bonds and result in the small pull-off force observed. Now when the (compressed) system is cooled to $T = -50^\circ\text{C}$ (or $T = -40^\circ\text{C}$ as in the first experiment), which is way below the rubber glass transition temperature ($T_g \approx -23^\circ\text{C}$), the elastic deformation field is frozen-in and not given back during the pull-off. This frozen-in deformation field can be clearly seen in fig. 1(b) where a flat rectangular region is observed on the top of the rubber cylinder. As the rubber heats up, this flat region disappears, and the rubber regains its original perfect cylinder shape with half-circular cross-section. However, a very large pull-off force is necessary to separate the rubber and a rigid countersurface if the rubber is below the glass transition temperature. Consider, for example, the limiting case where the rubber surface within the rectangular contact region (width $2a$) can be considered to be in full contact with the countersurface, and assume that *no elastic energy is given back during pull-off*. In this case, using the theory of cracks, we estimate the force needed for pull-off when the applied force is orthogonal to the flat contact region:

$$f_{\text{pull-off}} \approx (2\pi waE_1^*)^{1/2},$$

where E_1^* is the (effective) elastic modulus of the rubber in the glassy response region (or high-frequency modulus), which is of the order of several GPa. Using that $a \approx (R\delta)^{1/2}$ (where δ is the penetration) we get for $\sim 10\%$ compression (*i.e.*, $\delta = 0.1R$) $a \approx 0.5 \text{ mm}$. Thus, with $L = 2.6 \text{ cm}$ and $E_1^* = 2 \text{ GPa}$, we get the pull-off force $F = Lf \approx 17 \text{ N}$, which is close to what we observe for the smooth clean rubber O-ring section (see table 1). Here we have assumed [47] $w = 0.07 \text{ J/m}^2$ because when the rubber is in the glassy region it behaves as an elastic solid, and there is no viscoelastic enhancement factor. If the contact is incomplete within the nominal contact area, the work of adhesion and, hence, the calculated pull-off force is smaller.

When the rubber surface was roughened with sandpaper the pull-off force at low temperature did not decrease

as one would expect based on experiments at room temperature. This is due to the fact that the elastic deformation field imposed on the asperity contact regions during the contact formation at room temperature, is frozen-in when the temperature is lowered to $T = -50^\circ\text{C}$ (or to $T = -40^\circ\text{C}$ in the first experiment), and hence no elastic asperity deformation energy is given back during pull-off at $T = -50^\circ\text{C}$. Table 1 shows that the pull-off force at low temperature on the roughened surface is a factor $4.8/20.4 \sim 0.24$ smaller than for the smooth rubber surface. We interpret this as reflecting a change in the contact area: if we assume the work of adhesion w is proportional to the contact area we get a decrease by a factor of $(4.8/20.4)^2 \approx 0.06$ in the contact area upon roughening of the rubber surface. In particular, if the relative contact area $A/A_0 = 1$ for the smooth rubber surface we get $A/A_0 = 0.06$ for the roughened surface. The pull-off force at room temperature scales with w as $w^{2/3}$, so if the change in the contact area would be the only reason for the drop in adhesion at room temperature, one would expect the pull-off force at room temperature to be $(0.9\text{ N}) \times (0.06)^{2/3} \approx 0.14\text{ N}$. However, the observed pull-off force is $0.015 \pm 0.015\text{ N}$. This shows, as already pointed out, that the main reason for the low adhesion observed at room temperature is not the reduction in the contact area, but rather the elastic (asperity) deformation energy stored at the interface.

Discussion. – We have studied adhesion between rubber and flat PMMA and steel surfaces when the system is cooled (starting with a contact formed at room temperature) below the rubber glass transition temperature. When the separation between the rubber and the counter-surface occurs at room temperature negligible adhesion is detected, which is due to the elastic deformation energy stored at the interface, which is given back during pull-off and helps to break the adhesive bonds resulting in the negligible pull-off force observed in this case. When the system is cooled down below the glass transition temperature, the elastic deformation imposed on the system at room temperature is “frozen-in”, and the stored-up elastic energy is not given back during separation at the low temperature. This results in a huge pull-off force. Here we note that this phenomenon is similar to liquid glues which is put between two surfaces and harden with increasing time, *e.g.*, due to formation of cross-links or due to evaporation of a liquid component. This results in a solid in perfect contact with the substrate and without elastic energy stored-up at the interface, at least in the ideal case where no stresses develop in the adhesive during the transformation from the liquid to solid state. Thus, the absence of the elastic deformation energy can result in very strong adhesive bonds. A good example is cyanoacrylate adhesives (“superglues”) which cure through reaction with moisture held on the surface to be bonded.

In the second experiment reported on above, the pull-off force resulted from the thermally induced contraction of

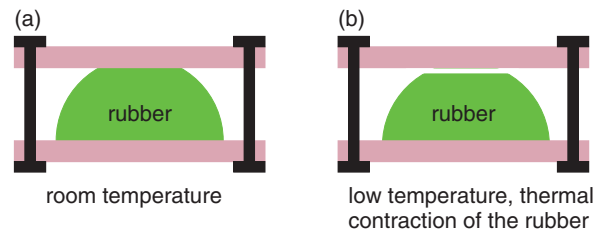


Fig. 4: (Colour online) (a) When the confined rubber cylinder is cooled below the rubber glass transition temperature, the deformation field imposed at room temperature becomes frozen-in. (b) If the system is cooled further below the glass transition temperature, the surfaces may separate at the adhering interface because of the thermal contraction mismatch. This assumes that the thermal contraction of the rubber compound is larger than the thermal contraction of the “box” containing the rubber cylinder, which is typically the case owing to the relative large thermal expansion coefficients of rubber materials. Considering low-temperature application of rubber seals, this large thermal contraction in combination with the poor shape recovery at and below the glass transition might result in an abrupt increase in the leak rate as the surfaces separate.

the rubber as it is cooled to low temperature. The thermal expansion coefficient of most rubber materials is much larger than that of steel and other metals or metal alloys. For example, for carbon steel and the rubber compound at $T = -33^\circ\text{C}$, the linear thermal expansion coefficient $\alpha = (dL/dT)/L_0$ (where L_0 is the length of the sample at room temperature) are $\approx 1 \times 10^{-5}\text{ K}^{-1}$ [48] and $\approx 8 \times 10^{-5}\text{ K}^{-1}$ [32], respectively. Thus from the glass transition temperature to $T = -50^\circ\text{C}$, we expect the rubber to contract with $\approx 0.2\%$, or (for the $2R = 0.53\text{ cm}$ rubber O-ring) $\Delta R \approx 10\ \mu\text{m}$ in the radial direction. If no contraction would be possible (*e.g.*, by adhesion to the confining plates), one would instead expect a stress of order $\sigma \approx E_1 \Delta R/R \approx 4\text{ MPa}$ to develop, and a normal force $F \approx L2a\sigma \approx 100\text{ N}$, which is larger than the adhesive force found above. Hence, the adhesive bond will break during the cooling down as indeed observed.

To summarize, when a rubber O-ring is confined between steel surfaces (as in a typical application) if the temperature is lowered below the rubber glass transition temperature, where the rubber deformation field is “frozen-in”, the mismatch between thermal expansion of the rubber and the confining box material, may result in a finite surface separation at low temperatures (see fig. 4). This would result in a dramatic increase of leakage of the seal. In another set of experiments [49], we indeed observed an abrupt increase in the gas leakage of rubber O-ring seals at low temperatures, which we interpret as resulting from the combination of the frozen-in rubber deformation field, and the thermal contraction of the rubber O-ring. In this case, we measure both the rubber contact force acting on the steel surface, and the gas leakage, which abruptly increased at the same time as the contact force

abruptly changed. The observed cold pull-off force for the seal rings in the leakage experiments is consistent with the measurements presented above.

Summary and conclusion. – We have studied adhesion between rubber and flat smooth PMMA and steel surfaces, when the system is cooled (starting with a contact formed at room temperature) below the rubber glass transition temperature. When the separation between the rubber and the countersurface occurs at room temperature, small (for smooth rubber surface) or no (for roughened rubber surface) adhesion can be detected, which is due to the elastic deformation energy stored at the interface, which is given back during pull-off and help to break the adhesive bonds resulting in the small pull-off force observed in these cases. When the system is cooled down below the glass transition temperature, the elastic deformation imposed on the system at room temperature is “frozen-in” and the stored-up elastic energy is not given back during pull-off at the low temperature. This results in a huge pull-off force. The study above is crucial for applications involving rubber at low temperatures, *e.g.*, in many applications where rubber seals operate at very low service temperatures, *e.g.*, in some mining and oil-field applications in regions with cold climate or in space applications.

* * *

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