

Double replication for characterizing cracks in surface-hardened PDMS

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Due to its stretchability the well-known elastomer PDMS is highly interesting for a wide range of applications¹. Typically, the surface of the PDMS has to be modified to improve its adhesion to other materials. Common methods for surface modification are plasma or (V)UV treatment; such tuning of the surface energy is inherently coupled with surface hardening so that surface cracks may occur, in particular under mechanical loading. In the majority of cases cracks should be avoided²; however, controlled cracking may be of interest, too³, provided that the cracks are well-characterized.

In our study the cracking of surface-hardened PDMS is induced in a motor-driven clamping unit that ensures a uniaxial straining of the sample. Beforehand, the PDMS is hardened by exposure to VUV light at 172 nm with an excimer lamp. The impact of preparational issues is addressed, in particular the aging of PDMS during storage and the effect of pre-evacuation before cross-linking. The experimental procedure is as follows. First the critical strain for cracking is detected, the strain at which the first cracks occur during stepwise stretching of the PDMS (Fig. 1a). To freeze this state, a first replication into OrmoStamp is done (Fig. 1b). To analyze the behavior of the cracks upon further stretching, additional replications at higher strain are performed. For quantifying the crack characteristics (shape, width, depth) cross-sections are beneficial. Therefore, a second replication into SU-8 on Si is conducted (Fig. 1c), which then is analyzed in cross-section by SEM.

Fig. 2 illustrates the effect of the preparation conditions on the critical strain for cracking. Upon storage PDMS softens, where pre-evacuation stabilizes the cracking behavior. Fig. 3 shows examples of replicated cracks after further stretching. Fig. 4 gives typical evaluation results for the characteristic parameters crack spacing, crack width and crack depth.

The study will show that reproducible cracking can be obtained when the preparation conditions chosen are adequate. The evaluated parameters will be discussed on the basis of crack theory^{4,5}.

¹ S. Wagner, S. Bauer, MRS Bull. **37**, 207 (2012).

² A. Polywka et al., Adv. Mater. **27**, 3755 (2015).

³ R. Seghir, S. Arscott, Sci. Rep. **5**, 14787 (2015); A. Polywka et al, Nanomaterials **6**, 168 (2016).

⁴ Z. C. Xia, J. W. Hutchinson, J. Mech. Phys. Solids **48**, 1107 (2000).

⁵ J. Huang et al., J. Mater. Sci. **49**, 255 (2014).

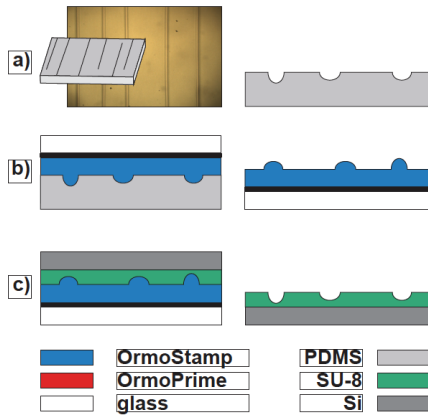


Fig. 1: Double replication for characterizing cracks.
 a) Hardened PDMS with cracks,
 b) 1st replication in OrmoStamp,
 c) 2nd replication in SU-8 on Si for preparing cross-sections.

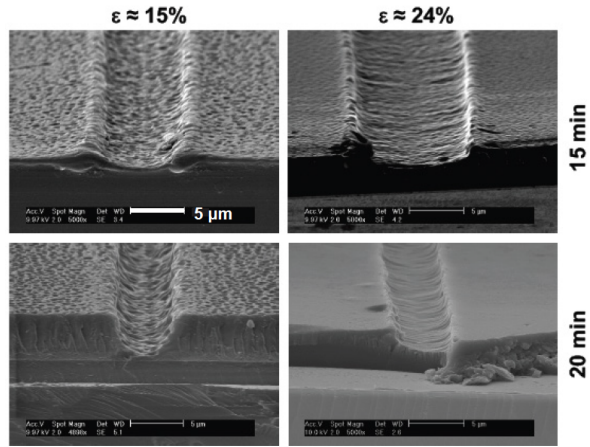


Fig. 3: Cross-sectional view of cracks.
 2nd replication (SU-8 on Si) of cracks at critical strain ($\approx 15\%$) and beyond ($\approx 24\%$) (pre-evacuated before cross-linking, storage time 1 day, 15 min/20 min VUV surface treatment, similar scale bars).

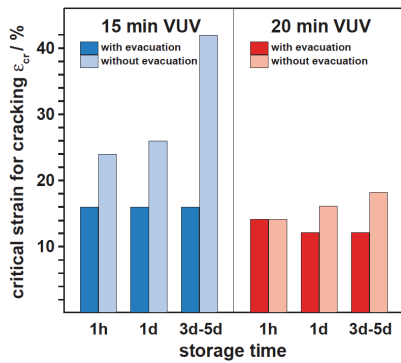


Fig. 2: Critical strain for initial formation of cracks (ϵ_{cr}) after different sample storage times. (1 hour, 1 day, 3-5 days), VUV treatment time 15/20 min, Full color: with pre-evacuation. Shaded: without pre-evacuation.

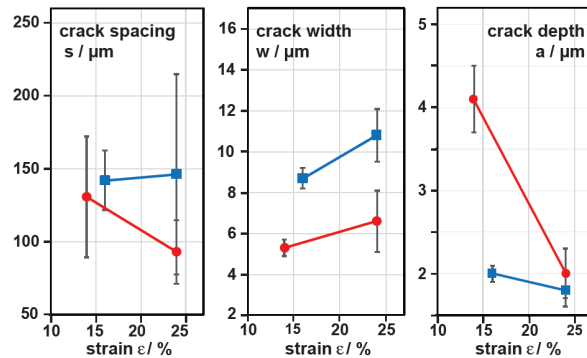


Fig. 4: Parameters crack spacing, crack width and crack depth at different strain. (with pre-evacuation before cross-linking, storage time 1 day, VUV treatment time blue: 15 min / red: 20 min), Left mark: at critical strain (ϵ_{cr}). Right mark: at increased strain.