

Characterization and microscopic precursors of failure in a polymer network

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We use space and time-resolved dynamic light scattering in the highly multiple scattering regime (Diffusing Wave Spectroscopy, DWS [1]) to probe the microscopic dynamics of amorphous solids during mechanical tests. Our experiment allows us to measure a coarse-grained map of the microscopic motion in the sample, by measuring the local mean squared displacement down to a resolution of a fraction of nm², averaged over regions of interest (ROIs) of typical size of tens of microns. Additionally, we measure the mesoscopic drift of the same ROIs defined for the microscopic dynamics.

We study a semi-crystalline polymer (PolyEther Ether Ketone, PEEK) and an elastomer (PolyDiMethyl-Siloxane, PDMS) charged with particles (Titanium dioxide, TiO₂) sized of 300nm, submitted to elongational mechanical tests. The test protocol consists of several stretching steps, each of them followed by a relaxation step during which the elongational strain is kept constant. The test is run until the material macroscopically fractures.

During the stretching step, the measured mesoscopic drift is in agreement with the affine deformation field expected from the imposed strain. During the first few steps, the microscopic displacement can be rationalized as being due to affine deformation, using a formalism developed in [2]. The relaxation steps following these early stretching steps are characterized by microscopic rearrangements that slow down with time, following the relaxation of the macroscopic stress required to keep a fixed strain.

A completely different scenario is observed as the material approaches macroscopic failure. The microscopic dynamics becomes order of magnitude faster than in the previous relaxation steps, unambiguous signalling microscopic rearrangements that eventually lead to macroscopic failure. Remarkably, this strong increase of the microscopic mobility occurs thousands of seconds before macroscopic failure, thereby demonstrating the great potential of these measurements as a way to detect precursor of material failure. Moreover, these dynamical precursor are spatially heterogeneous, the largest increase in microscopic mobility being observed in the region where a macroscopic fracture will occur.

We can translate this intensity correlation function to a mean square displacement over 10s using the

equation below[1]:

$$g_1(\tau) = \exp(-\gamma k_0 \langle \Delta r^2(\tau) \rangle^{1/2}) \quad (1)$$

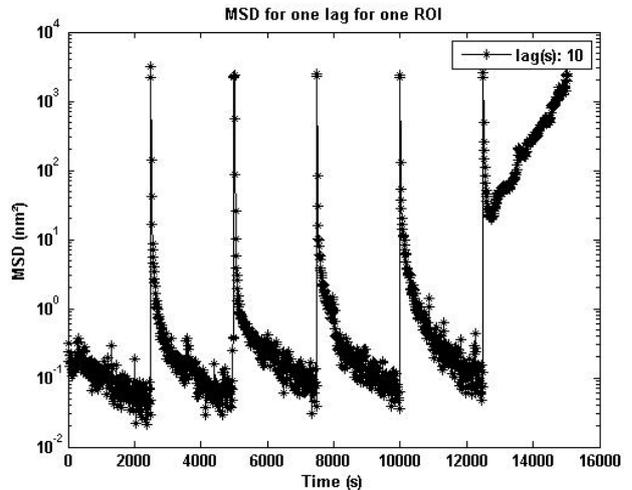


FIG. 1. $\langle \Delta r^2(10) \rangle$ Vs Time.

Then, we build a dynamic mapping (2D) to follow the microscopic dynamic of different region of interest on my sample. This Map indicates that the microscopic rearrangements present heterogeneity at relaxation phases when the sample is perfectly fixed. Moreover, if we compare the map result with an image after failure, we observe that the region with the more important microscopic rearrangements will be the region of failure.

That result allows us to pre-localize the region of the fracture.

These experiments show the interest of this technique as a method to predict and localize an eventual failure of a material.

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