PORE NUCLEATION AND GROWTH IN CELLULAR POLYMERS ANALYSED BY TIME RESOLVED SYNCHROTRON X-RAY µ-CT

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Summary: Spontaneous chemically-driven bubble generation and growth has been observed by means of real time synchrotron tomography in liquid polymers in presence/absence of nucleating nanoparticles. The 3D analysis allowed the determination of absolute nucleation rate versus time as well as the individual and average bubble growth dynamics.

1. INTRODUCTION

Bubble nucleation and growing is an interesting issue both from the applicative [1] and the theoretical [2] points of view. The simultaneous generation and later growth of these individual entities is the result of thermodynamic instability in a specific gas-liquid system. Nucleation process requires overcoming a certain energy barrier from the thermodynamical point of view [3]. The subsequent bubble growth is a separate process involving, as well, diverse physical interactions well described in the literature [4].

From the experimental point of view, there exist inherent difficulties for direct observation of nucleation since this phenomenon is strictly occurring at the nanoscale. In addition, the fast evolving dynamics require a high temporal resolution to visualize the created nuclei. Therefore the experimental approaches on nucleation are generally based on analysing already nucleated bubbles when have grown up to the micrometer range. If we assume no reversibility of nucleation and/or absence of any degeneration mechanism -likely improbable at this early stage- the study of micrometric size "pubescent" bubbles is equivalent. Nevertheless, most of nucleation and growth experimental studies are based in skewed 2D information. Thanks to the last developments at synchrotron beamlines it is possible to carry out time-resolved X-ray tomography reaching unthinkable time resolutions allowing 3D studies of the main foaming mechanisms in foaming systems

2. EXPERIMENTAL METHOD

X-ray tomographic microscopy was performed at the TOMCAT beamline of the Swiss Light Source at the Paul Scherrer Institute (Villigen, Switzerland). Monochromatic X-rays (20KeV, no filter) were used; generating 500 projections captured over 180° of rotation with a total scan time of 150ms and with adjusted pixel size to 3.2 µm. Span time of 5 s was used between scans.

A commercial bi-component polyurethane (PU) formulation from BASF was selected for this investigation. Two different cellular polymers were analysed, pure PU and PU containing 3 % by weight of a hydrophobic fumed nanosilica.

3. RESULTS

Reconstruction of the tomographic scans in time allows visualizing, in 3D, the process of bubbles nucleation and growth from the early stages of the foaming process in both studied materials. Fig.1 shows the corresponding 3D renderings for four selected instants during the foaming process. It is possible to appreciate the higher bubble size in the neat PU formulation and the smaller size together with a much higher density of bubbles in the

nanocomposite foam containing hydrophobic silica.

The analysis of the tomographic sequences allowed obtaining results concerning the evolution of both classical descriptors of cellular polymers such as mean cell size or number of bubbles generated and advanced features of the solid phase of cellular polymers such as fraction of mass in edges and mean cell wall thickness.

Acknowledgments

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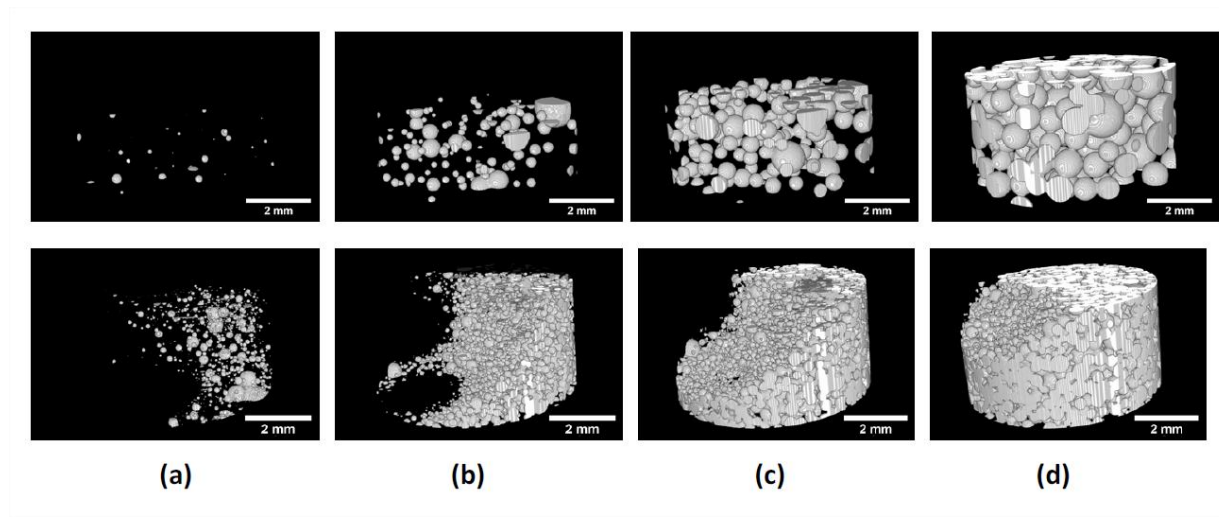(a)   (b)   (c)   (d)

Figure 1: 3D rendering at different instants for the two examined materials –top, pure PU; bottom PU with 3 by weight of nanosilica) at (a) 9.8s, (b) 21.7s, (c) 26.3s and (d) 32.6s.