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Application of a microfocus X-ray imaging apparatus to the study of cellular polymers

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ABSTRACT

This paper describes an X-ray based non-destructive imaging method to study polymer foams. A broad description of the main hardware elements – tube and detector specially adapted for low X-ray absorbing materials – is provided. Recommendations on the optimum imaging parameters for polymer foams are also reported. The mathematical equations and methodology to obtain quantitative information from the images are extensively explained, providing further discussion on the limitations and resolution of the technique. Some of the main potential applications of the technique are also summarised.

1. Introduction

Non-Destructive testing (NDT) comprises a wide group of analytical techniques used in science and industry to evaluate the properties of a material, component or system without causing damage [1]. The terms Non-Destructive Examination/Evaluation (NDE) and Non-Destructive Inspection (NDI) are also commonly used to describe these methods [2]. Because NDT does not alter the article being inspected, it is a highly valuable technique that permits saving both money and time in product evaluation, troubleshooting and research. Common NDT methods include vibration analysis, infrared thermography, acoustic emission analysis, ultrasonic imaging, X-ray computed tomography, digital radiography, ground penetrating radar, optical testing methods, magnetic-particles, penetrating liquids, gammagraphy, eddy-current testing and low coherence interferometry [2].

Since the 1920s, NDT has progressively developed from a laboratory curiosity to a powerful tool for science and industry. Modern non-destructive X-ray imaging is used by manufacturers to ensure product integrity and reliability, to improve product design and to provide in-line manufacturing control [3]. Alternatively, from the scientific point of view, X-ray imaging techniques in materials science have become more and more important since the early 1990's, and time and spatial resolution are, nowadays, beyond the microscale level [4]. In the particular case of cellular polymers, one of the most common non-destructive methods used by scientists is X-ray micro-tomography [5–10]. This ex-situ characterization method allows obtaining a detailed analysis of the internal architecture of the cellular polymer, which permits obtaining information both on the mesoscale (density profiles and presence of large defects) and on the microscale (cell size, cell wall thickness, anisotropy, etc). However, this powerful X-ray method has the main limitation of the relatively high scanning time required to obtain a high resolution 3D image, which makes extremely difficult to perform real-time experiments with time resolutions of a few seconds. In fact, this is only possible under very special conditions [11].

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In contrast, the alternative use of 2D X-ray imaging (radiography) and time resolved X-ray imaging (radioscopy) can be useful for the study of this type of materials both ex-situ and in-situ. Due to the small size of the structures to be resolved (cell walls thicknesses of a few microns) this technique necessarily involves the use of high resolution detectors and micro/nanofocus sources. This versatile technique enables observing structural details by encoding in its 2D projection the spatial variations in specimen density and atomic number though their transmitted intensities.

In-situ analysis of the foaming behaviour of cellular plastics has appeared in recent years. Different experimental challenges have to be considered depending of the type of base material: thermoset or thermoplastic. In the case of thermosets - usually foamed at room temperature and atmospheric pressure - the experimental set-up is simpler [12–15] than that needed for thermoplastic foams, foamed at higher temperatures. In this particular case, thermo-mechanical analysis [16,17] and optical expandometry [18] techniques have been used. In addition, advanced approaches to in-situ examine the foamability of thermoplastics in autoclaves by optical methods have been proposed recently [19]. On the other hand, the use of X-ray radioscopy to in-situ follow the foaming process of polymeric foams is still an emerging topic. Cunningham [20] evaluated the potential of X-ray radiography for the study of polymer foams and composites by film-radiography, showing diverse examples, and our laboratory has recently published a paper in this area, studying the foaming behaviour of a polyurethane system containing nanoclays [21].

X-ray radioscopy has been revealed as a powerful technique to examine the foaming behaviour of aluminium foams. The first successful results were obtained using synchrotron radioscopy in 2001 [22,23]. With this experience, a microfocus X-ray laboratory facility was built with the objective of analysing the foaming mechanism in these cellular materials [24–30].

Taking into account that there is a need for a better understanding of the foaming mechanisms in polymeric foams, a X-ray imaging system has been built. The low density of polymers and, as a consequence, the low contrast of the X-ray imaging makes the development of this system challenging from the technical point of view. This paper presents the technical requirements, the final design and the optimum operation parameters of this novel system. The method for density determination is explained in detail. In the final part, the outlook for the possible uses of this technique is provided.

2. Experimental techniques

2.1. Hardware description and mode of operation

The new microfocal X-ray equipment consists basically of two main elements. A closed, air-cooled microfocus X-ray source from Hamamatsu (Japan) is used to produce the X-rays (focal spot size: 5-20 μm, voltage: 20-100 kV, current: 0-200 μA,) with a maximum output power of 20 W. A flat panel (FP) detector from Hamamatsu (Japan) in combination with a frame grabber, Dalsa-Coreco, USA, provides the digital X-ray images. The high resolution detector is composed of a matrix of 2240 × 2344 pixels^2, with a pixel size of 50 μm, measuring 120 × 120 mm^2. Digital output is 12 bits depth resolution with an imaging capacity up to 9 fps at maximum acquisition velocity. Dedicated software from Hamamatsu (HiPic) is used to control the acquisition and storing of images.

The X-rays come from the source through a 150 μm-thick beryllium window forming an X-ray cone beam with an angle of 39° (see Fig. 1). Characteristic spot size is 5 μm for powers below 40% of the maximum level. For higher powers the spot size increases up to 20 μm. Flat panel (FP) detector technology essentially consists of a combination of a deposited scintillator material (CsI, GOS, Se, etc) layer in front of a CMOS detector.

The selection of both the X-ray tube and the detector was done taking into account that polymers are low absorbing materials and, therefore, we need to have sufficient soft X-rays to provide optimum contrast in the final image. In this sense, the source was selected due to its small spot size (related to final spatial resolution) and low minimum X-ray output energy (20 kV). On the other hand, the particular detector selected consisted of a custom-made *direct deposition* of CsI (scintillator material) on the CMOS surface. Direct deposition technology improves the efficiency by collecting in the CMOS detector a higher amount of the light produced in the scintillator, in comparison with other FP technologies in which scintillator is deposited on another substrate and there is a gap between it and the CMOS. This particular type of FP detector is covered by a 1 mm-thick carbon fibre plate, thus reducing the X-ray absorption as much as possible.

Both main elements are settled, one in front of each other, as shown in Fig. 1. In principle, it is possible to position them at any source to detector distance (*SD*) but, in general, optimum distances are in the range of 0.3 to 1.2 m. In this particular set up, the distance chosen was 600 mm.

The object is located at any position in between the detector and the source. The fact of having a cone X-ray beam allows for object magnification. Magnification factor, *M*, is a function of the object to source distance, *OS*, compared to *SD*:

$$M = \frac{OS}{SD} \tag{1}$$

As a first approach, pixel resolution is the result of dividing the pixel size by the magnification factor. Objects to be examined can, in principle, be of any size. Nevertheless, the maximum size depends on the magnification factor and on the total detector active area (120 × 120 mm^2). Thus, maximum field of view (at $M = 1$) corresponds to the active area and reduces with magnification. Object thickness (in the direction of the X-rays) also influences the image as discussed in the following sections.

2.2. Spatial, temporal and contrast resolution

Magnification, *M*, presents certain optical limits due to the finite size of both the focal spot, *F*, and the pixel size, P_s.