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# Detection of stress whitening in plastics with the help of X-ray dark field imaging

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ABSTRACT

The processing of thermoplastics can induce a wide range of defects such as stress whitening, cavitation and porosity, which can adversely affect the reliability of the final products. Hence, fast and effective non-destructive detection methods for such defects are highly important for quality assurance on production lines. In this paper, X-ray dark field imaging is presented as a new non-destructive testing method that allows the visualization of stress whitening or cavitation efficiently. The performance of the method is demonstrated for the case of an injection-moulded polyvinylidene fluoride part that exhibits stress whitening. Whereas the stress whitening could not be detected by conventional X-ray imaging, it was localized by an X-ray dark field image acquired within a few minutes. Once the precise location of the stress whitening was known, it was possible to verify the result by local micro X-ray computed tomography and by a micro section image.

## 1. Introduction

Deformation of thermoplastics during processing can induce a morphology with a range of specific defects [1] such as stress whitening (see for example [2]), cavitation (see for example [3]) or porosity (see for example [4]). Such defects or discontinuities may prove detrimental to the functionality, or at least reduce the expected service lifetime, of the parts. During injection-moulding, the material is highly deformed (strained) in the manufacturing process. As investigated and discussed in detail in [2] for polyvinylidene fluoride parts (PVDF), stress whitening under complex strain and temperature conditions is related to two microstructural mechanisms, namely the nucleation and the growth of micro voids. This can result in significant porosity which for commercial grades of PVDF initially is less than 1% [4]. In addition to that, mould shrinkage [5] can further contribute to damage under service conditions [6]. Hence, the detection and assessment of microscopic defects and discontinuities are of importance for quality assurance in production. On the other hand, there are PVDF polymer parts that are designed to contain pores providing specific functionality, such as semi-permeable membranes for various applications (see for example [7–10]), and information on uniformity of distribution and/or size of the pores may again be important. Non-destructive testing with advanced X-ray methods can provide information on pores and similar discontinuities in injection-moulded thermoplastic parts [11,12] or, in the case of functional porosity, on pore distribution and size. However, conventional X-ray testing methods, such as the ones presented in [11,12], cannot be applied economically in a production line for 100 % quality control. Synchrotron X-ray sources are too

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expensive and their accessibility is too limited. Inspection with X-ray tubes is either not sensitive to small defects with sizes of just a few microns (X-ray radiography), or the data acquisition requires a lot of time (typically 0.5 to 2 hours for computed tomography). Furthermore, with conventional X-ray methods, the region of interest is only about 3 orders larger than the spatial resolution.

In this paper, we evaluate the performance of X-ray dark field imaging [13] in detecting stress whitening in an injection-moulded polyvinylidene fluoride (PVDF) part. Stress whitening occurs when transparent or translucent plastic materials are put under mechanical tension giving rise to white stains. This phenomena is caused by local inhomogeneities of the refractive index which causes scatter of transmitted light in arbitrary directions. A possible cause for such inhomogeneities may be microvoids resulting from the injection moulding process [2,14]. If such microvoids are located inside opaque plastics they are concealed from visual inspection. Likewise, conventional X-ray inspection methods can reveal these voids only if the spatial resolution is high enough or if the number of voids is large enough to induce a measurable change in the mean density of the polymer [11,12]. However, dark field imaging with X-rays is very sensitive to clusters of small scattering centres within an object under investigation, even if the individual defects are 1-2 orders of magnitude smaller than the spatial resolution given by the actual pixel size of the X-ray image [15,16]. In this paper, small inhomogeneities of a plastic part causing stress whitening were found to scatter X-rays by a detectable amount. The detection of stress whitening with the new method was verified by local X-ray micro computed tomography and micro section images. The results obtained by these three methods are compared.

## 2. X-ray dark-field imaging

At low energies, the interaction of X-rays with matter can be described by a complex refractive index [17,18]:

$$n = 1 - \delta - i\beta = 1 - \frac{r_0}{2\pi}\lambda^2 \sum_q n_q f_q(0,E) \tag{1}$$

where 1-$\delta$ and $\beta$ are the real and the imaginary part of the complex refraction index, respectively, $r_0$ is the classical radius of an electron, $\lambda$ is the wavelength, $n_q$ is the number of atoms of type q per unit volume and $f_q(0, E)$ is the complex forward atomic scattering factor for atom q. The real and imaginary parts of $f_q$ are a function of the atomic photoabsorption cross section $\mu_a(E)$, and related to each other by a modified Kramers-Kronig relation [17,18]. The imaginary part $\beta$ describes the attenuation of the X-rays in matter and is related to the linear absorption coefficient $\mu_l(E)$ by:

$$\mu_l(E) = \frac{4\pi\beta(E)}{\lambda} \tag{2}$$

In conventional X-ray testing, the attenuation of an X-ray beam by the sample under test is measured. A more recent technique, differential phase contrast imaging (DPCI) [19,20], allows measuring the refraction of X-rays by the test sample with a standard X-ray tube and a Talbot-Lau interferometer setup [21,22]. A schematic representation of such a setup is shown in Fig. 1:

The grid $G_0$ splits the X-ray beam into several individual beams with an improved spatial coherence. The phase grid $G_1$ divides the X-ray beam into two beams with a phase shift of $\pi$ or $\pi/2$. The two beams interfere with each other and generate self-images of the phase grid at the Talbot distances [23]. Since the spatial resolution of the X-ray detector is not sufficient to resolve these self-images, an analyzer grid with the same pitch as the self-image at a chosen Talbot distance is used to determine the local amplitude, phase and mean value of the periodic self-image [21,24]. The mean value is a measure of the attenuation of the X-ray beam in the test object, as in conventional X-ray imaging. Due to the refraction of the X-ray beam within the test object by an angle $\alpha$, a change $\varphi$ of the phase of the self-image results, which is given by [25]:

$$\varphi = 2\pi\frac{\alpha d}{p_2} \tag{3}$$

where d is the distance between the phase and the analyzer grid and $p_2$ is the period of the self-image. The amplitude of the self-image depends on the local coherence of the X-ray beam: A reduction of the amplitude indicates that the X-ray beam is scattered by small inhomogeneities in the sample, e.g. small voids or a rough surface. This imaging mode is referred to as X-ray dark-field imaging [13]. This technique proved to be especially sensitive to small voids in a sample [15].

The whitening of some (semi-) transparent plastics under tensile stress, called stress whitening, is assumed to result from small voids or cracks which can occur if a plastic sample is put under stress [2,26]. These voids scatter incoming light in random directions and, therefore, clusters of them are macroscopically visible as white spots. X-rays are also scattered diffusively by these voids and the onset of stress whitening can be measured, for example, by small angle X-ray scattering (SAXS) techniques [27]. Therefore, it is expected that stress whitening can also be seen by dark-field imaging with the help of a Talbot-Lau interferometer setup.

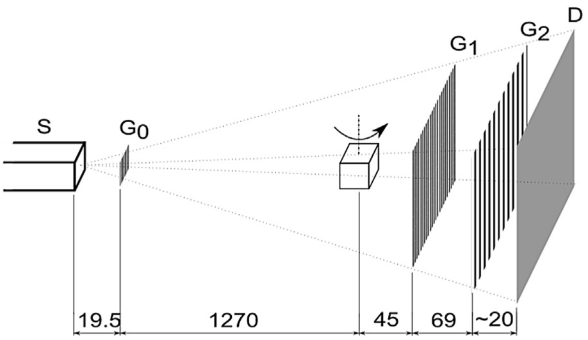

**Fig. 1.** Schematic setup of the Talbot-Lau interferometer used for the dark field imaging. S is the X-ray source, $G_0$, $G_1$ and $G_2$ the source, phase and analyzer grids, respectively. D is the X-ray detector. The numbers indicate the distances (in mm) used for the actual measurement.