X-Ray Dynamical Defectoscopy: A Way to study Damage Processes

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Abstract: *Recent theoretical models describe the dynamic development of voids and micro-cracks in materials under plastic deformation. For these models, experimental verification is needed. We propose direct and non-destructive observation of the material damage evolution by measuring changes in transmission of X-rays penetrating a stressed material, using a photon-counting X-ray imager. The present contribution demonstrates the application of a silicon hybrid pixel detector as such imager for measurements with a specimen of high-ductile aluminium alloy. The results of the first experiments aimed to detect the nucleation and evolution of damage clusters by the Medipix-1 detector are presented.*

1. INTRODUCTION

As many high performance structures in industry are constructed from ductile materials, there is a great requirement to determine the proper fracture criteria in the range of non-linear and large deformations. Intensive internal material damage, caused by different mechanisms, usually precedes the ductile fracture. For metals, for example, it is the process of nucleation, growth and coalescence of voids and microcracks described by a number of models [1]. These damage processes are time and loading dependent.

The sizes of voids and microcracks depend strongly on the stage of the damaging process and can grow from a few microns up to tens of microns depending on the material used. The velocity of physical processes connected with the development of voids and microcracks is rather high in the last stage of material failure in a gradually stressed specimen. Related experiments were done in the past [2] in context of fracture criteria testing. The duration of a fracture experiment was ≈10 min and the latest 20% of loading displacement proceeded in 5 sec typically.

The above-mentioned theories [1] need experimental verification of their assumptions and identification of a number of parameters for specification of the constitutive equations of a given material. For this purposes until now two tools exist: Fitting the experimental data of loading versus displacement dependence and metallographic stereology analysis.

We aim to observe the damage development by means of "X-ray Dynamic Defectoscopy" (XRDD) [3] based on a direct and non-destructive observation of changes in transmission of X-rays penetrating a stressed material and their registration using a position sensitive semiconductor single photon counting pixel detector (the so called Medipix detector) [4]. The measured changes of structural defects in time will be correlated with the time-dependent surface strain field measured at the same time using the optical method of "Deformed Circles" [5].

In this paper we report the results of our first experiments aimed to observe nucleation and evolution of damage clusters by a Medipix-1 pixel detector device.

2. THE X-RAY DYNAMIC DEFECTOSCOPY METHOD

The experimental set-up includes an X-ray source, a CCD-video system, a specimen with a crack fixed in loading equipment and a Medipix-1 X-ray detector assembly. For our experimental set-up for damage detection see Fig. 1. A hexagonal grid of dots is deposited on the specimen surface for optical measurement of strain field development.

The XRDD measurement is based on attenuation of an X-ray beam passing through the specimen. Spatial resolution in perpendicular direction is determined by the pixel size and the geometry of the illumination. The changes in attenuation of X-rays passing through a homogenous specimen of varying thickness were estimated experimentally. The difference between thicknesses is equivalent to an effective thickness reduction by the volume of defects and by contraction.

The thickness resolution was determined experimentally to be 40 μm in beam direction per one snapshot [3] for 5 mm thick specimen when full Medipix-1 counter ability is used. It means that it is possible to detect 0.8%

change in effective thickness reduction. A still better resolution in the beam direction will be possible by increasing the number of snapshots exposures for one loading level.

2.2. Imaging of X-ray beam attenuation

As an X-ray source we used the X-ray 35 kV tube of PHYWE apparatus with 1 μA current on Molybdenum anode. A diaphragm tube with a hole of 2 mm in diameter collimated the X-ray beam.

All transmission measurements were realized using Medipix-1 assemblies connected via a readout system interface board - called MUROS-1 (Medipix-1 re-Usable Read-Out System) [6] - to two commercial Nuclear Instruments (NI) cards inside a personal computer (PC). The MUROS-1 hardware with NI cards is controlled by dedicated software, called Medisoft 3 [6].

The Medipix-1 chip is formed by 64 x 64 square pixels of 170 μm pitch. It was designed at CERN within the framework of the Medipix-1 collaboration [7]. The Medipix-1 chip is bump-bonded to an equally segmented Si sensor (so-called pixel detector), which provides direct conversion of the energy deposited by single quanta of ionising radiation. Medipix-1 works fully linearly within its large dynamic range of 15 bits per pixel. Thanks to a very short dead time an amount of uncounted photons is negligible up to the count rate about 10^5 counts per second per pixel. This permits us to achieve high counting statistics per pixel and enables us to reach a high signal to noise ratio.

The detector position resolution and sensitivity are sufficient [8] with respect to our demands. The efficiency of photon-counting pixel detectors for X-rays is about fifty times higher than that for photographic emulsions or image phosphor plates. It also easily exceeds the efficiency and frame rate of CCD cameras with scintillator plates [9].

Output digital signals are recorded into a Personal Computer (PC), where they can be immediately processed. Thanks to the sensitivity, resolution and speed of the method, the nucleation and the growth of materials defects can be measured, even in a phase of their fast changes.

2.3. Method of Deformed Circles and video system

The development of internal damage is correlated with the strain field, as measured independently using the optical Method of Deformed Circles [5]. This method is based on optical monitoring of deformations of a hexagonal grid of dots deposited on the monitored specimen surface. Loading the specimen will deform a circle on the surface into an ellipse. Each ellipse is interpolated by six neighbouring dots on the hexagonal grid. The knowledge of the ellipse parameters yields directly the magnitude and the direction of main strain on the specimen surface.

A Mega pixel digital camera observes the strain evolution. The Video frames and MEDIPIX frames are grabbed into the PC for later processing.

The designed hexagonal grid has 0.2 mm distance between the dots. The interpolating circles on the unloaded specimen will then have 0.4 mm diameter, allowing determination of the positional shifts with 0.8 micrometer accuracy.

3. EXPERIMENTAL

The development of internal damage and its evolution in high-ductile aluminium alloy is the subject of this work. This material is well known from other experiments performed in the past; see e.g. [2]. A flat 5 mm thick specimen with a prefabricated central crack was selected. The specimens are loaded in uni-axial tension by grips displacement in a special frame. Experimental bodies are loaded step-by-step. Established damage and strain field is statically observed on each level of loading.

The loading equipment was designed as a simple mechanical frame; see Fig. 2. Two flat support beams with a central hole are used for measurement of the loading force. Two pairs of strain gauges, installed in both holes, establish this measurement. A pair of wedges supports each side of the specimen. Two pairs of screws passing through the support wedges establish the loading force. Displacement loading is controlled by two strain gauges on the sides of the specimen, which are positioned in the symmetry plane. This very stiff but simple design gives us the possibility to minimize the parasitic bending effect and results in a precisely controlled loading.

The experimental dependence of the strain on the uni-axial tension loading force is shown in the Fig. 3. Strain was measured on the sides of the specimen, as explained above. Marks represent the discrete loading levels investigated by X-rays.

The first damage cluster was observed at a loading level 90% of the maximal controlling gauges strain before macroscopic crack developing; see Fig. 4. Fig. 5 illustrates an increasing volume fraction of defects, which we have detected at the maximal loading force in diagram in the Fig. 3. Fig. 6 displays the X-ray image just before the crack instability. If we tried to go up with the loading beyond this level, the crack tips were propagating symmetrically up to 2/3 of the ligament; where both cracks stopped. Fig. 7 shows an image of the newly developed macroscopic crack. The axes in the figures have millimetres units. The coordinates [0,0] correspond to the initial crack tip. Contours level represents the volume fraction of defects in a percent scale. Contraction was separated by fast Fourier transform under the assumption that the spatial density variations due to contraction have a lower frequency than those due to the defects volume fraction. This approach simplifies the problem of separation of both components of effective specimen thickness difference but some influence of contraction remained.

4. CONCLUSIONS

This XRDD experiment shows that the resolution and signal stability of the Medipix-1 device are satisfactory for the observation of damage clusters by a collimated X-ray beam. However, if observation of single voids or microcracks is required, it will be necessary to use a micro-spot anode X-ray beam or synchrotron generated X-ray divergent beam to reach necessary magnification.

Applying the newly developed transferable loading frame we have been able to detect the stages of damage evolution resulting in crack growth and its instability. It will be necessary to use a very stiff dynamically controlled loading machine with fast feedback of immediate specimen displacement to observe the damage processes in the most interesting stage after passing the maximal loading force. This loading equipment will give us really dynamical analysis of the material time-dependent damage evolution. For the quantitative analysis and more precise tests, further methodological study is needed. It will be necessary to measure contraction independently in further studies.

The Medipix-1 spatial resolution is limited by its pixel size. The pixel size is 170 μm, which is several times worse than the resolution obtained in the thickness measurement. It is possible to improve the spatial resolution with the same pixel size, by using the magnification effect of a divergent X-ray source. Above that, the new Medipix-2 detector, which is under development [7], has a pixel size of 55 microns. This is more comparable to the resolution achieved at the thickness measurement. We plan to use the Medipix-2 and a micro-spot diverging X-ray beam in our next series of measurements.

Medipix-1 can take several hundreds snapshots per second, what seems to be enough to measure the development of voids and microcracks in the last stage of loading just before failure. To utilize the full counter capacity in this case, a high intensity tuneable X-ray source is needed. Such intensive X-rays are delivered by synchrotron beam based sources. The operation of a Medipix-1 device under high frame rate conditions with synchrotron equipment has already been reported in ref [10] and will be a subject of our further studies.

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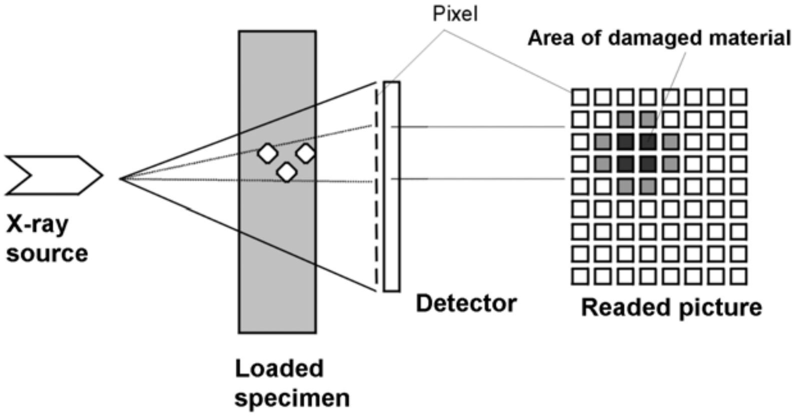

Figure 1: Schematic experimental setup

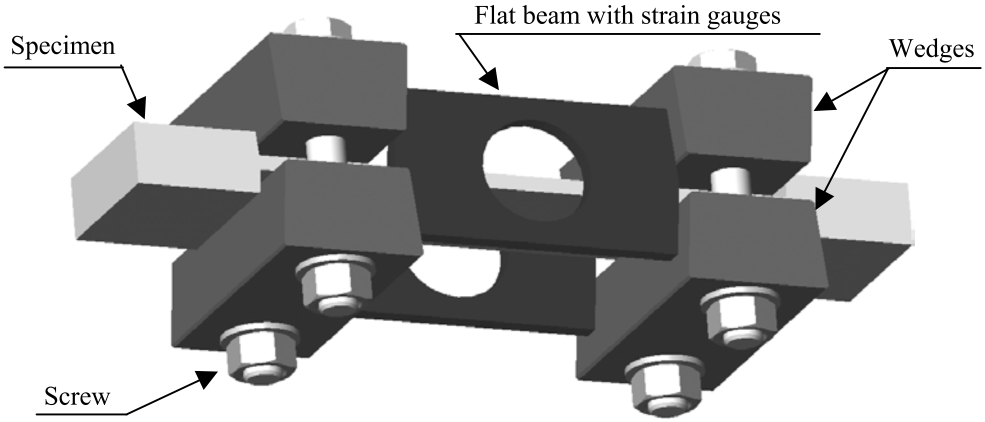

Figure 2: Static Loading frame. The loading is applied by a simple torque wrench on the 4 screws.

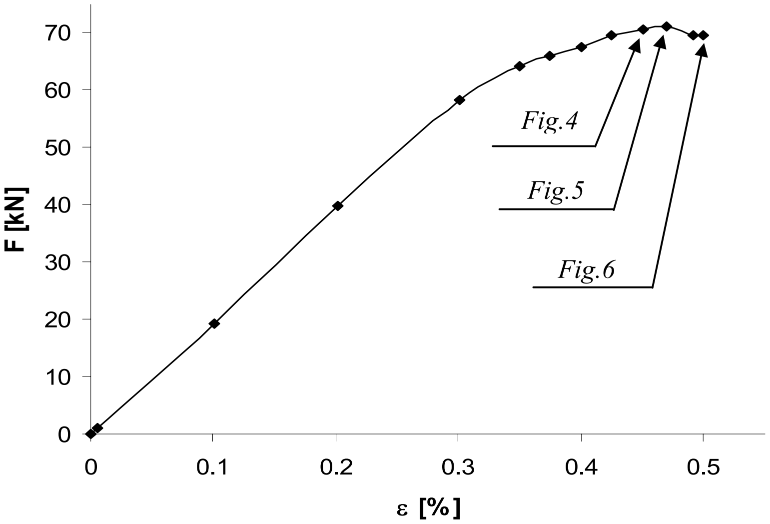

Figure 3: Experimental dependence of the expansion on the loading force-strain under uni-axial tension. The dots indicate the statically measured loading forces. Note that we were able to pass the point of highest force, without proceeding to full breaking of specimen.

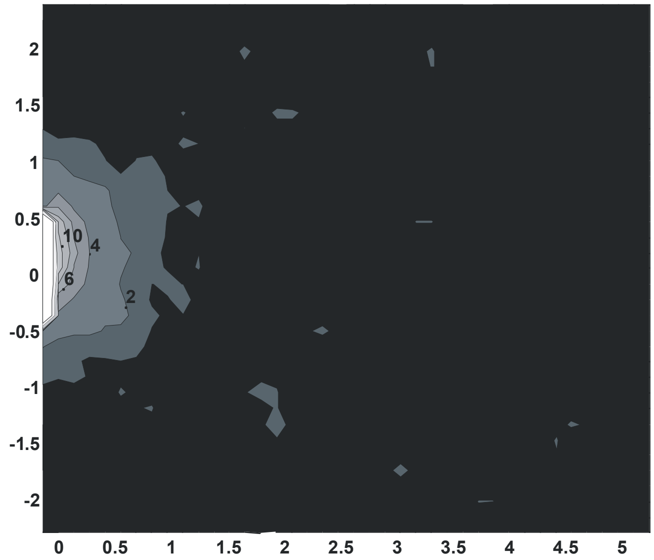

Figure 4: The first damage cluster was observed at a loading level 90% of the maximal controlling gauges strain before macroscopic crack developing;

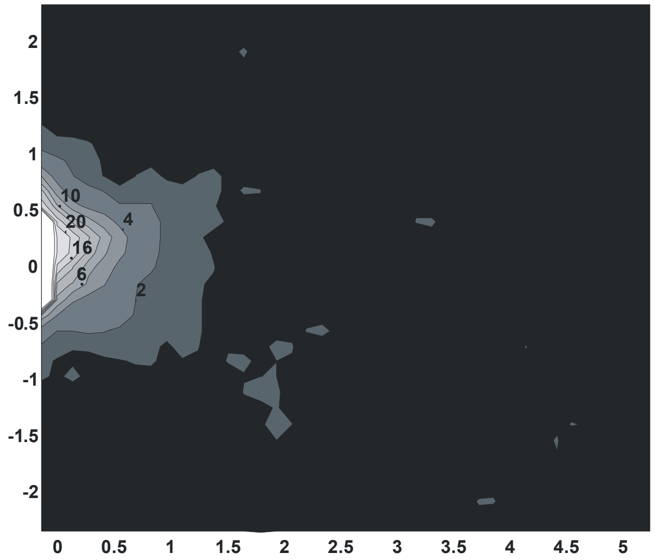

Figure 5: Damage, which we have detected at the maximal loading force in diagram in the Fig. 3

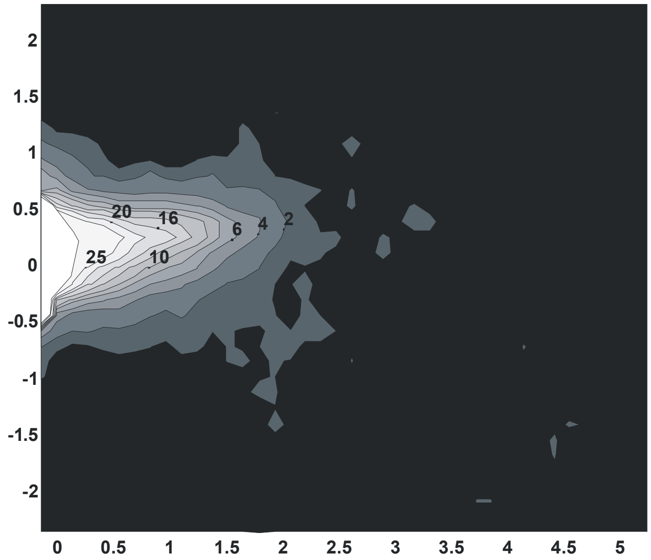

Figure 6: The X-ray image just before the crack instability

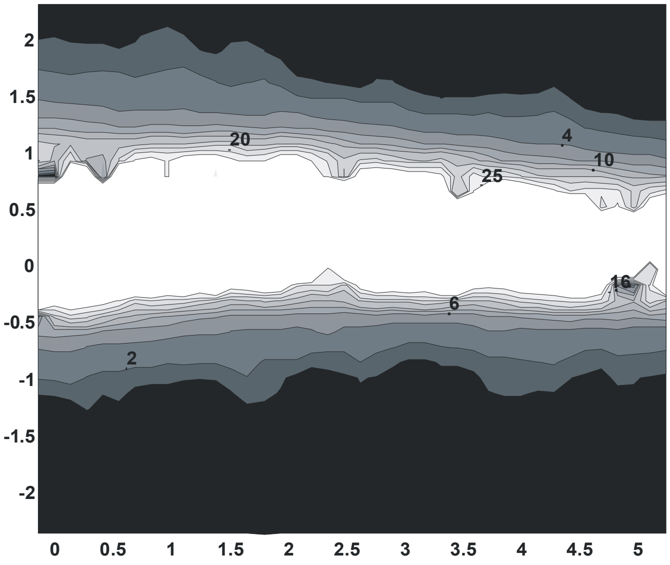

Figure 7: The image of the newly developed macroscopic crack.

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