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INTERGRANULAR CREEP CRACK MONITORING IN 316H USING DIGITAL IMAGE CORRELATION

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ABSTRACT: At elevated temperature (550°C) intergranular creep cracks have been observed in thermally and environmentally aged 316H stainless steel. To improve the understanding of mechanisms responsible of creep cracking, micromechanical experiments are proposed. An identification procedure of the crack tip position based on kinematic measurements is presented. Finite element simulations of intergranular cracks in bycristals have been performed and used as test fields to deform experimental images and to validate the image processing used for the identification.

1. INTRODUCTION

Since the 1980's, Digital Image Correlation (DIC) has been widely applied to study the deformation of materials [1] and to identify their behaviour. In parallel, significant advances have been made in microscopic image acquisition, especially in Scanning Electron Microscopy (SEM). The high resolution images acquired by microscopy can potentially be utilized to improve our understanding of crack incubation, initiation and growth in polycrystals. The DIC algorithms require spatially dense information and rely on local grayscale variations provided in our work by a surface deposition technique. A specific pattern was optimally designed for creep cracking characterization.

A phenomenological crystal plasticity law has been chosen to describe the plastic response of the 316H at the grain scale. This law was initially proposed by Méric and Cailletaud [2] and has already been validated for cyclic loadings in austenitic stainless steels [3]. Crystal plasticity laws are also relevant to model crystal creep deformation, for example on Ni-based single crystals [4] or in polycrystalline Ti-based alloys such as Ta6V [5].

At high temperature one of the most detrimental damage mechanism during creep is intergranular cracking. The aim of this paper is to propose and to validate a procedure allowing the monitoring of creep crack growth. The method is based on the analysis of kinematic fields obtained by Digital Image Correlation during *in-situ* elastic loading, at different steps of a conventional interrupted creep experiment.

The method is briefly described in the first paragraph. It is then validated on real images deformed by a known displacement field associated with the development of a crack either for a linear or non-linear behaviour, allowing a metrological characterization of the procedure.

2. EXPERIMENTAL PROCEDURE

Single Edge Notched Tensile specimens (SENT) will be submitted to interrupted microcreep tests and will be studied using SEM observations. By comparing SEM images of the unloaded specimen collected at different creep levels the plastic strains will be monitored at the microstructural scale. In addition, for each creep interruption, an *in-situ* loading device will be used to elastically deform the specimen. Combining this with an identification procedure similar to that proposed in [6], the crack tip position will be located.

3. KINEMATIC MEASUREMENT METHOD

We use a specific DIC formulation designated as "E-Kin" for Enriched Kinematics. It is based on a mesh discretization, as in the global approach discussed in [7]. The difference in this approach lies in the description of the kinematic fields where continuity conditions between the mesh elements can be adjusted. For example, the local approach can be retrieved when no continuity conditions are chosen, while normal and tangential displacement continuity can be introduced in the kinematics as in global methods.

The DIC formulation is based on texture conservation (Eq.1), where f(x) and g(x) are the reference and deformed images, respectively, and u is the unknown displacement field (generally taken as a low order spatial polynomial):

$$g(x) \cong f(x+u(x)) \tag{1}$$

Regarding local approaches, the texture conservation is generally considered in a least square sense on a given surface (the subset), leading to the introduction of the SSD (Sum of Squared Difference) criterion. The minimization of this criterion with respect to the introduced polynomial coefficients allows determining the local displacement field in the neighborhood of the subset. The minimization is performed separately onto each subset.

Firstly introduced in [8], the E-Kin approach allows introducing kinematical restrictions (continuity or jumps) between two adjacent subsets. When dealing with polynomial shape functions, these restrictions correspond to linear equations between the coefficients of the involved subsets. These equations act as constraints in the minimization of the correlation criterion.

Using a microlithography technique, a computer-designed pattern is deposited at the specimen surface, and consists in a combination of 10µm-step grid and of a random speckle pattern within each area (see Fig. 1a).This double-scale pattern is proposed to optimize the description of the strain gradients. The chosen mesh for the E-Kin

approach can be adjusted to this pattern by sake of consistency. In the example provided on Fig. 2c. it is made of quadrangles with a size of 30 pixels or $10\mu m$.

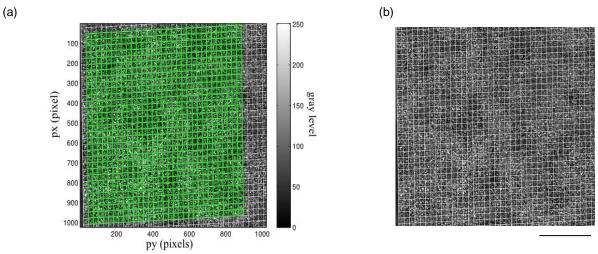


Figure 1- A possibility of mesh discretization used in the E-Kin correlation procedure (a) - Reference SEM image of the DIC pattern obtained experimentally and deformed using the computed displacement fields (b). The scale bar is 100µm in length.

4. VALIDATION OF THE DIC MEASUREMENT

The aim of this work is to assess the ability of different DIC approaches to precisely estimate the location of an inter-granular crack in different situations (*e.g.* elastic or plastic opening). In this paper, two error sources are addressed: the crack shape identification by DIC and the experimental errors related to the cumulated errors in the image acquisition chain: distortions [9], parallax errors, pattern evolution... In the following, we first present a numerical validation of the displacement field measurements associated with cracking. Then experimental errors are characterized through an experimental preliminary assessment involving re-positioning and pattern degradation.

4.1. Numerical validation

The numerical validation is performed using trial displacement fields computed using a reference displacement field obtained by a direct finite element calculation Code_Aster. An elastic isotropic behaviour is chosen. The crack position is perfectly known and uniform (planar) boundary conditions in displacement are used to load the 60mm-height structure, using a combination of modes *I* and *II*. The displacement fields obtained in mode *I* for an imposed vertical elongation of 0.4 mm are shown in Figure 2a. An experimental image obtained by SEM and shown in Figure 1a is then deformed using Gmic software [10] in accordance with the FE displacement field. The obtained deformed image is given in Figure 1b. This set of images is used to compare the efficiency of Local and E-Kin DIC methods. The Figure 2b 2c illustrate the measured displacement fields from which the crack position can be determined.

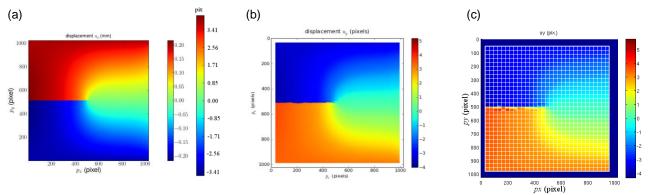


Figure 2- Vertical component of the displacement fields computed using Code_Aster (a), Local approach (b) and E-Kin approach with a horizontal mesh (c).

4.2. Experimental validations

In this section, we focus on errors that may arise from re-positioning of the specimen and the pattern damage due to oxidation at high temperature, both being related to our experimental procedure.

Re-positioning errors were addressed by capturing two SEM images separated by a specimen unmounting and remounting in the SEM holder, and performing the DIC (see Fig.3). To minimize errors, most of the SEM imaging parameters was kept constant for the two images acquisitions. The measured displacement is the combination of a rigid

body motion (displacement of the specimen) and of a localized deformation appearing as deformation band (amplitude: 0.005) (see Fig.4).

The creep experimental conditions (550°C in air) lead to oxide growth at the specimen surface. This oxide may change the quality of the pattern (contrast, heterogeneous oxidation, stains, *etc...*). A sample made of 316H covered with a tungsten pattern has been placed in a furnace during two weeks in the previously described environment (see Fig.5). The corresponding displacement field is illustrated in Fig. 5.c and 5.d. It corresponds to a rather homogeneous deformation.

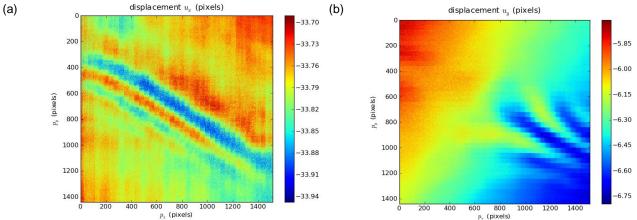


Figure 3- Displacement fields associated with repositioning in the horizontal (a) and vertical (b) direction, obtained with the local approach after an in and out way in the SEM chamber.

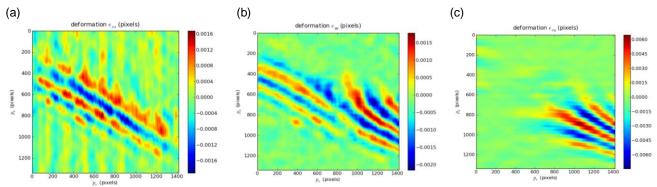


Figure 4- Strain fields obtained in the displacement fields of Fig. 3 in the horizontal (a), vertical (b) and in-plane shear components (c) directions with the local approach.

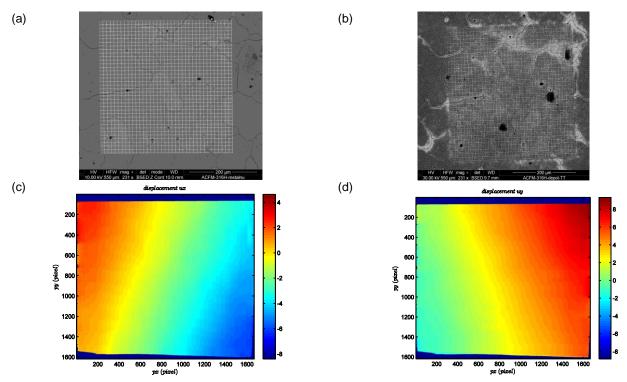


Figure 5- SEM images in BSE (Back Scattered Electron) Z contrast mode before (a) and after (b) ageing in air corresponding displacement fields in the horizontal (c) and vertical (d) direction, obtained with the local approach.

5. CONCLUSION AND PERSPECTIVES

At elevated temperature (550°C) intergranular creep cracks can potentially occur in thermally and environmentally aged 316H stainless steel. An identification procedure of the crack tip position based on kinematic measurements has been introduced (E-Kin). Its performance has been assessed using deformed images numerically built from displacement fields obtained by Code_Aster finite element simulations. An elastic isotropic behaviour and uniform boundary conditions in displacement are chosen. The displacement fields resulting from the loading of the structure have been used to deform an experimental SEM image. Based on the reference image, a mesh discretization relevant with the deposited pattern has been carried out. An explicit mesh of the crack allows capturing the displacement field in the cracked domain.

The SEM observations of the creep samples lead to a re-positioning procedure of the sample in the SEM. The measurement errors due to unintentional variations in the SEM imaging settings such as magnitication evolution have been estimated.

Further improvements related to the choice of the mesh element shape functions will be performed to better describe the kinematics in front of the crack and to locate the crack tip precisely. The effect of the environment (air at 550°C) on the DIC pattern conservation has been investigated. The surface preparation technique is still being improved to better fulfil the optical flow conservation hypothesis, while specific DIC criterions tolerant with pattern evolution at high temperature will be tested.

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