Deliverable 1.2 First strain fields from laboratory testing

Document type	**Deliverable D 1.2**
Document Version / Status	**1.00 - First issue**
Primary Authors	**Eric Blond, eric.blond@univ-orleans.fr, UORL**
Distribution Level	**PU (Public)**
Project Acronym	**ATHOR**
Project Title	**Advanced THermomechanical multiscale mOdelling of Refractory linings**
Grant Agreement Number	**764987**
Project Website	**www.etn-athor.eu**
Project Coordinator	**Marc Huger, marc.huger@unilim.fr, UNILIM**
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Change log

History of changes				
Version	**Date**	**Author (Organization)**	**Change**	**Page**
1.00	31.12.2018	Eric Blond (UORL)	First version of the D 1.2 deliverable for submission	all
1.10	05.02.2019	Eric Blond (UORL)	Updated and corrected	all
1.20	11.02.2019	Marc Huger (UNILIM)	Updated and ready for submission	all

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I Introduction

This report describes approaches for mechanical characterization of refractory materials using optical methods. Most of these optical methods are related to Digital Image Correlation (DIC) and are used from room to high temperature. The sub-sections below provide some basic definitions and a résumé of the main pros and cons of the different DIC methods for easy reading of the rest of the report. Thereafter, in Section II, the different types of DIC are detailed, giving particular focus to the Integrated Digital Image Correlation (I-DIC) and to the Two Parts Digital Image Correlation (2P-DIC), since they are more extensively used in the ATHOR project. Section III describes the main difficulties related to DIC at high temperatures (higher than 1 000°C), and also the possibilities to overcome them.

I a. Marker tracking

Marker tracking is an optical method that consists of tracking the markers that have been previously deposited on specimen's surface. It can be used to determine both displacement and strain fields during thermomechanical solicitation. This method requires regularly spaced markers in both directions and its precision depends both on the distance between the markers and the optical chain. Its main advantages are its theoretical simplicity, its relative insensitivity to variations of illumination and to in-plane sample rotation. This is similar to using a large number of extensometers simultaneously but it does not correspond to a real full-field measurement. The article of N. Bretagne et al.[1] fully describes the principles of this method, as well as its (in)sensitivity to experimental parameters.

I b. Digital Image Correlation (DIC)

Digital Image Correlation is an experimental full-field measurement method where a reference image, corresponding to the initial state of a sample, is compared with images of the same sample taken during the application of the load. The full displacements' field is computed by convolution product based on the gray levels conservation principle. This technique has been developed since the early 1980s, and since then it has substantially improved in terms of accuracy[2], although many challenges still exist, as will be explained in the following sections.

I c. In plane correlation (2D DIC) and stereovision (3D DIC)

The main difference between the 2- and 3-dimesional DIC is linked with ability to separate displacement field caused by in plane motions from the one caused by out of plane motions. The presence of the out of plane motions result in an artifact of plane displacement gradients. Depending of their level, they can cause distortions of the computed displacement field, thus altering measurement accuracy. Using standard 2D DIC (setup with one camera), the displacement fields in and out of the plane cannot be separated. The distortion caused by of out of plane displacement can only be minimized by increasing the distance between camera and sample or by usage of a telecentric lens[3]. Stereovision systems (3D DIC) have the capability to measure all three components of the displacement field. It is then possible to extract information about both in plane (first two components) and out of plane (third component) displacement and so to better evaluate de real in-plane strain field.

I d. Main pros and cons of DIC

The main advantage of the Digital Image Correlation technique is its ability to produce results from laboratory experiments comparable with numerical simulations. Indeed, this full-field measurement technique allows the direct comparison of the results from the Finite Element Method (F.E.M.) with the measured strain-field. Classical artifacts such the discrepancy between machine and specimen displacement, or between averaged strain field from LVDT, or the local one from the gauge and the real heterogeneous strain field at the surface of the specimen are avoided. Then it becomes possible to realize a full coupling between modeling (constitutive equations, boundary conditions) and the experimental measurements to enhance and enriched the material characterization. Moreover, the experimental setup is quite simple, no significant modifications of

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the room temperature set up are required. Relatively low requirements in terms of measurement environment (at RT) and a wide range of measurement sensitivity and resolution[4], as well as being contactless, non-intrusive, efficient and relatively cheap[5], make this method the most used in mechanics since the beginning of the 21st century.

Even-with the popularity of DIC today, it is essential to keep in mind three points. Firstly, only 2D motions are correctly captured with the standard set-up (projection in the plane of the camera). Secondly, the tested sample must have a random gray intensity distribution (natural or artificial) stable for the duration of the test (challenge at high temperatures) and, finally, that the measurements are highly dependent on the quality of the imaging system [4].

II Main DIC methods

In the literature, different Digital Image Correlation algorithms are described such as the Global-DIC[2], Local-DIC[6], Beam-DIC[7], 2P-DIC[8], I-DIC[9] and Stereo-DIC[10]. In the following sections, the algorithms considered in the ATHOR project will be briefly described.

II a. Local DIC and Global DIC

The classic DIC approach for the determination of displacements and strains' fields is based on the choice of the size of a region of interest (ROI) in the analyzed images, and the consequent tracking of the displacement of this region through the pixels' gray levels variation. What differs from one algorithm to another is the method used for the tracking.

In the subset-based DIC approach, each calculation point (attached to ROI) is tracked separately in the images, independently of the others, and the continuity of the displacements field isn't imposed as a priori condition[11]. The displacement is calculated for each sub-set. In the Finite Elements global DIC approach, all the discrete calculation points are connected by a mesh, such as in regular Finite Elements simulations, therefore ensuring the continuity, since all the displacements are calculated at the same time[11]. The displacement is calculated for each node between adjacent subsets.

In the literature, there are a considerable number of papers comparing both approaches regarding the accuracy, robustness and applicability to different cases. Both techniques have been applied successfully over the past few years and there is no consensus as to which is the best[5, 11, 12]. Finally, the choice of the most suitable one for a given application case relies on the level of familiarity of the researcher with each of them.

II b. Integrated-DIC (I-DIC)

Contrary to the previously described algorithms, in the Integrated Digital Image Correlation, the correlation is performed over the entire image and making a direct connection between the change in the gray levels of each pixel in the reference and deformed images[9]. Moreover, while DIC has been developed to measure displacement and strain field, I-DIC has been mainly developed to perform inverse identifications of material's parameters, since the algorithm intends to match the displacements' field from theoretically deformed images and real experimental images, as shown in Figure 1.

For I-DIC, a numerical simulation of real experiments, existing behavior laws are used with an initial guess for the material's parameters and boundaries conditions (from the modelling of the experimental set up). The numerical displacements' field obtained from the simulation is imposed on the reference image, and its pixels' gray levels are interpolated, creating the theoretically deformed image. This image is, then, compared with the deformed experimental image, and the relative error is calculated. If this error is below a given tolerance, the displacements' field is assumed to be correct, and also the behavior law and the material's parameters used in the numerical simulation. If not, an optimization algorithm, such as the Levenberg-Marquardt method, is used to predict the most suitable change in the parameters to converge towards the correct values.

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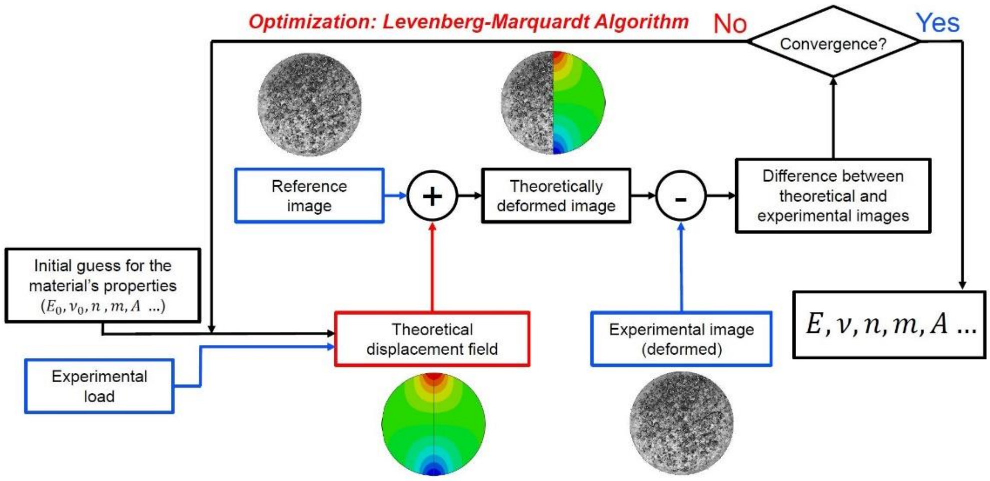

Figure 1 - I-DIC basic schematic algorithm

Levenberg-Marquardt algorithm

Due to its importance in the I-DIC algorithm used in ATHOR project, the Levenberg-Marquardt algorithm is briefly summarize hereafter.

The gradient based methods, so called because they require the calculation of the gradient g, which is well known to be the direction of higher increase/decrease of the function, are iterative in nature. Several methods have been proposed in the literature to solve the problem of finding the minima of functions with the best speed and accuracy as possible[13]. One of the most commonly used methods is the Steepest Descent Method, which uses the direction of maximum gradient descent to calculate the next guess for the parameters. Nevertheless, this method is slow in practice, since the gradient is a local property, and the method may end up iterating in a zig-zag fashion. The Gauss-Newton Method proposes the use of an approximation of the Hessian matrix in the determination of the variation of the parameters. However, the approximated Hessian isn't necessarily a positive definite, except near the minimum, which can lead to inconsistent results. To solve this problem, Marquardt [14] proposed a new method based on the fact that, if P is a positive definite matrix, then $A + \lambda P$ is positive definite for sufficiently large λ for all A. Using this concept, this method solves the following equation

$$\left(J^{T^k} J^k + \lambda^k I + H^k\right) \delta^{k+1} = -J^{T^k} r^k + g^k \tag{1}$$

where J is the Jacobian matrix, H is the second derivative of the error function and r is the residual. In this case, the identity matrix I was chosen to be the positive definite matrix considered.

This method is also advantageous considering that it can be seen as an interpolation between the Steepest Descent Method and the Gauss-Newton Method. As explained before, the first one isn't efficient close to the minimum because it assumes a zig-zag fashion convergence, while the second one isn't reliable far from the minimum, since the assumption of simplification of the Hessian matrix was made considering the residuals to be sufficiently small. Therefore, it seems to be a good idea to use the Steepest Descent Method far from the minimum and the Gauss-Newton Method close to the minimum.

Example of I-DIC application

During the course of the project ATHOR, a considerable effort has been made to improve the accuracy and speed of the available I-DIC code. Figure 2 shows the example of an identification conducted on virtually deformed images of Brazilian disc tests. A known displacements' field, generated using a numerical simulation, an isotropic linear elastic law and the given material's parameters were imposed on a reference image and, providing an initial guess for the Young's modulus different from the correct one, the identification procedure was launched. As can be observed, the recently implemented evolutions of the code, mainly related to a better implementation of the optimization algorithm, drastically reduce the number of necessary iterations to achieve

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convergence. This evolution of the code is important because it allows the researchers to test several behavior laws and initial guesses in a shorter time.

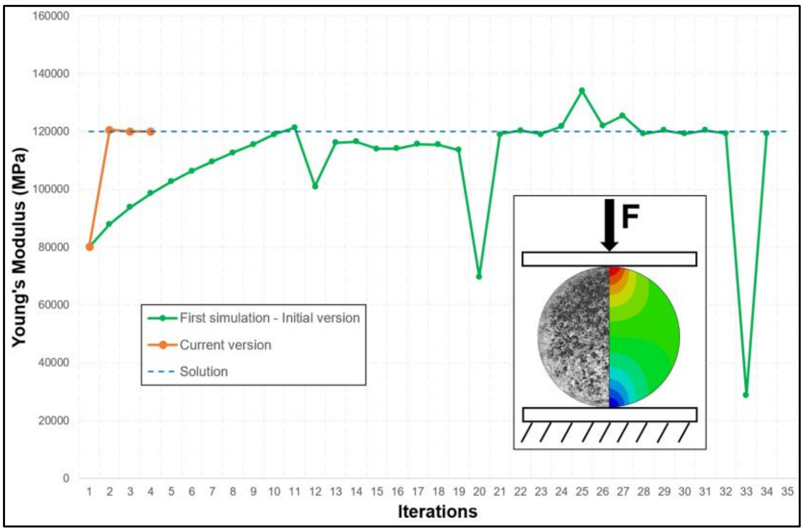

Figure 2 - Identification of the Young's modulus of a material using I-DIC

II c. Two parts digital image correlation (2P-DIC)

The DIC algorithm usually assumes the optical flow continuity on the surface and homogenous transformation of a material in each subset. That leads to the relatively low accuracy of the displacement field in the proximity of material discontinuities, such as cracks. A new technique has already been developed to improve the accuracy close to displacement discontinuity (i.e. cracks). It bears the name of the Two Parts Digital Image Correlation (2P-DIC), which originates from the implemented feature, enabling every subset to split in two parts. Therefore, the crack resolution is improved and better measurement accuracy is achieved. The 2P-DIC allows the detection of not only the presence and the position of a crack, but also its opening and crack length. Detailed description of the 2P-DIC algorithm is presented in the article of Jean-Christophe Dupré et al.[8]

III High temperature DIC

DIC measurements performed at high temperature require considering additional high temperature related phenomena. They are listed below together with methods already tested, reducing their distortive effect.

The refractive index variation caused by irregular flow of hot air between a sample and a camera produces a “heat haze” which blurs the image. This effect is at the origin of the artefact and loss of accuracy. It can be reduced by three main methods:

- Long integration time and neutral density filter(s) [15] producing a noise averaging.
- Neutral density filter[16] producing a noise averaging by reduction of light intensity (uniformly for a range of wavelengths). Available transmittance values of 50%, 25% or 12.5%.
- Small fan between sample and camera [17] – it tends to reduce heat haze in the measurement field;
- Sealed furnace with internal deflector and cooling windows to reduce the air flow between camera and sample.

The effect of the black body radiation, which modify the light intensity during the heating up and during the test depending on the thermal regulation, could be reduce using:

- Blue bandpass filter - transparent for the wavelengths in a range of 440-460 nm, which are less influenced by black body radiation (>550 nm);
- Additional illumination (wavelength depends on the wavelength absorbed by the bandpass filter – if blue bandpass filter, then blue light is used).

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The speckle pattern must remain unchanged at high temperature for a long time (creep test). Different solutions can be found in the literature, as shown in Table 1. It can be ensured using:

- High temperature resistant material applied by plasma spraying technique [15] – in order to obtain the contrast a mesh is used;
- High temperature resistant inorganic adhesive mixed with a high temperature resistant powder;
- Structure of a material as a pattern [18];

Table 1. Summary of a short bibliographical review about high temperature DIC experiments.

Specimen(s)	Experimental temperature	Speckle pattern	Reduction of black body radiation	Year
Chromium-nickel austenite **stainless steel** (1Cr18Ni9Ti)	**1200°C**	**Black cobalt oxide** blended with commercial high-temperature inorganic adhesive	**Blue band pass filter**	**2010**[19]
Carbon fiber	**2600°C**	Plasma spraying of **tungsten powder** (protective atmosphere) stable up to 2600°C	**Blue band-pass filter, neutral filters, polarizing filter**	**2014**[16]
Isopressed zirconium silicate product	**1350°C**	**Silicon carbide** powder	**Blue band pass filter (proposed selection of blue and green wavelengths)**	**2015**[15]
Fully annealed 309 **stainless steel** bars	**1150°C**	**White Y2O3 paint stable** up to 1500°C; **Black** high temperature **silica-based ceramic paint**	**Blue band-pass filter**; **Additional illumination**	**2016**[20]
Low-carbon 304 **stainless steel**	**900°C**	**White, thin layer of yttrium oxide** spray paint (resistant up to 1500°C). **Black high temperature silica-based ceramic paint**.	**Blue band pass filter**	**2017**[21]

Due to the temperature level intense light reflections are present, caused by e.g. interference effect of light reflected from the chamber with the one reflected from the sample.. This can be reduced by application of linear polarizing filters[16] which reduce reflections from the sample's surface.

III a. High temperature resistant speckle pattern developed for ATHOR project

One of the main objectives of the ATHOR project is to perform high temperature mechanical tests, such as the Wedge Splitting test and the Brazilian test using optical techniques (e.g. DIC). High-accuracy optical measurement techniques require optimal and persistent speckle patterns. For this reason, a few aspects should be taken into account:

- First of all, both investigated material and material of speckles should **resist the high temperature environment**. This feature has been evaluated by comparison of the sample's surface before and after heat treatment. The Figure 3 shows such comparison.
- Any possible **chemical reactions** between the materials should be **limited**. Otherwise, the speckle pattern might change (the color or shape of the speckles) and therefore information might be lost.
- Secondly, it is important, to **decrease possibility of powder debonding** from the sample's surface, as it would significantly modify the speckle pattern.
- The speckle size influences the accuracy of the measurement[22–25]. It is a work in progress. The tests consist of comparing the measured and imposed displacement for different speckle sizes. The **optimal speckle size** is often mentioned as being in a range of 3 to 5 pixels.
- It should be also noted, that **high contrast** between speckles and the investigated material, as well as **random speckle distribution,** are the key points of this measurement and should be a priority.

Ongoing development: Speckle pattern from Brown Fused Alumina powder (BFA)

Brown Fused Alumina (BFA) refractory powder (provided by Imerys) is one of the materials that have been tested for the potential application in a high-temperature resistant speckle pattern. White high strength alumina adhesive (903 HP Adhesive), supplied by Rebond, was applied to ensure reliable bonding between the investigated material and the BFA powder particles.

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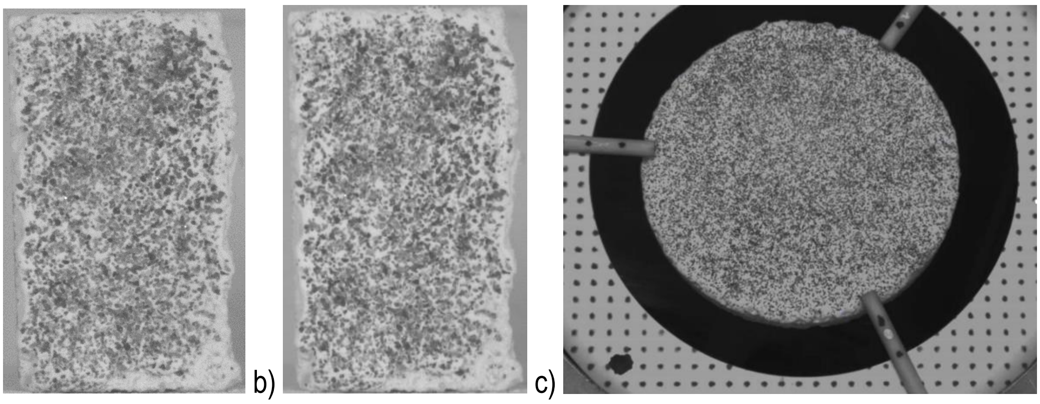
Comparing the speckle design to the requirements for the refractory speckle pattern, it can be stated that:

- **Resistance to high temperature** (above 1000°C) – is sufficiently high, as both adhesive agent (resistant until 1780°C) and brown fused alumina (produced by thermal fusion at temperatures around 2000°C) are stable in the investigated temperature range;
- **Possible chemical reactions** – are limited, as brown fused alumina powder and alumina adhesive should not react significantly (if at all);
- **Possibility of powder debonding** – is limited, as it is bonded using white high strength alumina adhesive;
- **Optimal speckle size** – obtained by applying a powder with selected grain size (depending from the measurement distance) corresponding to 3-5 pixels;
- **High contrast** – is ensured, as the brown powder adheres to the white material with the help of white adhesive;
- **Random speckle distribution** – is provided by powder spreading through the mesh;
- **Low powder porosity** – is another advantage of brown fused alumina. It is important, as the material with low porosity has less free surface, so the powder is more stable (less free surface to be reduced) at high temperature.

The current sample preparation procedure is:

1. Machining of a (disc shaped) sample (from a fired alumina-spinel refractory);
2. Manual mixing of white alumina adhesive to obtain homogenous suspension, which is then spread on the sample's surface;
3. Spreading of the brown fused alumina powder through the mesh;
4. Sample and image acquisition setup positioning – distance between camera and sample surface (e.g. for Wedge Splitting Test is about 800 mm.)
5. Taking a picture (Figure 5a) of the sample before heat treatment.
6. Heat treatment up to 1200°C – with heating rate of 5°C/min, dwell time of 1h and cooling rate of 5°C/min.
7. Taking a picture (2) of the sample after heat treatment.
8. Comparison of the two pictures.



a) b) c)

Figure 3 - High temperature resistant speckle pattern made of brown fused alumina and alumina adhesive: a) photo from temperature resistance test – without heat treatment; b) temperature resistance test – after heat treatment using thermal cycle: heating/cooling rate – 5°C/min, dwell temperature – 1200°C, dwell time – 1h; c) photo from ATHORNA device (disc shaped sample with a diameter of about 100 mm).

III b. First results: ATHORNA device and measurements

The objective of the ATHORNA device is to study material behavior after a sudden application of a thermal shock. A disk shaped sample with a diameter of 100 mm is positioned on a bench and heated by laser at the center of the top side. The aim of the equipment, currently under development, is to detect and localize crack formation, 3D displacement field and temperature distribution fields. This is done using 6 acoustic emission sensors, 2 visible light cameras and one infrared camera, respectively (see Figure 4). Materials behavior, such

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as resistance to crack initiation and its propagation will be studied thanks to the 2P-DIC method. The acoustic emission sensors will enable the localization of crack formation and, together with a stereo DIC setup, will give complementary information for analysis of the crack formations and propagation.

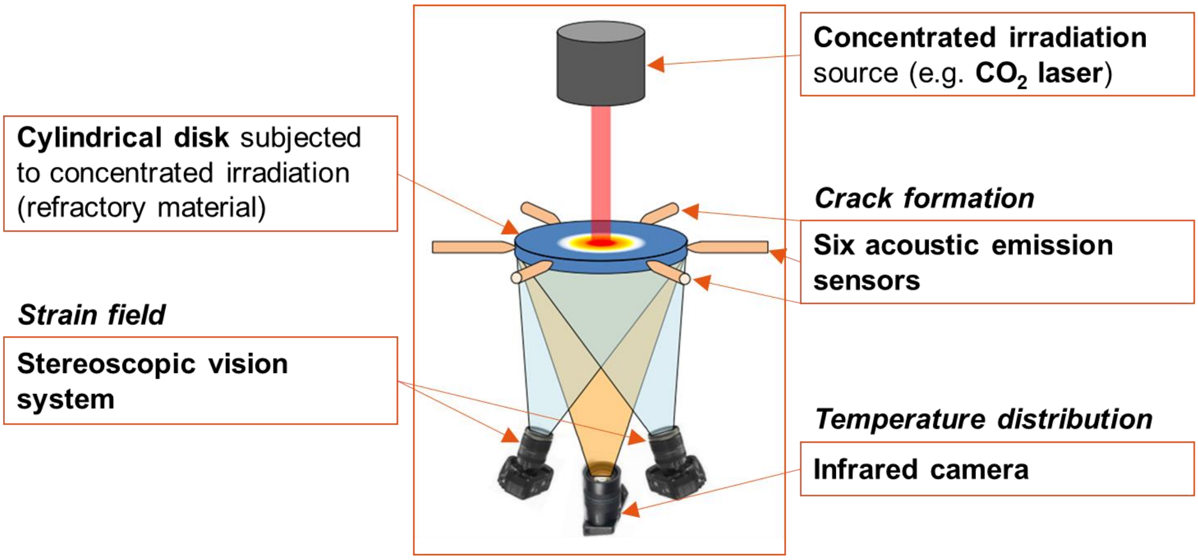

Figure 4 - Schema of the thermal shock bench (ATHORNA device).

The very first results of sample fracture detected by the 2P-DIC on the ATHORNA device are presented in Figure 5. The tested sample is an alumina spinel material with the refractory speckle pattern made of alumina cement and brown fused alumina grains. In image 5a we observe two main cracks located at the opposite sides of the sample (left and right). After cooling, the right one crosses the sample through the middle. While the second one reduces its opening below the threshold opening value.

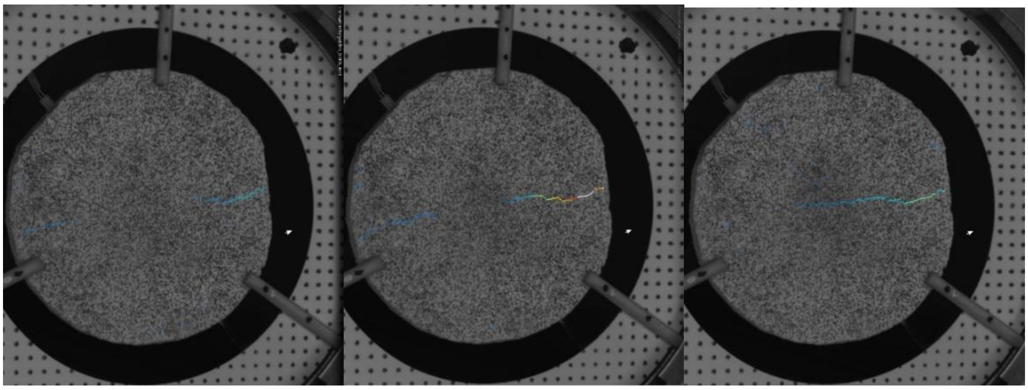

Figure 5 - Crack evolution on alumina-spinel material (from left to right) for Alumina-Spinel material: a,b) heating stage, c) cooling stage.

IV Conclusions and perspectives

This report summarizes the work done by the ATHOR community to improve the experimental and numerical methods used in the displacements and strains' fields determination applied to mechanical tests at high temperatures, and the further identification of material's parameters.

New software implementations were applied to improve the efficiency and accuracy of the identification procedures, and new techniques to obtain stable speckle patterns at high temperatures were also developed.
The I-DIC will now be applied to the identification of creep parameters of refractory materials at high temperature using Brazilian disc tests and 2P-DIC will be used for the tracking of cracks in refractories under thermal shock, using the ATHORNA device.

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