Final Report

Advanced THermomechanical multiscale mOdelling of Refractory linings

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Primary Authors	**Marc Huger, marc.huger@unilim.fr, UNILIM** **Glyn Derrick, glyn.derrick@unilim.fr, UNILIM**
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Project Coordinator	**Marc Huger, marc.huger@unilim.fr, UNILIM**
Document Contributors	**Dietmar Gruber, dietmar.gruber@unileoben.ac.at, MUL** **Alain Gasser, alain.gasser@univ-orleans.fr, UORL** **Joao Pereira, jpereira@civil.uminho.pt, UMINHO** **Glyn Derrick, glyn.derrick@unilim.fr, UNILIM** **Marc Huger, marc.huger@unilim.fr, UNILIM**

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1 Introduction

Refractories are heat-resistant materials used as inner linings of high temperature furnaces, reactors and processing units. Unique and advanced ceramic materials, refractories sustain complex combinations of thermomechanical stresses and chemical/physical wear generated by fluids and chemical agents used during high temperature fluid processing. As the only low-cost family of materials able to sustain operating conditions, at temperatures typically above 1000°C, refractories are widely used in energy intensive industries such as Iron and Steel production. Vessels commonly lined with refractories include the steel ladle which, due to the harsh environment, typically require the lining to be changed every 8 weeks. Extending the lifetime of refractories under such conditions is thus of importance both economically and environmentally.

ATHOR was a H2020 MSCA European Training Network combining seven academic and eight industrial partners across Europe. The project focussed on the training of 15 young researchers in multi engineering required fields for a better understanding of thermomechanical behaviour of refractory linings. The research was aimed at the design of more robust and reliable refractory linings, undertaken through the combination of advance numerical modelling with both small- and large-scale laboratory research (from the micro to the 10 meter scale). This will allow the development of innovative numerical simulations that will form the basis of future research as well as monitoring form a safe location. Ultimately, it represents a reduction of the refractory costs, an increase of the equipment's availability and an enhanced process control.

ATHOR ended on March 31st, 2022. All the tasks have been achieved, with the reorganisation and extension of several due to the unavoidable interruption of the project by the COVID-19 pandemic. In total, 16 milestones have been past, and 43 deliverables have been submitted.

This final report is an overview of the project, the achieved results and the socio-economic impact.

2 The ATHOR approach to refractories research

The European steel industry is a world leader in innovation and environmental sustainability. At the start of the ATHOR project, a turnover of around €170 billion was achieved, with the direct employment of 320,000 highly skilled people and an average of 170 million tonnes of steel produced per year. More than 500 steel production sites across 24 EU Member States provide direct and indirect employment to millions more European citizens. Closely integrated with Europe's manufacturing and construction industries, steel is the backbone for development, growth and employment in Europe. In particular, the European refractories industry historically grew simultaneously with the industrial revolution, Iron & Steelmaking (I&S) representing 60% of the refractory materials' market today [1].The EU is still one of the first regions in terms of development and production of high quality refractories and the second largest producer after China, with productions of 4.1 MT valued at € 3.9 billion and 29.5 MT valued at € 14.3 billion respectively. These materials are directly related to the competitiveness of European Steel companies and contribute to the development of major European economic sectors [2].

A constant engineering of refractories is needed to cope with new and more demanding requirements in the steel making process. The steel ladle forms the hearth of the steel making process and is a major refractory using facility accounting for about ¼ of all refractory consumption [3]. Steel ladle practices can significantly differ between regions and between steel plant according to different metallurgical processes, working conditions and refractory concepts. Dependent on its purpose and position in the steel ladle, the requirements for the refractory materials are various ranging from high thermal stability, to high erosion resistance, high corrosion resistance, penetration resistance, thermomechanical stability, impact resistance, flexibility and creep resistance [4]. The concerted work in the ATHOR program was therefore be focused on the steel ladle as model installation.

The main scientific goals of ATHOR were to adapt and develop the most advanced modelling strategies and experimental technologies to the refractories field such that reliable computations and measurements, in the temperature range of the applications of these materials, could be performed. ATHOR targets the development of high-end engineering technologies in the fields of material's science and numerical simulations to give a substantial contribution through the design of more robust and reliable refractory linings.

The main training objective was to prepare the 15 Early-Stage Researchers (ESRs) to be the next generation of highly employable scientists and engineers in the refractory and related sectors. With the evolution of industry towards digital twins and the use of advanced numerical simulations based on real world data, the ESRs need a basis in both numerical simulations and fundamental materials science. This was achieved through a series of Refractory Training Courses (RTCs) and online webinars, which were recorded and stored in a database such that future interested PhD students can benefit from the time and energy given by the ATHOR industrial and academic experts. The highly interconnected and international nature of this project meant that the ESRs were required to communicate extremely precisely with each other as well as the industrial and academic partners to determine the exact nature of the data required for the numerical simulations and the parameters used in the lab to generate this information. Communications skills were thus also developed as well as soft-skills important for the future employability of the ESRs such as leadership skills. The training was provided at both the local universities and via centrally organised activities.

The specific objectives of the ATHOR network were defined as:

- Deliver the best training to young researchers to reach a scientific level by addressing their scientific and transversal needs.
- Enable young researchers through their tailored training and regular mobility to "cross-pollinate" industry and academia;
- Give the young researchers the opportunity to be close to the industry and to face the actual engineering challenges;
- Support the development of new characterization devices and modelling methods to offer solutions to current S/T challenges;
- Create a multidisciplinary environment where industries and academia can share ideas and find solutions together.
- Explore the capability of advanced numerical modelling in order to design better materials and better refractory linings, consequently improving energy efficiency and thermomechanical properties of products.
- Assist the European refractory and steelmaking industries to identify technological improvements by providing high performance characterization and modelling tools which can comfort their competitiveness.

To guarantee a maximum economic impact when ATHOR methodologies were implemented in practice, the consortium decided to carry out research on different refractory materials, in well organised Work Packages. These Work Packages:

- involved materials with different chemical composition and microstructure so as to cover a wide diversity of end-user applications;
- involved different key scientific topics sufficiently critical and/or of economically importance;
- were sufficiently generic for the European market to cover the different current interests of the industrial partners.

2.1 The organisation of the scientific work program

The ATHOR project brought together 7 academic and 8 industrial partners to carry out cutting-edge research, using state-of-the-art technology, in highly specialized areas including materials science, structural engineering and numerical modelling. Research ranged from, the development of techniques to initiate, track and analyse small scale crack formation, to the construction and analysis of large-scale structures, such as a 3D pilot steel ladle, which were subjected to various heating and loading regimes.

The scientific work program consisted of 4 highly interconnected, scientific Work Packages, Figure 1.

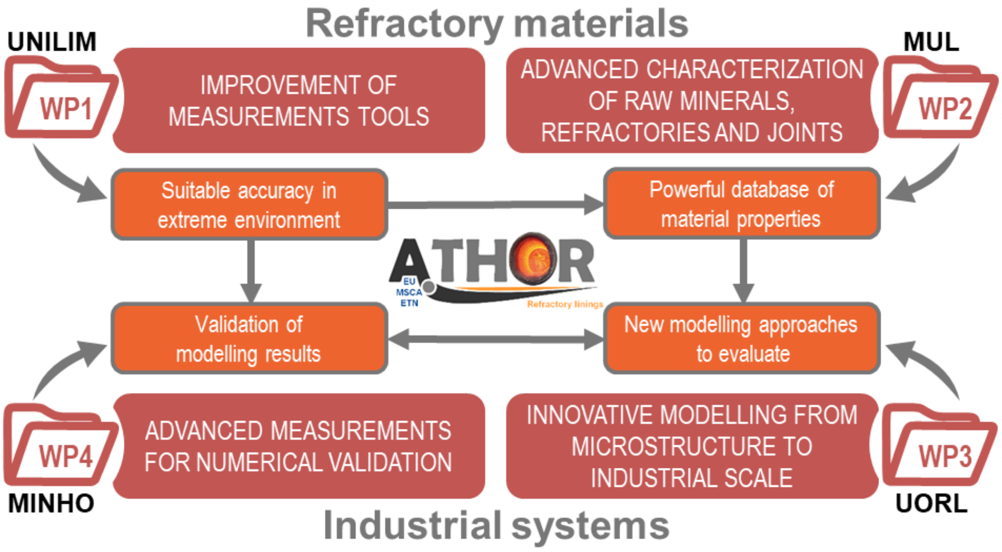


Figure 1 : The four highly interconnected, scientific Work Packages of the ATHOR project.

WP1 was dedicated to the improvement of measurements tools through the development of experimental methods (contactless optical techniques, high temperature strain gauges…). These tools were then employed in the collection of the mechanical data necessary to study refractory materials in service conditions (temperature up to 1500°C, large structures, industrial environment). Previously, these techniques were mainly used for different materials in a laboratory environment up to 500 °C and for small structures. The aim of WP1 was thus to improve these experimental optical methods in order to accurately quantify low level of strain fields and to make measurements possible in extreme conditions associated to the environment of refractory materials in service.

WP2 was dedicated to the characterization of selected materials at room temperature and elevated temperature in virgin or corroded state by conventional and specific experimental techniques. This WP has provided all the experimental results needed for characterisation and understanding of material behaviour. Furthermore, the input parameters for the finite element simulation have been determined for the modelling from micro to macro scale. WP2 took advantage of the ATHOR network to apply the most appropriate experimental device to each task. Where available, different methods within the ATHOR network were used to validate the experimental results and improve the testing procedures.

WP3 was dedicated to the production of dedicated modelling methods and numerical tools to optimize the design of industrial refractory linings. The different proposed approaches cover all the scales that impact the thermomechanical refractory lining behaviour: from the material scale (i.e. microstructure) to the industrial lining (structural computation). The multiscale approach was developed from micro to macro to establish the models and then, inversely, from macro to micro to optimize each scale from the final vessel requirements point of view.

WP4 was dedicate to Advanced measurements for numerical validation. A such, closely interacted with WP1 and WP3, the improved and newly developed methodologies for thermal and mechanical characterization of refractories within WP1 were used in WP4. Using a multiscale approach, measurements were performed to validate the advanced numerical models developed in WP3.

2.2 Improvement of measurements tools

Expertise developed by ATHOR partners, for example on Digital Image Correlation, mark tracking in 2D and stereovision configuration were applied to monitor deformation and temperature of the steel shell of the industrial ladle to validate the results of numerical modelling based on mesoscopic characterization. Moreover, a bi-axial high temperature testing device, for a 1m^2 masonry wall, available through ATHOR network, was improved thanks to additional instrumentation. A second laboratory pilot device, at around 2 m in diameter, was designed to reproduce the stress state induce by the cylindrical shape of industrial vessels. They were used to validate, at mesoscopic level, the results of numerical modelling based on new mathematical and numerical method taking into account mesoscopic materials and contact properties. All the different experimental technologies used for the characterization of individual materials, subsystems (e.g. bricks with or without mortar, castable-based linings, bed joints…) and the overall thermomechanical behaviour of selected industrial devices, were strongly enhanced through WP1. These included thermal and strain fields instrumentation as well as devices for thermo-physical properties characterisation, thermomechanical characterisation and validation of masonry and vessels.

2.3 Advanced characterization of raw materials, refractories and joints

The experimental results, of the refractory materials under investigation, that were required to characterise and understand the materials behaviour were obtained here. The microstructure and its evolution was described, thermal and mechanical characterisations were carried out, fundamental corrosion mechanisms were described as well as the mechanical characterisation of corroded samples and finally the behaviour of the joints was defined. Thermal expansion and thermal conductivity were tested by laser flash or hot wire method, Young's modulus by ultrasonic echography or by Resonance Frequency and Damping Analyser (RFDA), damage monitoring by acoustic emission, stress-strain and creep laws were determined for tension and compression and work of fracture by wedge splitting test.

2.4 Innovative modelling from microstructure to industrial scale

Different modelling techniques were used depending on the material being modelled. The Discrete Element Method (DEM) was used for the modelling of the microstructure and thermomechanical behaviour. When regarding the modelling of bricks and joints, the multi-surface plasticity theory and nonlinear homogenisation method were used and compared. For the full-scale industrial steel ladle, the simulation of a representative volume element was also compared with the multi-surface plasticity theory and nonlinear homogenisation method.

2.5 Advanced measurements for numerical validation

Refractory masonries were characterized experimentally, in order to obtain data for the validation of advanced analysis methods, as well as for subsequent simulation of industrial problems to allow industrial device optimization. The selected materials and structures were characterized under different conditions, and their thermal and mechanical properties investigated under complex limit conditions. A large and comprehensive experimental campaign was implemented, wallets were constructed and subjected to uniaxial and biaxial loading from room temperature to high temperature. A 3D pilot steel ladle was designed, constructed and tested. The 3D pilot scale and the full-scale models investigated have provided unique and valuable data for the calibration and validation of the advanced numerical macro-models developed in WP3.

3 Results

In this section the results from the ATHOR project are summarised. The results are discussed in greater detail in the Periodic reports, deliverables, publications and theses that are available on the ATHOR website.

3.1 WP1 - Improvement of measurements tools

The aim of WP1 was to improve experimental optical methods in order to accurately quantify low level of strain fields and to make measurements possible in extreme conditions associated to the environment of refractory materials in service. The strain fields coupled with temperature values obtained by a pertinent combination between classical thermocouples and infrared cameras have been used then to study the thermomechanical behaviour of various studied systems (at different scales, from micro up to pluri-meters industrial structures).

3.1.1 Task 1.1 - Thermal instrumentation

The lining of each steel ladle is composed of different materials with specific temperature dependent properties. The acquisition of the temperature data at different points through the steel ladle lining (taken here as an example of application) and the effect of this on the properties of the materials used, is therefore of paramount importance. Hence, the main goal of this task was to improve the methodologies (thermocouples, infrared camera, laser scanner) used for temperature measurements both at laboratory scale and within industrial applications. At laboratory scale, the ATHORNA device (Advanced measurements for in-situ THermo-mechanical monitoring of large sample uNder thermal gradient) has been developed in the framework of ATHOR project. In this device, the measurement of temperatures by an infrared camera at the bottom face of the disk-shape sample (10 cm in diameter) is presented in Figure 2. Figure 2 (a) and (c) represent the thermal image and the diametral temperature at the beginning of the test, respectively. Figure 2 (b) and (d) present the measurements during heating of the the top face of the sample by a CO_2 laser.

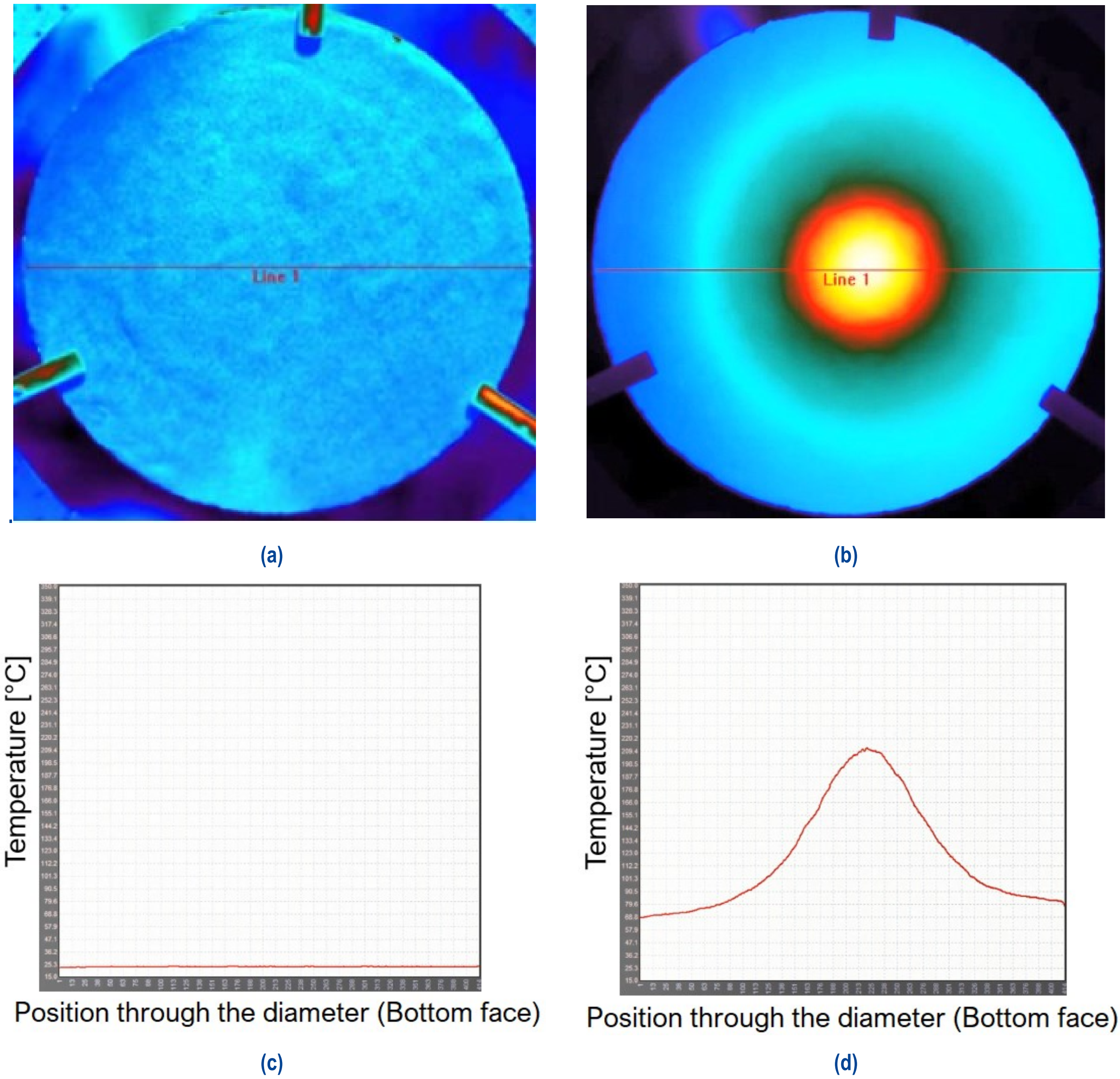


Figure 2: Temperature measurements using IR Cameras on ATHORNA device:
(a) IR Camera image - Beginning of the test, (b) IR Camera image - During laser application,
(c) Diametral temperature - Beginning of the test, (d) Diametral temperature - During laser application

3.1.2 Task 1.2 - Strain field instrumentation

Among the different techniques of strain measurements, the Digital Image Correlation technique offers the great advantage to produce full strain field measurement results which can be thus easily compared to results from Finite Element Method (F.E.M.). Nevertheless, high-temperature mechanical tests coupled with Digital Image Correlation (DIC) on ceramics which exhibit rather low level of strain require to overcome extreme experimental conditions that usually can reduce significantly measurement accuracy. Thermal resistance of speckle pattern, black body radiation and heat haze are in fact three main concerns, which should thus be taken into account while designing a high-temperature image acquisition setup for later DIC analisys. One of the main challenge of the ATHOR project was thus to perform high temperature mechanical tests (Brazilian test, Wedge Splitting test or ATHORNA), using optical techniques (DIC). In this case, high-accuracy optical measurement techniques required optimal and persistent speckle patterns. 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.

An example of sample fracture detected by the 2P-DIC on the ATHORNA device are presented in Figure 3. The tested sample is an alumina spinel material with the refractory speckle pattern made of alumina cement and brown fused alumina grains. In image 3a, 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.

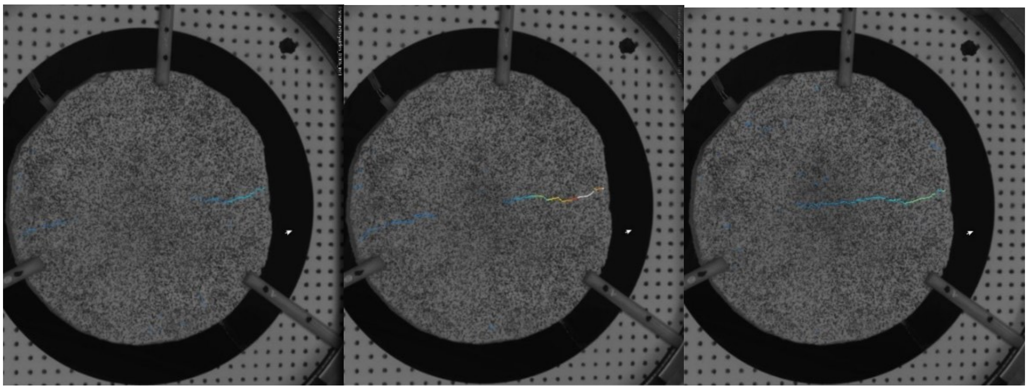


Figure 3: Crack evolution on alumina-spinel material (from left to right) for Alumina-Spinel material: a,b) heating stage, c) cooling stage.

3.1.3 Task 1.3 - Devices for thermo-physical properties characterization

This task was entirely dedicated to the development of devices for thermo-physical properties characterization of different refractory materials, such as insulating boards, fireclay bricks, microporous sheets, alumina spinel bricks and castables, magnesia carbon bricks, bauxite etc. The characterization included methods for the determination of thermal conductivity, thermal diffusivity, specific heat capacity and thermal expansion.

The used available devices were the following:

- Laser flash equipment for thermal diffusivity measurements at room and high temperature (IRCER-UNILIM);
- Hot disk equipment for thermal conductivity measurements at room temperature (IRCER-UNILIM);
- Hot disk equipment for thermal conductivity measurements at room temperature and medium temperature (ISISE-UC);
- Hot wire equipment for thermal conductivity measurements at high temperature (GHI-RWTH);
- Dilatometry equipment for thermal expansion measurements at high temperature (IRCER-UNILIM / GHI-RWTH);
- RUL/CIC apparatus for thermal expansion measurements at high temperature (GHI-RWTH).

Figure 4: Hot disk device for high temperature measurements.

As one example, the hot disk device allows simultaneous measurements of thermal conductivity, thermal diffusivity and specific heat capacity to be made. With a specific equipment (Figure 4) available at ISISE-UC, it has been possible to manage measurements up to 500°C.

3.1.4 Task 1.4 - Devices for thermomechanical characterization

In this task, specialized fracture mechanical tests have been developed. Uniaxial tensile tests have been carried out at UNLIM. Additionally, further tests with inhomogeneous stress states have been carried out. This includes a mini wedge splitting test for the observation of the fracture process in a microscope and the adaption of digital image correlation (DIC). This has been done by MUL. Furthermore, a Brazilian test has been combined with DIC by UNILIM and UORL. The different tests have allowed the characterisation of the stress/strain response for different stress states. The inverse identification of behaviour laws parameters has been managed thanks to two different up to date techniques: the FEM-U and i-DIC. These methods have been refined and validated for high temperature.

The ATHOR project was dedicated to the development of experimental and computational methods to characterize and model refractory materials. This has required very accurate thermomechanical material characterisation to determine material parameters to be used as input parameters in FEM simulations. It was thus very important to characterise many material properties at room temperature and at application temperatures as well, because the material behaviour usually varies significantly versus temperature. The characterisation has included methods for the determination of the Young's modulus and Poisson ratio, the fracture mechanical behaviour and the creep behaviour. Young's modulus and Poisson ratio define the linear elastic material behaviour. Due to the fact that in refractories material failure cannot be avoided, the characterisation of the fracture mechanical behaviour and the creep behaviour were also essential for the understanding of the material behaviour and for further improvement of refractories and linings.

3.1.5 Task 1.5 - Devices for validation on masonry and vessels

This task was dedicated to setup and develop different large scale experimental devices to be used for the thermomechanical characterization and monitoring of refractory masonry subsystems (masonry wallets and a 3D pilot vessel). This included tests on: a) compressive behaviour of an assembly of two bricks with or with mortar (both at ambient and at elevated temperature); b) uniaxial compression behaviour of masonry walls (both at ambient and elevated temperatures); c) bi-axial behaviour of masonry walls (elevated temperatures), as well as well as a 3D pilot (elevated temperatures) to mimic a simplified steel ladle (cylindrical assembly of an overall lining with reduced size in comparison to a real stell ladle).

Just as an example, compressive tests has been performed on refractory walls composed of high content alumina bricks with dry joints at elevated temperatures. High content alumina bricks with the dimensions of 250×124×76 mm have been used. The masonry wall was constituted of six bricks in length and fourteen bricks in height. Therefore, the final dimensions has been 1500 mm in length by 1 064 mm in height. These masonry wallets have been placed at the border of an electric furnace. It was thus possible to apply a pre-compressive load and then to progressively heat the furnace under a constant rate. The test setup is presented in Figure 5. On top of the masonry wallet, two large steel beams have been used to ensure the stiffness required to control the displacements of the hydraulic jacks used to apply the pre-compressive load. In parallel, different experimental devices (not presented the figure) have been successfully used to monitor, during the experiment, the in plane and the out of plane displacement in different point of the masonry.

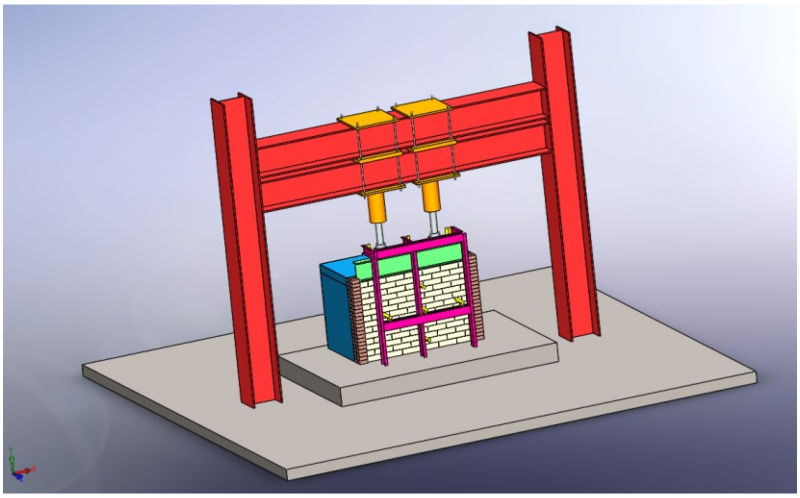


Figure 5: General schematic of the uniaxial compression tests for masonry wallets at high temperature.

3.2 WP2 - Advanced characterization of raw materials, refractories and joints

WP2 dealt with the characterization of selected materials at room temperature and elevated temperature in virgin or corroded states by conventional and specific experimental techniques. Thermal expansion and thermal conductivity were tested by laser flash or hot wire method, Young's modulus by ultrasonic echography or by Resonance Frequency and Damping Analyser (RFDA), damage monitoring by acoustic emission, stress-strain and creep laws were determined for tension and compression and work of fracture by wedge splitting test. Microscopical and mineralogical investigations were carried out to provide the basis for understanding. Furthermore, the behaviour of joints between bricks and the influence on the behaviour of walls was tested. Finally, this WP provided the experimental results needed for characterisation and understanding of material behaviour. Moreover, the input parameters for FE simulation were determined for the modelling from micro to macro scale. WP2 took advantage of the ATHOR network to apply the most appropriate experimental device to each task

3.2.1 Task 2.1 - Microstructure description and evolution

Task 2.1 focused on microstructure investigations (by XRD, HT-XRD, SEM, EDS, EBSD) of the studied materials in their different states (including corrosion). The aim here was to build a better understanding of the relationships existing between microstructure (and its evolution) and thermomechanical properties for investigated materials. Six different refractory material types (fired alumina spinel bricks, resin bonded alumina-carbon bricks, alumina-spinel castables, vermiculite bricks, insulating fireclay bricks and fused silica castables) have been examined according to their initial characteristics as well as their characteristics after heat treatment, corresponding to their application in the steel ladle. The phase changes, the microstructure evolution and some basic physical properties, have been studied for the six different materials. Globally, these results provide an initial insight into the microstructural description of the different materials which were investigated (from thermal, mechanical, thermomechanical and corrosion points of view) throughout the ATHOR project. Investigations in the initial state provided the starting point for investigations after some heat treatment, corresponding to the localisation of these materials within the steel ladle, provides a vision of their potential evolution related to temperature (without any applied loads or corrosion). A detailed description of all the results of task 2.1 is given in deliverable 2.1.

3.2.2 Task 2.2 - Fundamental corrosion mechanisms

The degradation process of refractory materials, and the related parameters, play an important role. The chemical and mechanical wear consist of complex reactions and processes called "corrosion". The corrosive mediums, gas, molten metals, molten glasses, or molten salts (slag) result in the loss of thickness and mass; therefore, refractory materials are replaced repeatedly. The objective of this deliverable is to identify fundamental corrosion mechanisms of refractory linings used in the steel ladle. Therefore, selected refractories (Al_2O_3-Sp and MgO-C) were corroded by specified slags. The samples were investigated by (a) chemical analysis (XRF) (b) phase composition (XRD) and (c) SEM/EDS for chemical analysis in micro areas. The basic corrosion mechanisms of the two refractory materials, alumina-spinel (Al_2O_3-Sp) and magnesia-carbon (MgO-C), will be detailed. These materials have been selected due to their use in the steel ladle: the alumina spinel bricks are used for the side wall and the MgO-C are used for the slag line. Corrosion of alumina-spinel and magnesia-carbon bricks is discussed. Details about the corrosion of alumina-spinel and magnesia carbon bricks were investigated.

The corrosion tests provided insights into the corrosion mechanisms of alumina spinel and magnesia carbon bricks in contact with calcium aluminate slags.

In the case of alumina spinel bricks, the samples were infiltrated and the Ca^{2+} ions reacted with the alumina grains to form CA, CA_2 and CA_6. In addition, in the samples corroded using the $10SiO_2$ slag, more gehlenite formed compared to the sample corroded using the standard slag. Gehlenite is a low melting point phase, when present in the sample, it promotes the further infiltration of the slag. Hence, the samples corroded using the $10SiO_2$ were corroded to a greater extent than the samples corroded using the standard slag.

In the case of magnesia carbon brick, the carbon matrix was oxidized due to the carbothermic reaction between MgO and C. This leads in turn to the formation of Mg gas. The Mg gas further reacted with easily reducible elements, FeO and MnO, from the slag to form a MgO layer at the surface of the brick. With the 10MnO slag, the amount of reducible elements was higher, hence the formation of the magnesia layer increased. Additionally to the oxidation of carbon, the magnesia grains were dissolved by the slag. Due to the higher concentration to saturation of MgO in the 10SiO2 slag, the extent of corrosion of the sample treated using the $10SiO_2$ slag was higher than with the two other slags. A detailed description of investigations of the corrosion mechanisms is given in deliverable 2.2.

3.2.3 Task 2.3 - Thermal characterisation

Thermal properties are important input parameters that drive thermomechanical behaviour of refractory linings in application. Specific heat, thermal conductivity and thermal expansion are thus three thermal properties essential for thermomechanical modelling in WP3. The present results are dedicated to the description of these properties as a specific output of Task 2.3. The values of these properties in-fact vary with both temperature and the evolution of the microstructure. In other words, the behaviour under in-service conditions and at room temperature is not necessarily the same and, in addition, room temperature values could be affected by heating cycles. Since refractory materials work at very high temperatures, it is fundamental to study the evolution of the thermal properties with temperature.

Measurements obtained were reviewed on the studied materials, for; i) coefficient of thermal expansion (CTE), ii) heat capacity (Cp) and iii) thermal conductivity (λ). The measurement of CTE in the temperature range 20 °C to 1500 °C is a rather standard procedure, the access to accurate values of Cp and λ, however, is still a challenging topic. In task 1.3 different aspects regarding experimental devices which can be used for the measurement of CTE, Cp and λ are detailed. Results of this task include (a) the best practice for thermal properties measurement on refractory materials and (b) characterization of Cp and λ on selected commercial refractories and model materials for numerical modelling. The results are summarized in detail in deliverable 2.3.

The specific heat is the amount of heat energy necessary to increase the temperature of a substance by one degree Celsius. It is not always easy to have an accurate value in the temperature range which is of industrial interest. However, it was demonstrated that the rule of mixtures works very well for this purpose. The comparison between the Differential Scanning Calorimetry and the rule of mixtures, in fact, exhibits a difference of 1 % in the temperature range 200 - 900 °C.

The thermal conductivity is the ability of the material to propagate the heat, both in the solid and in the pore phases. It can be measured with different techniques, which lead to some variations in the obtained values. However, taking into account some parameters such as heat losses and humidity, the maximum discrepancy could be reduced within 10 % when the same heat flow direction through the material is considered. Heterogeneity and anisotropy are other factors that should be taken into account to better interpret the results.

The thermal expansion is the tendency of a material to change its volume or its dimensions due to a change of temperature. Normally, the coefficient of linear expansion is estimated by dilatometry measurements. However, the RUL/CIC apparatus may give comparable results and, additionally, could be advantageous for materials containing bigger aggregates (such as refractories) or when the influence of additional factors (load) should be investigated.

3.2.4 Task 2.4 - Mechanical characterisation

Due to the high temperature application of refractories, in-situ measurements and observation of their thermomechanical behaviour during application is difficult. In this regard, thermomechanical simulation of refractory lining behaviour is of great help. Determination of material mechanical properties is a prerequisite for modelling the behaviour of these materials as the properties are needed in FE-simulation. Several experimental approaches are thus defined here to identify different parameters for material models.

Different material models have been applied for mechanical behaviour simulation of refractory materials, which were explained in detail in deliverable 3.2. In service, refractories experience thermal gradients and repetitive thermal shocks, which may generate significant stresses resulting in tensile failure of the material. On the other hand, due to thermal expansion, high compressive stresses are common in refractory materials, which may cause crushing of the material. The concrete damaged plasticity model, of the commercial software Abaqus, contains the tensile cracking model formulation as well as the compressive crushing model. The constitutive model uses the concept of isotropic damage evolution to represent the inelastic behaviour of the material. Additionally, to represent the shear failure, the Drucker-Prager model is commonly applied. This model is mostly applied for materials in which the cohesion depends on the hydrostatic pressure. Finally, creep at high temperatures is very important in refractory linings because of its influence on the stress magnitude in the lining and in the steel shell. The classical Norton-Bailey creep model and Ducker-Prager creep model are available in Abaqus for modelling of the creep behaviour of refractories.

The results obtained in task 2.4 comprise of the mechanical properties of the studied materials. This includes elastic material properties like Young's modulus and Poisson's ratio as well as thermomechanical properties like thermal expansion, fracture mechanical data (for example the tensile strength) and the specific fracture energy, creep parameters and shear failure parameters such as cohesion and friction angle. The parameters were obtained either by direct measurement or by inverse evaluation of experimental results. The measurement devices were explained thoroughly in deliverable 1.3 and 1.4. A detailed overview and interpretation of the results is given in deliverable 2.4.

3.2.5 Task 2.5 - Mechanical characterization of corroded samples

Corrosion is one of the most common wear mechanisms of refractories. Corrosive attacks lead to chemical and microstructural changes. Hot corrosion compromises chemical and/or physical interactions. Thus, the process is complex and not yet fully understood. Currently, corrosion is investigated post-mortem by means of X-ray diffraction or scanning electron microscopy. These methods have the drawback that some information is lost on cooling. In-situ measurements, however, take measurements within the process. As an outcome of WP2, which is dedicated to the "Advanced characterisation of raw materials, refractories and joints", the results of this task review the mechanical characterization of corroded samples. The relationship between the corroded microstructure and thermomechanical behaviour of refractory linings will be analysed.

With the in-situ and ex-situ measurements two different approaches have been chosen. Both studies have been performed individually with specifically produced materials.

The steel ladle, a metallurgical vessel which is the common thread for all the research carried out within the ATHOR project, is widely used in secondary metallurgy. One of the key issues faced by the steel ladle during its lifetime, is corrosion. In this task in-situ and ex-situ approaches have been used to investigate the effects of corrosion on laboratory prepared and industrially supplied, refractory castables. In-situ measurements have been used, at room and elevated temperatures, to investigate the impact of different, synthesised, slag compositions on several, laboratory prepared, alumina-based refractory materials. These in-situ investigations (along with a novel testing of coupled thermal shock with corrosion) are detailed in Deliverable 2.5. The development of an ex-situ testing procedure for the Brazilian test after corrosion of the sample as well as some initial results is also described.

It is notable that within the task 2.5, significant testing novelties were implemented that enabled the thorough comprehension of the corrosion/corrosion-thermal shock behaviour of refractories. Moreover, the combination of these novel testing methods with the most common and simpler analytical techniques (e.g. SEM) provided an overall investigation of the corrosion and corrosion-thermal shock phenomena.

The in-situ RFDA monitoring of samples offers a direct evaluation of the corrosion phenomena with the aid of the newly designed hollow space samples. Moreover, RFDA offers indirect evaluation of the corrosion resistance of a material, as it is possible to correlate the final properties relevant to corrosion/corrosion-thermal shock resistance (e.g., Young's Modulus) with their evolution upon first firing. Therefore, we can design improved corrosion/corrosion-thermal shock resistance materials either by tuning the components of the refractory material or, by optimising the drying and sintering processes via RFDA monitoring.

The innovative coupled corrosion-thermal shock methodology developed and presented in this task, combined with microstructural and mineralogical analysis, enriched the knowledge regarding the effect of important components (e.g., sodium-MA spinel) on corrosion and thermal shock resistance. The new corrosion-thermal shock method can be further optimised with the aid of DIC method so as to monitor the cracking initiation/evolution with and without the presence of corrosive medium.

Another important testing innovation achieved within the task 2.5 framework, was the ex-situ measurement by Brazilian test that was explored for the first time on corroded refractory materials. The study showed that there are some criteria that must be taken into account for preparing an appropriate refractory sample for thermomechanical investigations as well as thermal and mechanical boundaries which could affect the thermomechanical test performance in laboratory condition. The findings showed that it is easier to prepare laboratory samples compare with post-mortem samples since the condition and parameters could be controlled in laboratory condition, whereas post-mortem bricks are full of cracks and steel inclusions.

The findings of ex-situ measurements showed that the slag impregnation must be representative within the microstructure. Therefore, it is important to design the corrosion test in laboratory conditions with respect to the computational thermodynamic simulation and analytical laboratory methods. According to the achieved results, the digital image correlation (DIC) technique, can be used to monitor crack initiation and propagation path as well as validating the Brazilian test results at high temperature.

The microstructural analysis, for all testing methodologies presented in this report, unveiled the corrosion mechanisms for each specific pair of corrosive medium-refractory that was investigated. This enabled the correlation of mechanisms with different material resistance to degradation. Moreover, both the in-situ and ex-situ approaches within the task 2.5 framework offered new tools towards the improvement of testing methodologies in order to understand the corrosion phenomena on refractory materials and their impact on the thermo-mechanical behaviour of refractories. A detailed review of the results in found in the deliverable 2.5.

3.2.6 Task 2.6 - Characterization of joints behaviour

In this task experimental data regarding the mechanical behaviour of joints as a specific component of the refractory lining within the steel ladle is summarized. The data were used in the numerical simulations within WP3. The results presented in this document come from experimental campaigns developed at University of Coimbra, TataSteel and University of Orléans. The experimental campaigns at University of Coimbra and TataSteel focuses on dry joint (without mortar) behaviour. Here, the objective is to study the behaviour of the working lining of the model steel ladle, which is composed of dry-stacked alumina spinel bricks. The experimental campaigns at University of Orleans focused on joints with mortar.

Joints without mortar (for alumina-spinel bricks corresponding to the working lining) were studied for both normal and shear behaviour. A classical joint closure test was performed in two stacked bricks and compared to joint closure measurements on a refractory wallet measured by DIC. It was observed that the heterogeneity on the joint closure was higher in the masonry wallet. In the latter case, the geometric imperfections in a full course influence the contact conditions of the bricks, resulting in joints with higher heterogeneity and compressibility. Classical joint closure tests were also performed at different temperatures (600 ºC, 800 ºC, 1000 ºC and 1200 ºC), and it was found that the contact pressure required to close the joint decreased with the increase in temperature, this behaviour is explained by the reduction in the elastic modulus of the material at higher temperature. A novel test setup, for evaluating the friction coefficient between bricks in dry joints, was developed and presented. The equipment was used to evaluate the friction coefficient of the alumina spinel bricks at ambient temperature, 300 ºC, 600 ºC and 900 ºC. A reduction of the friction coefficient was found when subjected to high temperatures.

Concerning joints with mortar (for bricks corresponding to the safety lining), slanted shear tests at room and high temperature were performed to characterize the shear failure criteria of the refractory brick-mortar interface. Two refractory brick materials and one ready to use mortar have been tested. The two refractory materials are bauxite and chamotte, while air hardening mortar was used to glue the refractory bricks together to produce the samples. From the first obtained results, it has been shown that similar values of the internal friction angle are obtained at room and high temperature. However, higher values of cohesion are obtained at high temperature. A detailed review of the results can be found in deliverable 2.6.

3.3 WP3 - Innovative modelling from microstructure to industrial scale

The objectives of this WP were to produce dedicated modelling methods and numerical tools to optimize the design of industrial refractory linings. The different proposed approaches cover all the scales that impact the thermomechanical refractory lining behaviour: from the material scale (i.e. microstructure) to the industrial lining (structural computation). The multiscale approach is developed from micro to macro to establish the models and then, in the reverse way, from macro to micro to optimize each scale from the final vessel requirements point of view.

3.3.1 Task 3.1 - Microstructure modelling by Discrete Element Method

The objective of this task was to investigate the role played by the microstructure (i.e. grain size distribution, grain properties and interface characteristics) in the macroscopic thermomechanical behaviour using a Discrete Element Method based model of the refractories microstructure. This model is used to define the optimal microstructure to enhance the macroscopic characteristics identified as critical from the point of view of the vessel lifespan. The developments are implemented through open-source software (GranOO) to enhance their disseminations. However, by reviewing other potential DEM contact models, the recently developed Flat Joint Model (FJM) showed greater potential for the goals of this study. This FJM is implemented within a software called Particle Flow Code (PFC) from ITASCA company.

Even if PFC with FJM is well-adapted to account for multiple crack propagation within a quasi-brittle material, unfortunately up to now, this model is not able to account for mechanical interactions and temperature variation simultaneously. To overcome this current drawback, a randomisation of local fracture criteria within the virtual sample using a Weibull distribution was proposed.

Based on a previous study of the fracture behaviour of pure MgO and Magnesia Hercynite (MH15), the Two Parts-DIC technique was used to detect and visualise the crack propagations during Wedge Splitting Test (WST). In the present study, numerical modelling has been managed in order to mimic this WST experiment. Two virtual samples were thus constructed: one with a uniform value of 6 MPa for the local tensile strength to exhibit the Pure MgO brittle behaviour, and the second, using a Weibull distribution of the local tensile strength, to exhibit the MH15 quasi-brittle behaviour. Figure 6 shows these two samples at the end of the simulations.

At first glance, it shows that in the simulation of the pure MgO, the sample was broken with a rather straight fracture path, without any crack branching, which is underlining a highly brittle material. On the other hand, in the simulation of the MH15, the sample exhibits a deviated crack path and crack branching, which is due to the quasi-brittle behaviour of the material. Overall, these results are showing acceptable qualitative fracture behaviour in comparison to the real materials, which confirms the applicability of the proposed DEM numerical approach in WST. In fact, by using this approach, the numerical models could dissipate more energy through the initiation of microcracks and crack branching, which results in a higher fracture energy for the quasi-brittle samples.

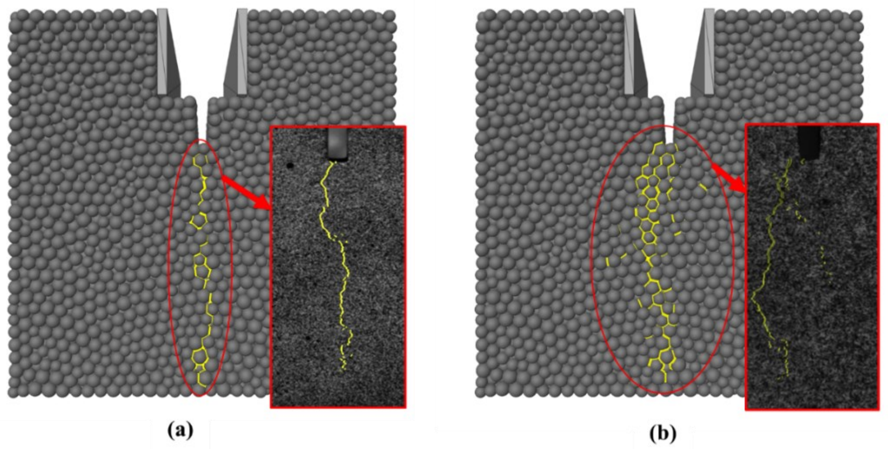


Figure 6: Comparison of the crack paths from DEM simulation vs DIC outputs (in the red frame): (a) case of pure MgO with a rather straight crack path, (b) case of MH15 with a deviated crack path.

3.3.2 Task 3.2 - Modelling of thermomechanical behaviour

The data resulting from tests performed in Tasks 2.3 and 2.4 allow the identification of the parameters of the constitutive equations, already implemented in commercial F.E. codes for a benchmark. The comparison of the results from all the numerical simulations has allowed the identification of the most relevant model. This model is enhanced to take into account the creep of refractory in stage I and II (asymmetric creep). Indeed, it has been shown that, for an alumina-spinel material used in the working lining of steel ladles, it is important to consider the effects of asymmetric primary and secondary creep. This feature is not currently available in commercial finite element analysis software such as Abaqus, therefore is addressed in the models proposed in this work. Furthermore, a model combining tension failure and creep was proposed.

In order to identify the material properties of the model, Brazilian tests were performed at a temperature of 1300°C. The vertical displacements field for two different values of load are shown in Figure 7.

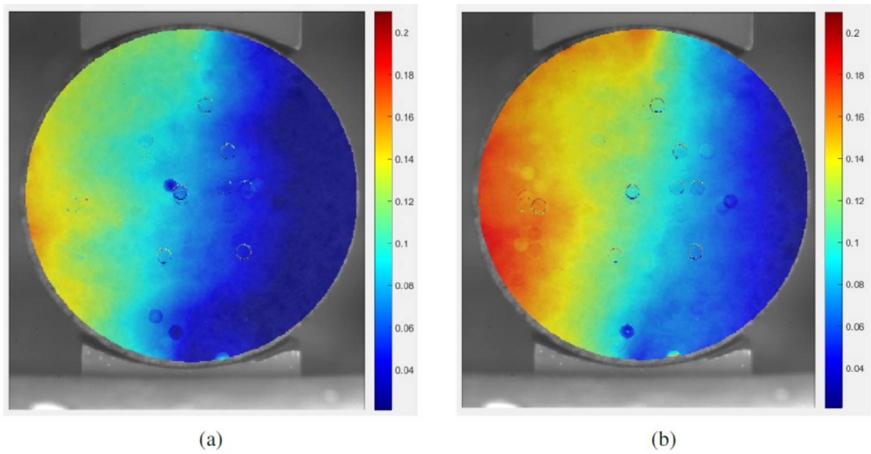


Figure 7: Brazilian tests: Vertical displacement (in mm) at 1300°C (for two different values of load) - DIC measurements.

Figure 8 shows the results of the numerical simulations using the asymmetric creep model. It is possible to observe that the displacement maps of Figure 7 and Figure 8 have a good equivalence, despite the experimental errors and the simplicity of the identification procedure used.

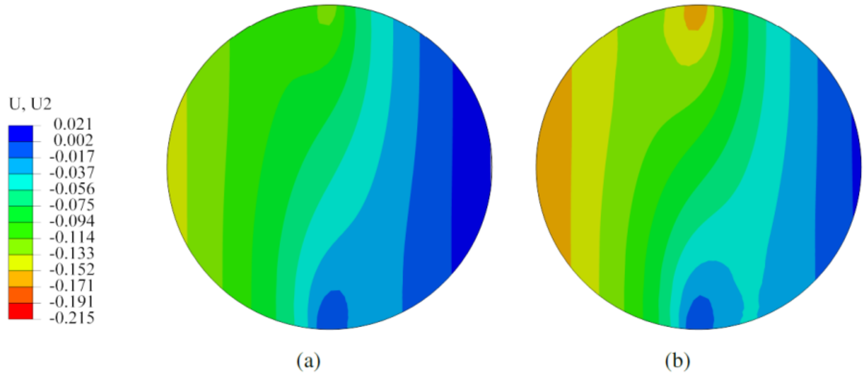


Figure 8: Brazilian tests: Vertical displacement at 1300°C (for two different values of load) - Simulation.

During service, refractory materials are exposed to thermal gradients often coupled with a reactive chemical environment that might induce corrosion. To consider the effect of all these complex service conditions on the refractory material, the application of multi-physics modelling via numerical studies becomes necessary. Therefore, the most suitable constitutive equations identified from task 3.2 must be enhanced to take into account the effects of the corrosion on the mechanical behaviour in the framework of the thermodynamic irreversible processes.

Corrosion of refractories can be defined as refractory wear by change of mechanical properties and loss of thickness and mass from the exposed face of the refractory as a consequence of chemical attack. This chemical attack, a process in which the refractory and the corrosive liquid react, approaches a chemical equilibrium in the zone of contact between the refractory and the liquid. Refractory corrosion involves a combination of different mechanisms, such as dissolution and invasive penetration, where diffusion, grain boundary, and stress corrosion may all be present. Oxidation-reduction reactions, where absorption, desorption, and mass transport phenomena also come into play, generally under pressure and temperature gradients.

The local values of the temperature field are used in corrosion studies to select suitable material for linings that are in danger of corrosion. Furthermore, it is essential to understand the corrosion to approach the appropriate material's composition for each zone (slag line, working line, etc.). Even though the corrosion problem is strongly connected to the thermomechanical design of the structure, coupling thermomechanical and thermochemistry modelling is not the norm. Usually, in the best-case scenario, these two points are studied sequentially and sometimes, iteratively. As shown in Figure 8, coupling the thermomechanical behaviour to the corrosion and phase change is possible thanks to broader information regarding the corrosion, refractories and developments in the computational capacities for simulations.

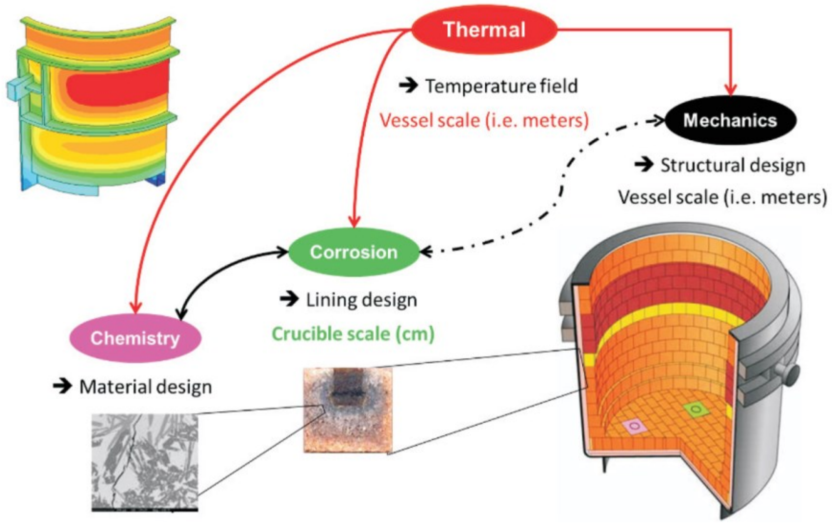


Figure 9: Refractory lining design; a multiscale and multi-physics problem.

A theoretical basis on the multi-physics modelling applicable to corrosion, phase changes, and mechanical behaviour was proposed. The coupling between thermochemistry and thermomechanics was described within the framework of the Thermodynamics of Irreversible Processes. The results of mechanical tests at high temperature on corroded samples (Task 2.5) will permit to identify the parameters of this new model and to validate it.

3.3.3 Task 3.4 - Non-linear modelling at subsystem scale

This task is related to the modelling of the nonlinear behaviour of the bricks and the joints using a micro-modelling approach (UMINHO) and a macro-modelling approach (UORL). Elasto-viscoplasticity and joints (with or without mortar) are taken into account to generate local behaviour models that will feed the structure computational approach developed by UMINHO and the mesoscopic homogenization approach developed by UORL. These models were implemented in the commercial F.E.M. software (Abaqus and DIANA) through free user subroutines.

The working linings of the steel ladles studied in this work are built using refractory masonry with dry joints (see Figure 10). The working lining is built up from thousands of tapered refractory bricks. The bricks are periodically arranged in running bond texture. Dry joints are separating the bricks from each other. These joints are resulting from the shape, dimensional tolerances and surface unevenness of the bricks.

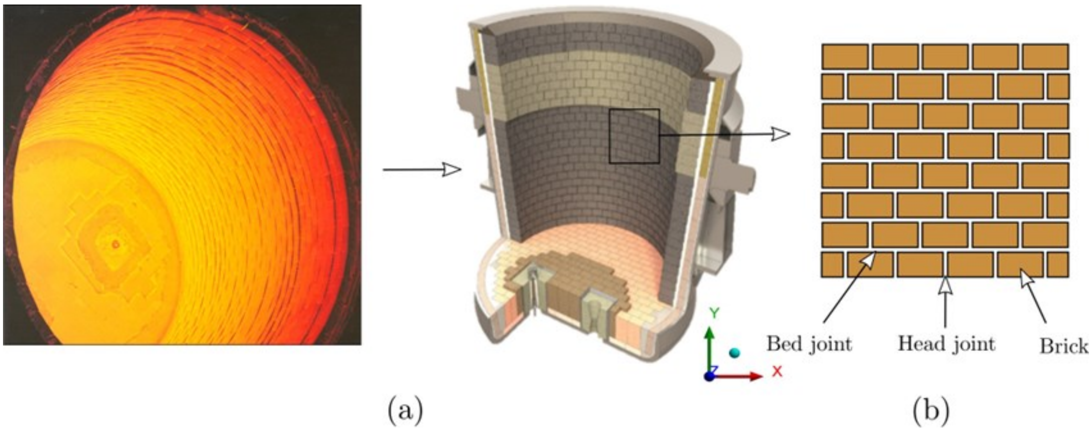


Figure 10: (a) Steel ladle lined with refractory masonry with dry joints, and, (b) Schematic of masonry wall with dry joints showing the bricks and the gaps between them.

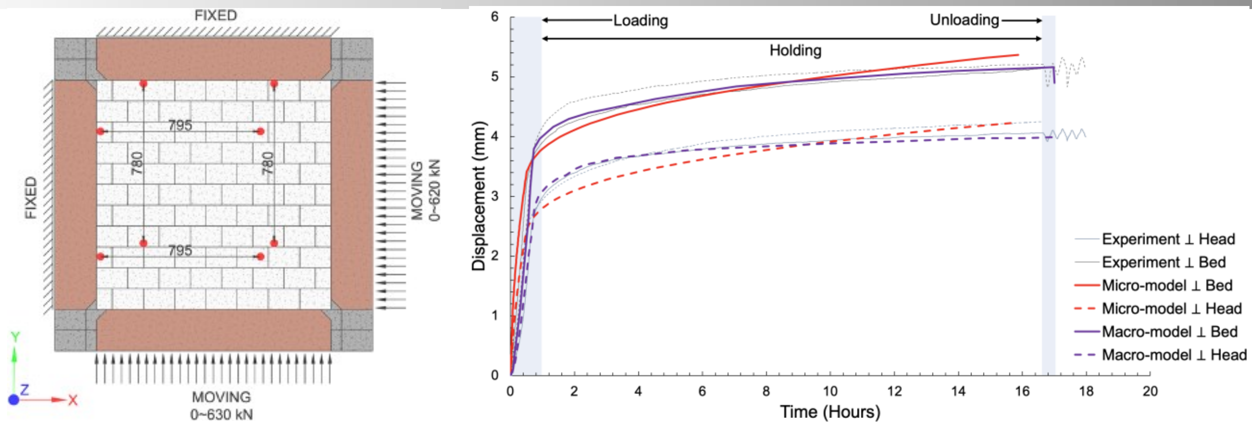
In the micro-modelling approach, the units and mortar are modelled as continuum elements and the interfaces between bricks and mortar (or between bricks and bricks) are represented by discontinuous elements. Even though accurate results are obtained using the micro-modelling technique, there is a significant drawback, the large computational resources required to run the analysis. Nevertheless, this approach provides detailed results on the behaviour of the bricks and joints. The numerical analyses for this approach were performed using the finite element software DIANA FEA and Abaqus.

The macro-modelling considers the masonry as a homogeneous structure made of a material that has a behaviour equivalent to the behaviour of the masonry. This behaviour, orthotropic, elastic-viscoplastic, temperature dependent, is determined using a nonlinear homogenization method. For the purpose of considering the impact of joints closure and reopening on the homogenized elastic-viscoplastic behaviour of masonry structures, four possible joint patterns are predefined. Each pattern is based on the state of both bed and head joints (i.e., open or closed) and represents different periodic masonry structures with different equivalent behaviours. The advantage of this macro-modelling is that it can be used for the computation of large industrial vessels containing refractory masonries, without a large computation cost and convergence problems.

3.3.4 Task 3.5 - Structure modelling at 3D laboratory pilot scale

The thermomechanical tests performed at meso-scale on masonry panels (uniaxial tests at UMINHO, biaxial tests at RHI-Magnesita and laboratory pilot test at TATASTEEL), developed in Task 1.5 and used to obtain experimental data at large scale in Task 4.5, were modelled with the two methods developed in Task 3.4 (micro-modelling and macro-modelling). A comparison of the results from these two methods with the experimental results at meso-scale was done. Different loads and boundary conditions were considered to check the ability of each approach to cover a large field of loading cases.

For example, the results of a biaxial test at high temperature are presented. Two refractory walls were tested at 1500°C. After the load application step, the applied forces were held constant for 16 hours. During the load application, holding and unloading, experimentally measured forces were applied to the two moving rigid plates (Figure 11a).

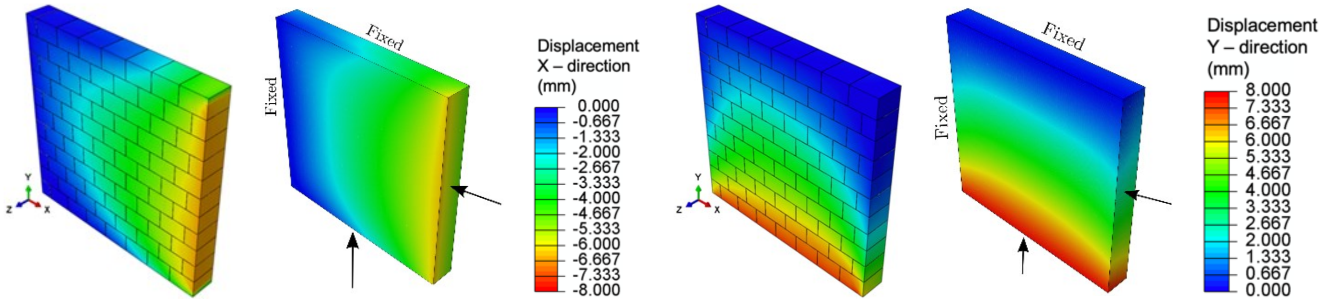


(a) (b)

Figure 11: Test series S10.HT.CBI - Biaxial creep behaviour: a) boundary conditions; b) experimental and numerical time variations of the displacements during loading, holding and unloading stages.

Comparisons between the experimental and numerical displacement – time diagrams in the directions normal to bed and head joints during loading, holding and unloading steps are shown in Figure 11b. Good agreement between the experimental and numerical results can be observed. The maximum displacement in the direction normal to bed joints is higher as compared to that in the direction normal to head joints due to the difference between the number of bed and head joints in the wall. However, the observed displacements are lower compared to the uniaxial creep tests performed at high temperatures. This reduction is due to biaxial force application on the refractory masonry wall which generates lower stresses in the materials and higher friction forces with the loading beams. During the holding time, the increase in the displacements in both directions were almost the same indicating full closure of bed and head joints during the loading step and therefore isotropic in-plane viscoplastic behaviour.

The displacement fields, by the end of the load holding step, in the direction normal to bed and head joints obtained using the micro and macro modelling approaches are shown in Figure 12. Displacement distributions between the models are similar.



(a) (b) (c) (d)

Figure 12: Test series S10.HT.CBI - biaxial loading and unloading: Displacement along the X direction (mm): a) Micro-model; b) Macro-model. Displacement along the Y direction (mm): c) Micro-model; d) Macro-model.

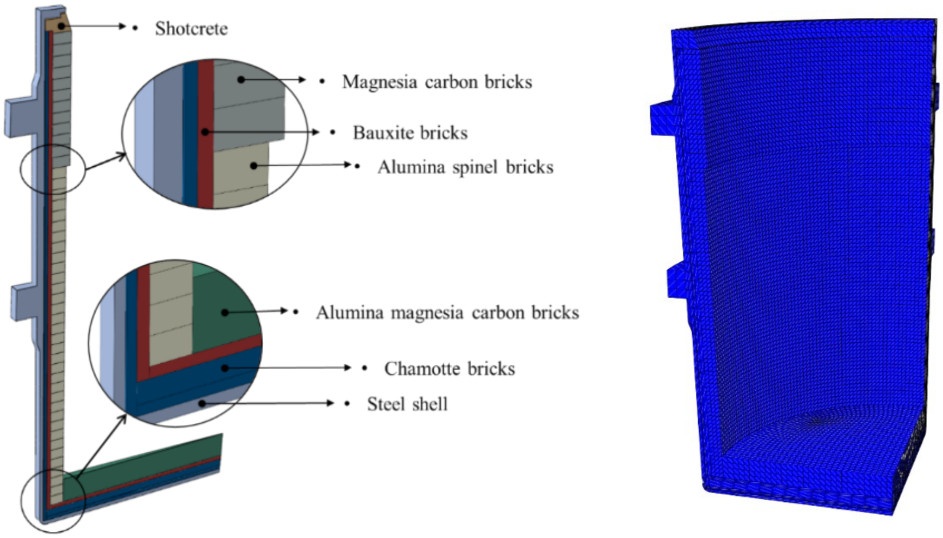
From all the results obtained in Task 3.5, it can be observed that both modelling approaches provide a reliable prediction of masonry response at the ambient and higher temperatures, validated with the experimental results. Moreover, the response obtained by these approaches are in good agreement with each other.

3.3.5 Task 3.6 - Structure modelling at industrial scale

The aim of this task was to model a real industrial structure (steel ladle) using two different approaches: a unit-cell modelling developed by MUL, and a macro-modelling developed and validated in Task 3.4 by UORL.

The unit-cell modelling technique employs a representative volume element that contains all constitutive data, such as all units and joints, and is a repeatable piece of the lining. Therefore, this method requires the knowledge of each constituent. In the case of complex structures, the modelling will be rather time consuming. Simulating a representative volume element instead of the complete structure results in a smaller model size and therefore lower computational cost. Here, a 3D representative volume element model of the steel ladle (shown in Figure 8a) was built using the commercial software Abaqus according to its industrial design and dimensions. The model has a cone shape and because of the existing symmetry conditions, its thickness was considered as half of an alumina spinel brick, which is used in the working lining barrel zone. Details about the material of each layer are given in Figure 13.

In the second approach (macro-modelling), the refractory masonries are replaced by a homogeneous equivalent material whose effective mechanical properties depend on the state of bed and head joints (open or closed). The real nonlinear behaviour of this equivalent material was determined using a non-linear homogenization method. The developed 3D solution domain of the steel ladle is shown in Figure 8b. Only one quarter of the ladle was modelled to reduce the computational size of the problem. This approach is valid since the studied ladle is symmetric.



(a) (b)

Figure 13: Steel ladle: (a) 3D unit-cell model, (b) macro-model.

Boundary conditions, loadings and their evolution versus time are defined in agreement with measurements on industrial vessels from Task 4.5. The two approaches were used two model the ladle behaviour during preheating and five cycles (each cycle with pouring, processing, casting and idle time) with two brick behaviours, elastic and elastic-viscoplastic.

In Figure 14, radial, circumferential, and axial stresses at the hot face and the middle of the working lining were shown during all process time. The first observation was that a biaxial compressive stress state existed in the centre of brick, i.e., radial stress was negligible compared to axial and circumferential stress. The stress magnitudes during the preheating are around the same magnitude for elastic and creep models. In addition, the creep decreased the stress on the hot face in the beginning of 1st cycle, and in the middle of the brick, during the 2nd cycle. The creep occurred after the temperature increased sufficiently and it started in the middle of the brick with a delay from the hot face. Therefore, the equivalent creep strain was higher on the hot face than the other parts of the brick. The differences between the resulting thermomechanical stresses obtained by both modelling approaches are caused by the major difference between the solution domains of both approaches and the related boundary conditions applied to the sides of the ladle.

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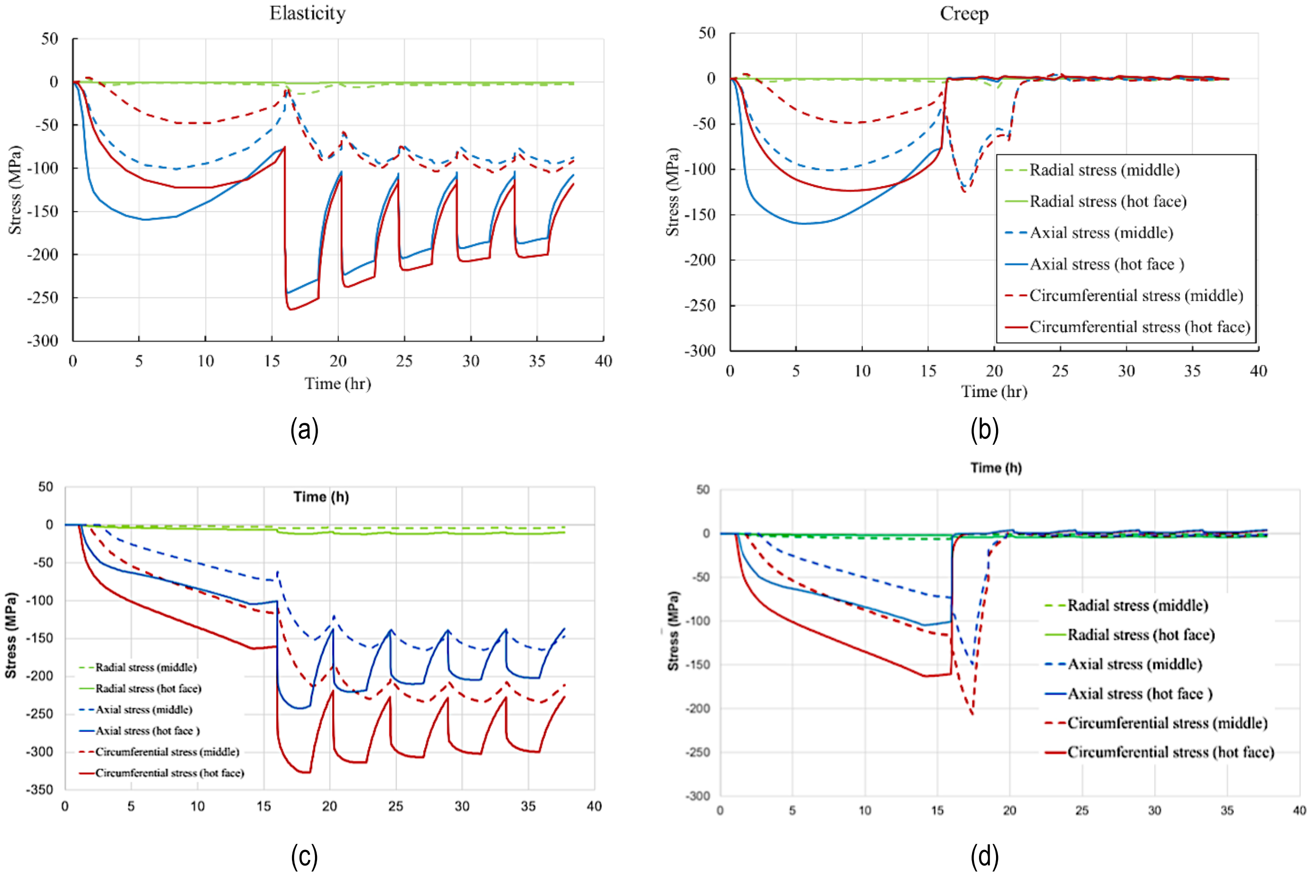


Figure 14: Stress at the hot face and middle of the working lining: (a and b) 3D unit-cell model, (c and d) macro-model.

Parametric studies investigating the impacts of dry joints thickness, dry joints behaviour and constitutive material models used for the different linings of the ladle were carried out using the steel ladle macro-model. They have shown the importance of the thickness and behaviour of dry joints (the increase of joint thickness decreases the stresses in the different layers).

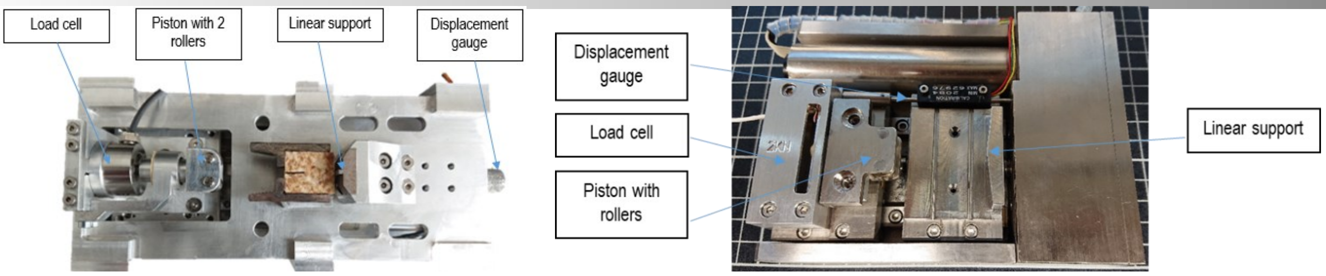
In conclusion of this WP3, several tools were built to model refractory structures at different scales: DEM at micro-scale (scale of the material microstructure), micro-modelling (bricks and mortar) and macro-modelling (homogeneous material equivalent to a masonry) at meso-scale (scale of a masonry panel), and 3D-unit cell and macro-modelling at macro-scale (scale of an industrial vessel). All these tools were validated by comparison to experimental measurements. They will bring an accurate help for the industrial vessel design to improve their lifespan.

3.4 WP4 - Advanced measurements for numerical validation

The main objective of this WP was to characterize refractory masonry experimentally, in order to obtain data for the validation of advanced analysis methods, as well as for subsequent simulation of industrial problems to allow industrial device optimization. The selected materials and structures were characterized under different conditions, and their thermal and mechanical properties were investigated under complex limit conditions. A large and comprehensive experimental campaign was collected. It ranges from the characterization of the microstructure of materials to the full-scale industrial steel ladle. This database is paramount for the validation of the numerical models developed within WP3, as well for subsequent developments in the field of refractories.

3.4.1 Task 4.1 - Quantification of microstructure evolutions under load

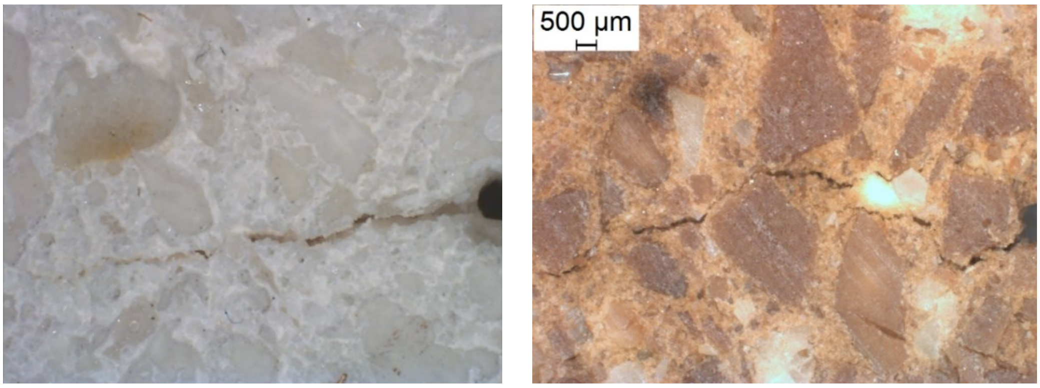
The wedge splitting test (WST) is today a standard experimental method that allows to quantify specific properties, such as fracture energy and brittleness number, of refractory materials. To aid the interpretation of the WST results with microstructural aspects of the investigated materials, it would be very useful to monitor crack propagation through the microstructure at the surface of the samples during loading. However, the sample size used for classical WST does not allow direct observation with advanced techniques, such as SEM (Scanning Electron Microscopy). To overcome this issue, two different devices were developed at UNILIM and MUL. Both developed devices (Figure 15) were developed in the intent of performing wedge splitting tests (WST) on small samples.



a) b)

Figure 15: Wedge splitting test device:
a) Miniaturized device at MUL; b) Mini device at UNILIM (based on DEBEN Microtest device).

Several different materials were characterized within the scope of this task: a) Alumina spinel refractory bricks; b) Magnesia spinel refractory bricks; and c) Six different castables. In the case of alumina spinel and magnesia spinel bricks, all results showed crack propagation within the matrix and at the interface between grains and matrix (Figure 16), but rarely in the grains. Some grains rotations and some crack branching have been also observed. These events could explain the sudden load increases that appear sometime within the second part of the load/displacement curves. Regarding the results obtained on refractory castables, as a general observation, it was possible to establish a relation between fracture behaviour and its thermal treatment at 500°C. A detailed description of all the results of task 4.1 is given in deliverable 4.1. All the experimental results obtained within this task are valuable towards the development of Discrete Element Models (DEM).

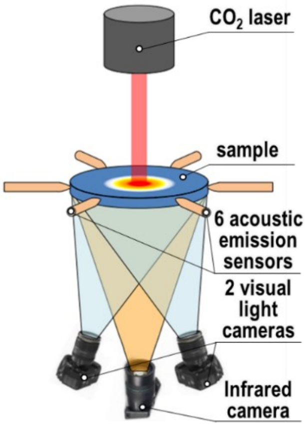


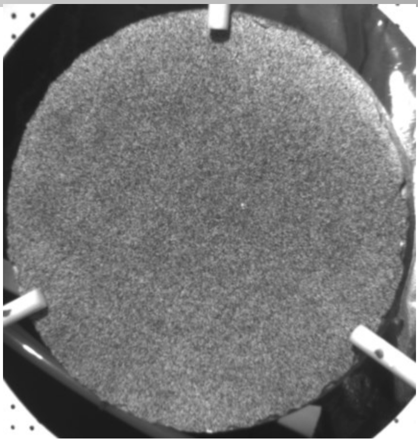
a) b)

Figure 16: Example of a crack propagation: a) within alumina spinel sample; b) within magnesia spinel sample.

3.4.2 Task 4.2 - Quantification of the thermal gradient effect on materials

A dedicated device – ATHORNA (Advanced measurements for in-situ Thermomechanical monitORing of large sample uNder thermal gradient) has been developed to monitor the behaviour of a cylindrical disk subjected to a concentrated heat flux under cyclic thermal gradient. A CO_2 laser allows the modulation of heating power and adjustment of cycles applied at the top face of the sample. An infrared camera allows the measurement of the temperature fields at the bottom face of the sample. A stereo-vision cameras and the digital image correlation allow the measurement of displacements and strains (Figure 17a). Application of DIC with two subsets located on both sides of a crack is useful to determine crack opening (detection of crack initiation time) and to compare this crack initiation time with the time for which predicted numerical stress can reach material strength (Figure 17b).





a) b)

Figure 17: ATHONA device: a) overall schematic of the device; b) speckle pattern for DIC.

To demonstrate the potential of the developed device, an alumina spinel specimen was subjected to twelve thermal cycles and in parallel, a numerical model was developed to simulate the performed test. It was demonstrated that the numerical model was able to properly replicate the observed behaviour. It was possible to confirm that the are two thermal gradients in the sample: *i*) through the thickness of the sample and *ii*) between the centre and the borders. It was also possible to identify a good correlation between the local strength of the material evaluated by the numerical model and the observation of crack propagation obtained by DIC (Figure 18). A detailed description of all the results of task 4.2 is given in deliverable 4.2.

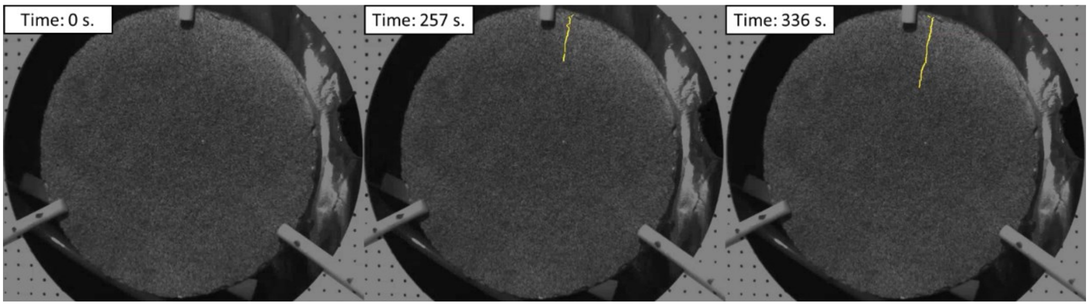
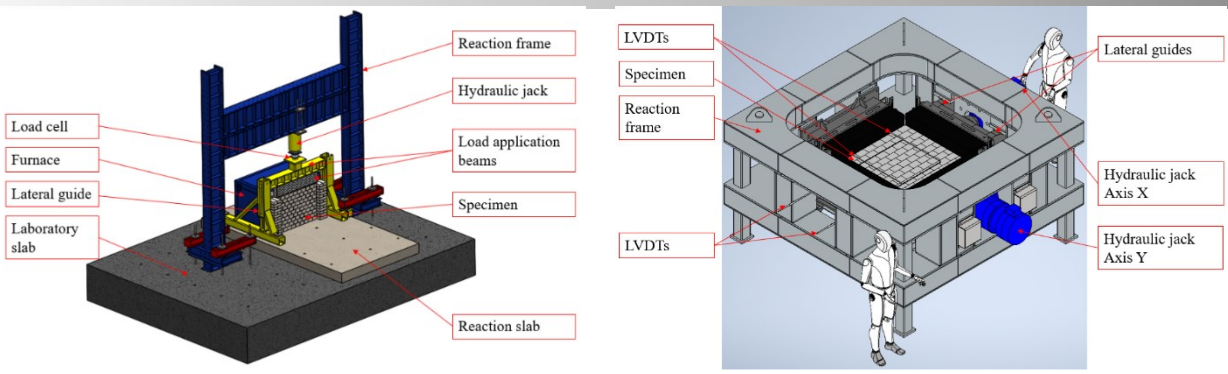


Figure 18: Example of crack propagation on alumina spinel samples (obtained with 2P-DIC).

3.4.3 Task 4.3 - Quantification of the structure effect at subsystem scale

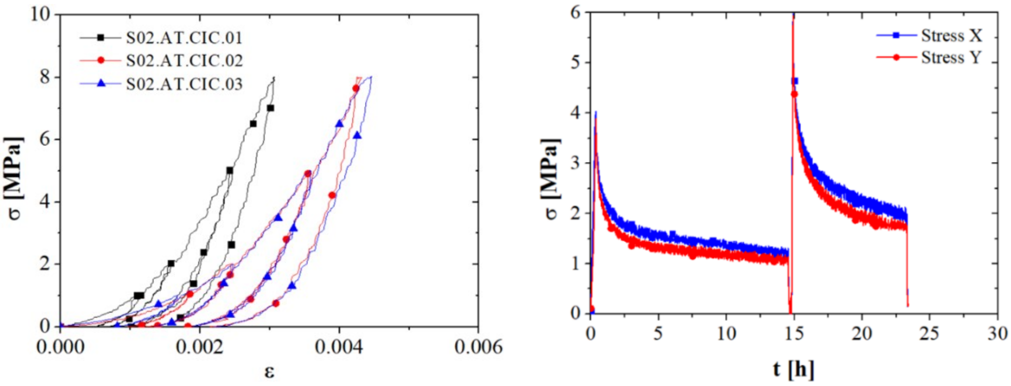
This task gathered the data obtained in the different experimental campaigns on the thermomechanical behaviour of refractory masonry at the subsystem scale. It includes the experimental campaigns performed in masonry wallets at UMINHO and RHI-Magnesita. The objective was to characterize refractory masonry (composite) constituted of alumina spinel brick used in the working lining of the steel ladle. Masonry wallets were tested under different conditions: (a) uniaxial compression at UMINHO (Figure 19a); (b) bi-axial compression at RHI-Magnesita (Figure 19b).

Twenty-six walls composed of alumina spinel bricks were tested within the scope of this task, at both ambient temperature and high temperatures (up to 1500°C). The objective was to gather a large database on the thermomechanical behaviour of masonry walls under different loading conditions, including different support conditions (Figure 20). The idea being that the numerical models developed within WP3 could be validated against a robust experimental database. A detailed description of all the results of task 4.3 is given in deliverable 4.3.



a) b)

Figure 19: Masonry wallets test setup: a) uniaxial tests at UMINHO; b) biaxial tests at RHI-Magnesita.



a) b)

Figure 20: Example of thermomechanical results on masonry walls:
a) uniaxial cyclic test at ambient temperature; b) biaxial relaxation test at 1500°C.

3.4.4 Task 4.4 - Quantification of the structure effect at laboratory pilot scale

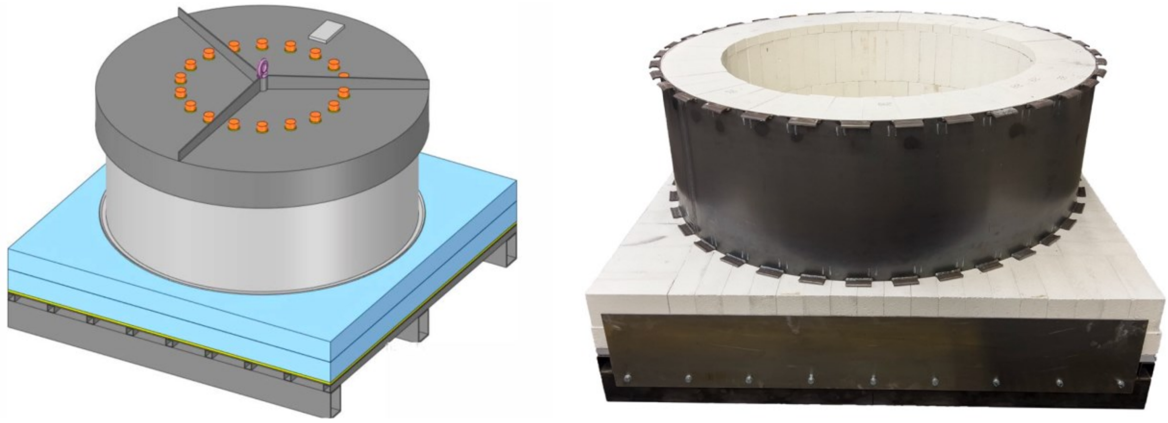
With the objective of gathering data for the validation of advanced numerical models, experimental characterisation of the refractory masonry that undergoes similar thermomechanical loadings as an industrial ladle was deemed necessary. Within this task, an unprecedented experimental installation was envisaged – a 3D pilot ladle. This represented a novel approach towards designing an experimental setup for large-scale refractory masonry. It required original test configuration, from designing the geometry, insulation, heating requirement, measurement devices, and installation.

This 3D pilot model is a scaled representation of a ladle (Figure 21), which includes all the linings as an industrial ladle and is expected to be tested under the thermal loads of similar magnitude of an industrial ladle without the presence of molten steel which will also allow to characterise and monitor the inner surface of the linings mechanically. This installation was developed at the Ceramics Research Centre (CRC) of Tata Steel in Ijmuiden, Netherland.

The objective of the campaign is to investigate the performance of refractory linings, in a configuration representing large-scale industrial installations. It will be possible to characterise the behaviour of refractory linings and steel shell from the experimental data while isolating the effect of ladle bottom and molten steel. This characterisation will ultimately provide insights into the thermomechanical behaviour of refractories in a cylindrical shape, such as joint behaviour, the interaction between the linings, creep, plasticity, and damage of the materials.

It should be noted that the objective was to present, within the time frame of ATHOR, all the experimental results obtained with such experimental campaign. However, due to the COVID pandemic, the work within this task was delayed to a point where the experimental results are not available within the timeframe of ATHOR (March 2022). It should be also noted that extra funding was procured to fund the ESR responsible for the development of this work (at UMinho, for 18 extra months). The Partner Organization involved in the development of such application (TataSteel) has also shown great interest in continuing pursuing its successful development. Although the experimental results are not yet available, they will become available after the timeframe of ATHOR. A detailed description of all the work performed towards the development of this installation is given in deliverable 4.4.

These results will then be used to calibrate advanced nonlinear micro and macro numerical models. This calibration process will give essential data regarding the influence of different material and geometric parameters on the global behaviour of the pilot ladle. Identifying these critical parameters will validate the numerical models with the industrial steel ladle and assist in optimising the material utilisation of different refractory linings.



a) b)

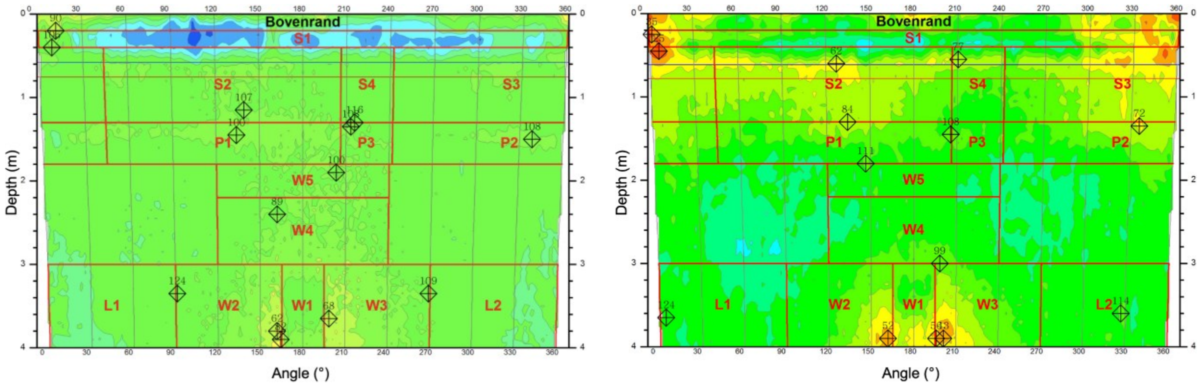
Figure 21: 3D pilot ladle: a) schematic of the installation; b) current state of the installation.

3.4.5 Task 4.5 - In-situ measurements on industrial steel ladle

In-situ measurements of an in-service steel ladle is a very challenging task considering the constant movement, restricted access, and safety regulations of the ladle. However, it is equally important to gather the data of thermal fields and displacement fields while the ladle is in service because it assists in calibration and validation of various constitutive models.

Thermal measurements obtained with thermocouples showed the thermal behaviour of the different linings during various stages of the steel ladle utilisation, such as, preheating, before, during and after the loading of the molten steel. Observation of the temperature at the steel shell, showed that the temperature remains relatively constant, after the preheating. This constant distribution of temperature shows the effectiveness of using an additional layer of the microporous insulation board. Moreover, observing the trend in the temperature changes between the load cycles, can also help identifying certain key thermal parameters, such as heat transfer coefficient and thermal conductivity.

Obtaining continuous displacement field measurements is very complex. Laser scanning offers an advantageous and innovative way of gathering the wear data periodically (Figure 22). The data obtained with this method still can be very useful to validate numerical models by comparing the wearing and viscoplasticity data between the numerical outcome and observed values. A detailed description of all the results of task 4.5 is given in deliverable 4.5.



a) b)

Figure 22: Example of working lining thickness determined with laser scans: a) after half life cycle; b) after full life cycle.

4 Dissemination and exploitation

As part of the ATHOR training program, the 15 ESRs have had the opportunity to disseminate the results of their cutting-edge research through workshops, summer schools, international journals and international conferences across the globe. The ATHOR ESRs have participated in conferences in Japan, China, America and Europe and have published their work in Rank A journals such as the Journal of the European Ceramic Society, Ceramics International and the International Journal of Mechanical Sciences. These published results are openly available via the ATHOR website and on the Zenodo platform, the list of published articles is presented below:

VITIELLO Diana, NAIT-ALI Benoit, TESSIER-DOYEN Nicolas, TONNESEN Thorsten,
LAÍM Luís, REBOUILLAT Lionel, SMITH David,
Thermal conductivity of insulating refractory materials: Comparison of steady-state and transient measurement methods,
Open Ceramics 2021, 6, 100118.
https://doi.org/10.1016/j.oceram.2021.100118.

KACZMAREK Robert, DUPRÉ Jean-Christophe, DOUMALIN Pascal, POP Octavian, TEIXEIRA Lucas, HUGER Marc,
High-temperature digital image correlation techniques for full-field strain and crack length measurement on ceramics at 1200°C: Optimization of speckle pattern and uncertainty assessment,
Optics and Lasers in Engineering 2021, 146, 106716.
https://doi.org/10.5281/zenodo.6556728.
https://doi.org/10.1016/j.optlaseng.2021.106716.

ASADI Farid, ANDRÉ Damien, EMAM Sacha, DOUMALIN Pascal, HUGER Marc,
Numerical modelling of the quasi-brittle behaviour of refractory ceramics by considering microcracks effect,
Journal of the European Ceramic Society 2022, 42(3), 1149-1161.
https://doi.org/10.5281/zenodo.5877203
https://doi.org/10.1016/j.jeurceramsoc.2021.11.016

REYNAERT Camille, SNIEZEK Edyta, SZCZERBA Jacek,
Corrosion tests for refractory materials intended for the steel industry – a review,
Ceramics-Silikáty 2020, 64 (3), 278 - 288.
https://doi.org/10.13168/cs.2020.0017.

KIELIBA Ilona, DOMINIK Ireneusz, LALIK Krzysztof, TONNESEN Thorsten, SZCZERBA Jacek, TELLE Reiner,
Self-Excited Acoustical System Frequency Monitoring for Refractory Concrete under Uniaxial Compression,
Energies 2021, 14, 2222.
https://doi.org/10.3390/en14082222

TADAION V., ANDREEV K., ZHU Q., WANG W., YIN Y., TONNESEN T.,
Thermal and mechanical cyclic tests and fracture mechanics parameters as indicators of thermal shock resistance – case study on silica refractories
Journal of the European Ceramic Society 2019, 39(4), 1650-1659.
https://doi.org/10.1016/j.jeurceramsoc.2018.12.062.

TEIXEIRA Lucas, GILLIBERT Jean, SAYET Thomas, BLOND Eric,
A creep model with different properties under tension and compression - Applications to refractory materials,
International Journal of Mechanical Sciences 2021, 212, 106810.
https://doi.org/10.1016/j.ijmecsci.2021.106275.

TEIXEIRA Lucas, SAMADI Soheil, GILLIBERT Jean, JIN Shengli, SAYET Thomas, GRUBER Dietmar, Blond Eric,
Experimental investigation of the tension and compression creep behavior of alumina-spinel refractories at high temperatures,
Ceramics 2020, 3, 372-383.
https://doi.org/10.3390/ceramics3030033.

ALI Mahmoud, SAYET Thomas, GASSER Alain, BLOND Eric,
Computational Homogenization of Elastic-Viscoplastic Refractory Masonry with Dry Joints,
International Journal of Mechanical Sciences 2021, 196, 106275.
https://doi.org/10.1016/j.ijmecsci.2021.106275.

ALI Mahmoud, SAYET Thomas, GASSER Alain, BLOND Eric,
Transient thermo-mechanical analysis of steel ladle refractory linings using mechanical homogenization approach,
Ceramics 2020, 3, 171-189.
https://doi.org/10.3390/ceramics3020016.

OLIVEIRA Rafael, RODRIGUES João, PEREIRA João, LOURENÇO Paulo, LOPES Rúben,
Experimental and numerical analysis on the structural fire behaviour of three-cell hollowed concrete masonry walls,
Engineering Structures 2021, 228, 111439.
https://hdl.handle.net/1822/69519
https://doi.org/10.1016/j.engstruct.2020.111439

OLIVEIRA Rafael, RODRIGUES João, PEREIRA João, LOURENÇO Paulo, MARSCHALL Ulrich,
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https://hdl.handle.net/1822/72527
https://doi.org/10.1016/j.engstruct.2021.112361.

OLIVEIRA Rafael, RODRIGUES João, PEREIRA João, LOURENÇO Paulo, MARSCHALL Ulrich,
Normal and tangential behaviour of dry joints in refractory masonry,
Engineering Structures 2021, 243, 112600.
https://hdl.handle.net/1822/73394
https://doi.org/10.1016/j.engstruct.2021.112600.

DARBAN Sina, REYNAERT Camille, LUDWIG Maciej, PROROK Ryszard, JASTRZĘBSKA Ilona, SZCZERBA Jacek,
Corrosion of alumina-spinel refractory by secondary metallurgical slag using coating corrosion test,
Materials 2022, 15(10), 3425.
https://doi.org/10.3390/ma15103425.

SAMADI Soheil, JIN Shengli, GRUBER Dietmar, HARMUTH Harald, SCHACHNER Stefan,
Statistical study of compressive creep parameters of an alumina spinel refractory,
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https://doi.org/10.1016/j.ceramint.2020.02.267.

SAMADI Soheil, JIN Shengli, HARMUTH Harald,
Combined damaged elasticity and creep modeling of ceramics with wedge splitting tests,
Ceramics International 2021, 47(18), 25846-25853.
https://doi.org/10.1016/j.ceramint.2021.05.315.

SAMADI Soheil, JIN Shengli, GRUBER Dietmar, HARMUTH Harald,
A comparison of two damage models for inverse identification of mode Ⅰ fracture parameters:
Case study of a refractory ceramic,
International Journal of Mechanical Sciences 2021, 197, 106345.
https://doi.org/10.1016/j.ijmecsci.2021.106345.

SAMADI Soheil, JIN Shengli, GRUBER Dietmar, HARMUTH Harald,
Thermomechanical finite element modeling of steel ladle containing alumina spinel refractory lining,
Finite Elements in Analysis and Design 2022, 206,103762.
https://doi.org/10.1016/j.finel.2022.103762.

This list of publications will continue to be added to as the ESRs finalise their PhDs which is standard for this type of project. However, while the PhDs have come to an end, the work that has been produced during ATHOR will continue to be exploited. For example, measurement tools that have been developed and improved, such as the ATHORNA device, bi-axial press and 3D pilot plant, will continue to be used by the industrial partners to generate data for future projects. A database of thermomechanical behaviour of large-scale specimens has also been developed for different testing configurations. The advanced characterisation of raw materials, refractories and joints has led to a plethora of information being made available to partners to use as a baseline against which future products can be compared. This work also demonstrated how microstructures can improve resistance against thermomechanical loads by, for example, inducing a dedicated microcrack formation. This phenomenon has the potential to be exploited in other refractory applications such-as in the aluminum, foundry, cement and petrochemical industries. Several numerical models have been developed over the course of ATHOR to determine material parameters and simulate the thermomechanical behaviour of refractory materials. These models are now being employed in both academic and industrial partners in various ongoing projects.

5 Conclusions and socio-economic impact of the project

ATHOR was built on an international network of academic and industrial institutes (Federation for International Refractory research and Education - FIRE) focused on the development of the refractory industry. This network aims to promote the refractories industry and provide training to young researchers. This solid basis for the ATHOR project has led to a free-flowing exchange between the partners from the very beginning of the project and this strong foundation has enabled the consortium to adjust to the changes to the initial planning due to the COVID-19 pandemic. This has limited the effect the pandemic has had on the project and resulted in high impact achievements on several levels.

5.1 Direct impact of ATHOR on the ESRs

The employability of the ATHOR ESRs has been greatly impacted through participation in this project. They have received training on cutting-edge research projects using state-of-the-art technology, and on transferable skills. Simultaneously, the ESRs have developed an international network and have gained experience working in an interdisciplinary and intercultural environment. This was achieved, for example, by the completion of at least one secondment, per ESR, with a project partner. Currently 11 out of 15 ESRs are employed as postdocs, engineers or R&D professionals with the four remaining ESRs writing their theses.

The communication skills of the ESRs have also benefitted from ATHOR in several ways. The ESRs have all presented work at international conferences and "ATHOR industrial feedback events" to scientific audiences, improving their presentation skills and increasing their networks at the same time. On top of this, communication with the general public was achieved through making personal videos for the website, as well as contributing to a short video, drawmylife and a short documentary produced by the BBC. ESRs were also face-to-face with the general public in events such-as the European researchers night and during the presentation of the ATHOR travelling exhibition "CeramiK".

The "CeramiK" exhibition is aimed at 11-18 year old students and combines technical concepts, presented in plain language (English and the corresponding translation depending on the location), visually impressive imagery and hands-on experiments and games. The ESRs were all implicated to some extent in the production of the text, images and suggestions of experiments that could capture the imagination of younger students.

All of these activities have enhanced different aspects of the ESRs communication skills while at the same time increasing the visibility of the project on both the scientific stage and to the general public.

Over the course of the project, eight ESRs were also chosen by their peers to represent the ESRs on the ATHOR Supervisory board. This provided ESRs with first-hand experience on how an international, interdisciplinary and intercultural project is managed as well as actively participating in the decisions and actions that were addressed.

5.2 The impact of ATHOR at a local level

The refractories industry is known within the industry as the "hidden industry". This is because, while refractories are fundamental to the production of materials at high temperature (steel, cement, glass etc.) the general public are unaware of their existence. The lack of visibility has resulted in difficulties recruiting talented research engineers who are required to modernise a business that is relied on by industries across Europe. As such, 15 ATHOR ESRs have been trained with the skills needed to directly respond to this need for high quality engineers in the short term and the visibility of the industry amongst future R&D engineers has been increased to respond to this need over the long term.

This new group of highly skilled, young professionals will induce a new movement in the industry towards new technologies and scientific approaches. They will act as light-towers, multiplicators through their social networks, and be the ambassadors to attract new talents to the refractory industry in the near future. The training received by the ESRs, at the ATHOR Refractory Training Courses and a series of webinars by academic and industrial experts, was recorded and stored on the database ELEANOR. Different levels of access can be granted (ranging from "general public" to "ATHOR consortium only" depending on the preference of the author of the content) that can be accessed by future refractories students free of charge. Thus, local universities have access to high quality, industrially relevant teaching materials, which will continue to be updated after ATHOR, to provide to future students. It will also be used in an industrial context to train new generations of researchers, engineers, and for those who want to refresh their knowledge.

Regarding the long-term, two main tools were developed during the project, the dedicated videos and the "CeramiK" exhibition. These tools will be used to attract new students in the domain of refractory materials and structures. They will most likely be the best marketing tools to attract people far beyond the four years of the project itself. They will give visibility of the refractory industry to all levels of the society including policy makers, young talent looking to choose their career paths, and to researchers and engineers in industries beyond refractories. Those tools will definitely facilitate the attraction of excellent talent to join the relatively unknown and small refractory community that is so essential to all high temperature processes and to our daily lives.

5.3 The impact of ATHOR on a scientific level

From refractory producers through to end users, all sections of the refractories value chain have benefitted from the research carried out during the ATHOR project.

New and improved measurement tools have been developed including the ATHORNA device, a bi-axial high temperature testing device and a 3D pilot steel ladle. These were used to validate, at mesoscopic level, the results of numerical modelling based on new mathematical and numerical method taking into account mesoscopic materials and contact properties. Both the measurement tools and models will continue to be exploited by both the industrial and academic partners.

A large database of data generated from the advanced characterisation of raw materials used in refractories manufacture has been developed. This will remain available to the project partners for future use.

The optimisation of industrial devices will be made possible through future simulations of industrial problems. In order to obtain data for the validation of advanced analysis methods, refractory masonries were characterized experimentally under different, complex conditions. A large and comprehensive experimental campaign was implemented, wallets were constructed and subjected to uniaxial and biaxial loading from room temperature to high temperature. A 3D pilot steel ladle was designed, constructed and tested. The 3D pilot scale and the full-scale models investigated have provided unique and valuable data for the calibration and validation of the advanced numerical macro-models developed and will continue to be exploited by our partners.

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One of the most significant and direct impacts is without a doubt that made by the direct employment of ERSs by the industrial partners. With their expertise on modelling and characterization, the ESRs will be part of new development activities to reduce cost and time required for the development of new materials and technologies.

The scientific and technical exchanges in the ATHOR consortium will continue beyond the life of the project and will facilitate future cooperation between refractory suppliers and steel producers. In addition, having some academic partners beyond the classical ceramic/refractory domain was very useful as the transfer of knowledge was possible and very efficient, specifically in the case of expertise on joints and mortar from the civil engineering field. New ideas have been triggered by the mix of expertise.

This network has already and will continue to foster collaboration and procurement of funding towards the development of other research projects. For example, the recently awarded Horizon Europe MSCA-DN-ID project CESAREF has several original partners from the ATHOR project.

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