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A Creep Model with Different Properties Under Tension and Compression – Applications to Refractory Materials

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Abstract

Refractories are materials designed to work at high temperatures, and are applied in steel making, cement making, aerospace engineering, and other fields where a combination of chemical and mechanical stability is paramount. Due to such high temperature applications, creep strains play an important role in the mechanical performance of refractories, that often present an asymmetric behavior, i.e., different creep strain rates under tension and compression. The aim of this work is to propose an asymmetric creep model that can be used to simulate the time-dependent non-linear primary and secondary creep behavior of refractories at high temperature. The proposed model uses a split of the stress tensor into positive and negative parts and further calculation of the contribution of each stress sign to the overall strain rate using a weighted average over the equivalent stresses. An experimental procedure is proposed in order to identify the tensile and compressive parameters of an alumina-spinel refractory used in steel ladle linings, using a Brazilian test and a subset-based Digital Image Correlation (DIC) technique, for a temperature of 1300 °C.

Keywords: Asymmetric Creep, Refractories, Finite Elements, Modeling

1 List of Symbols

<u><u></u><i>σ</i></u>	Second rank stress tensor
σ_{eq}	Von Mises equivalent stress
σ_x	Stress in direction x
σ_y	Stress in direction y
f_y	Yield stress
<u>\$</u>	Second rank deviatoric stress tensor
σ_t	Tensile strength
σ_c	Compression strength
$arepsilon_x$	Total strain in direction x
ε_y	Total strain in direction y
p	Equiv. creep (viscoplastic) strain
$\underline{\dot{\varepsilon}}^{cr}$	Second rank creep (viscoplastic) strain rate tensor
E	Young's modulus
ν	Poisson's ratio
A,n,m	Temperature dependent creep parameters
w	Weights
e	Sample's thickness
ϕ_s	Sample's diameter
ϕ_j	Jaws' diameter
Ī	Identity matrix
Tr()	Trace
$\langle \rangle$	Macaulay brackets
x^{\pm}	Positive and negative parts of variable x
F	Force

- t Time
- $\mathbf{2}$

3 1. Introduction

Refractories are materials designed to work at highly aggressive environments, and need to resist to thermo-mechanical stresses and strains, corrosion and erosion at temperatures that can reach over 2000 °C (Banerjee, 2004). In the production of steel, cement, glass, copper, between other materials processed at high temperatures, the goal of refractory linings is to protect the vessels used in the production process, normally made of steel, from overheating and consequent mechanical failure, as well as to control the heat losses from the process 10 (Schacht, 2004). 11 The transformation of raw materials into finished steel products involve high 12 temperature processes, and different vessels lined with refractories are used to 13 contain the molten metals, such as blast furnaces, basic oxygen furnaces (BOFs), 14

electric arc furnaces (EAFs), ladles and tundishes. Among those vessels, the 15 steel ladle has a considerable importance, since it is used in the secondary met-16 allurgy, where a considerable amount of time and financial resources have al-17 ready been previously employed on the production process, and is responsible 18 for a non-negligible consumption of refractories (Dutta & Chokshi, 2020). The 19 integrity of the ladle is of central importance to the safety of the production 20 site, since it transits between other equipments and workers, carrying molten 21 steel, and a failure can lead to serious consequences. 22

Figure 1a shows a cutaway view of a steel ladle, and Figure 1b shows a schematic representation of the top view of one ring of a ladle lining. The choice of the refractories to be applied in each layer of the lining greatly influences the temperature distribution in the ladle, therefore playing also a crucial role on its thermo-mechanical behavior (Volkova & Janke, 2005) and on the energy consumption.

An important phenomena to be considered in the design of the steel ladle is the creep and stress relaxation of the refractory lining. The absence of a non-

 $_{\rm 31}$ $\,$ linear viscoplastic behavior can lead to stress values that can't be supported by

 $_{2}$ the vessel, causing a mechanical failure. Nevertheless, if the material presents



Figure 1: Example of a steel ladle. (a) Cutaway view showing the refractory linings. (b) Schematic representation of a line ring, top view.

33	excessive creep, it will have a considerable reduction on its size caused by the
34	compression stresses at the hot face. During the cooling of the ladle, joints
35	may open, making the lining loose and allowing for steel infiltration in posterior
36	production cycles. Therefore, the design of the refractory lining and of the steel
37	shell can be nefit from a numerical model that can predict more accurately the
38	stress and strain variations over time, and the effect of thermal cycles can be
39	taken into account to maximize the vessel's life.
40	Due to the complexity of their mechanical behavior, the modeling and sim-
41	ulation of refractory materials has often been restricted to the application of
42	simple constitutive models, in some cases even neglecting the well known non-
43	linearity of the material (Jin et al., 2011). In other cases, creep models have
44	been applied to simulate the thermo-mechanics of refractories, but considering a
45	symmetric behavior and using the compressive mechanical properties (Jin et al.,
46	2020). There were also attempts to use a Drucker-Prager-based creep model to
47	simulate the effect of the material's asymmetry of RH-degassers lining behavior,
48	but the authors concluded that this model might not be ideal and that more
49	complex representation of the actual behavior should probably be used (Jin et
50	al., 2015).

- In this work, an asymmetric creep model is proposed, which is able to repre-51
 - 4

sent the different behavior that refractory materials present under tension and 52 compression. The main goal is to obtain a relatively simple constitutive model, 53 that can have the material parameters identified using the least possible num-54 ber of mechanical tests, due to the need to characterize the material at several 55 temperatures, which requires a high time and energy consumption. With the 56 proposed model, the lifetime of refractory materials can be predicted more ac-57 curately than the using current engineering practice (linear elastic or symmetric 58 creep models), since it accounts for the significant increase in the creep strain 59 rate when tensile loads are present in the lining. Similarly, the model allows for a better comparison between the performance of products with different 61 shapes and compositions, reducing field trials and improving the availability of 62 the equipments. 63

Section 2 provides a description of the current creep models available in the 64 literature, explaining what are the missing features when the simulation of refractory materials need to be done. Section 3 describes the proposed model, 66 and comments on possible numerical difficulties that can arise when implement-67 ing it, as well as numerical strategies that can be used to mitigate them. Sec-68 tion 4 presents numerical simulations used to theoretically validate the proposed 69 model. Section 5 describes the high temperature experimental procedure used 70 to characterize the material, with a detailed explanation of the reason why the 71 Brazilian test method was used. Finally, Section 6 presents the identification of 72 the material parameters of an alumina-spinel brick used in steel ladles. 73

74 2. Symmetric and asymmetric creep models

The creep behavior of ceramic materials can be split in three stages. The first stage, called primary creep, presents a time-dependent strain rate which decreases with time. In the secondary creep stage, the strain rate is considered to be constant, and an approximate equilibrium between hardening and softening processes can be assumed (Naumenko & Altenbach, 2007). Finally, in the third creep stage, the strain rate increases with time until the failure of the material

81 (Jin et al., 2014).

82 2.1. Symmetric creep models

One of the most traditional creep models available in the literature is based on the one-dimension Norton-Bailey's law, known on its multi-dimensional version as Odqvist's law (Lemaître & Chaboche, 1990), which relates the creep strain rate $\underline{\dot{\varepsilon}}^{cr}$ to the stresses using the following equation:

$$\dot{\underline{\varepsilon}}^{cr} = \frac{3}{2} \underline{\underline{\varepsilon}}^{A} \sigma_{eq}^{n-1} p^m \tag{1}$$

where $\underline{\underline{s}}$ is the deviatoric component of the stress tensor, σ_{eq} is the von Mises equivalent stress, p is the equivalent viscoplastic strain (sometimes refered to as equivalent creep strain) and A, n, and m are temperature dependent material parameters. For the case of secondary creep, m = 0. The Norton-Bailey's law have been extensively used to characterize refractory materials, due to its simplicity and good fitness to experimental results (Jin et al., 2014; Samadi et al., 2020; Schachner et al., 2019; Sidi Mammar et al., 2016; Teixeira et al., 2020).

If an elastic region needs to be considered in the model, a simple modification
in Equation 1 can be done to take it into consideration (Lemaître & Chaboche,
1990):

$$\underline{\dot{\varepsilon}}^{cr} = \frac{3}{2} \frac{\underline{s}}{\sigma_{eq}} A \left\langle \sigma_{eq} - f_y \right\rangle^n p^m \tag{2}$$

where f_y is the yield stress and $\langle \rangle$ are the Macaulay brackets.

It should be noted that different uniaxial laws other than Norton-Bailey's law can be applied to relate the stresses to the creep strains (Lemaître & Chaboche, 1990). The choice of which law should be used depends on how accurately it fits the mechanical tests.

103 2.2. Asymmetric creep models

As can be noticed in Equations 1 and 2, the Norton-Bailey creep model is not able to represent materials with different behaviors under tension and

¹⁰⁶ compression (asymmetric creep).

The asymmetry in the material's response was approached by Altenbach (2001) using a creep strain rate formulation that depends on three linear independent invariants of the stress tensor. Alternatively, Mahnken (2003) proposed an asymmetric creep model that uses a scalar variable expressed in terms of the ratio of the second and third invariants of the deviatoric stress tensor, called stress mode angle in the octahedral plane in the deviatoric stress space. Although these models are mathematically consistent and were successfully

applied, they present the inconvenient to require a considerable number of experiments to determine the creep parameters, since tension and compression
behaviors are not decoupled. For the case of refractories, that need to be characterized at several temperatures, this represents a limitation.

More recently, Samadi et al. (2021) coupled the asymmetric primary creep behavior of refractories with a damage model. In this model, the equation for the creep strain rate is selected according to the sign of the principal stresses, without any particular weighting strategy, and the equations are solved using an explicit integration scheme.

To account for the asymmetric creep of refractories, Blond et al. (2005) extended the Norton-Bailey's model, using a split of the principal stress vectors into a positive and a negative parts, to propose a secondary creep model, resulting in:

$$\underline{\underline{\sigma}} = \langle \underline{\underline{\sigma}} \rangle - \langle -\underline{\underline{\sigma}} \rangle \tag{3}$$

This split results in the definition of the model in terms of independent tension and compression parameters. In this sense, the two parts of the deviatoric stress tensor are given by:

$$\underline{\underline{s}}^{\pm} = \left\langle \pm \underline{\underline{\sigma}} \right\rangle - \frac{1}{3} Tr(\left\langle \pm \underline{\underline{\sigma}} \right\rangle) \underline{\underline{I}}$$

$$\tag{4}$$

 $_{130}$ $\,$ where the indexes \pm indicate the positive and negative parts of the variables,



Figure 2: Yield surface of Blond's model.

¹³¹ respectively. The equivalent von Mises stresses are, then:

$$\sigma_{eq}^{\pm} = \sqrt{\frac{3}{2}\underline{\underline{s}}^{\pm}} : \underline{\underline{\underline{s}}}^{\pm}$$
 (5)

¹³² resulting in a viscoplastic strain rate of the form:

$$\underline{\dot{\varepsilon}}_{cr} = \frac{3}{2} \frac{\underline{\underline{s}}^{+}}{\sigma_{eq}} A^{+} \left\langle \sigma_{eq}^{+} - f_{y}^{+} \right\rangle^{n^{+}} - \frac{3}{2} \frac{\underline{\underline{s}}^{-}}{\sigma_{eq}} A^{-} \left\langle \sigma_{eq}^{-} - f_{y}^{-} \right\rangle^{n^{-}} \tag{6}$$

where A^{\pm} and n^{\pm} are material's constants in tension (+) and compression (-). Figure 2 shows a representation of Blond's model in the 2D principal stresses domain. In this example, the tensile yield stress is $f_y^+ = 2$ MPa and the compressive yield stress is $f_y^- = -8$ MPa. In the figure, the stress states D and E marked as green triangles are inside the elastic region, therefore no creep is observed.

Point *A* shows a stress state in the second quadrant, with a compressive component in direction 1 and a tensile component in direction 2. Therefore, the stress split described by Equation 3 results in other two stress states, shown as circular markers. As can be seen, the point (-5, 0) is in the elastic region, so no creep is expected for the compressive component. On the other hand, the point (0, 3) lays outside the elastic region, so the first term of the right hand side of ¹⁴⁵ Equation 6 is activated, and creep in tension is observed.

Point B corresponds to the opposite situation, i.e., creep is activated in compression but not in tension. The stress state represented by Point C results in creep in both tension and compression.

From Figure 2, it is also evident that Blond's yield surface presents singularities at the points where it crosses axis 1 and 2, as well as in the corners present in the second and fourth quadrants. When the structure being modeled has stress states close to these singularities, difficulty in the convergence may arise. Nevertheless, this should not be a problem if the yield stress is equal to zero, i.e., the material starts to creep as soon as a load of any magnitude is imposed, which is often the case for refractories (Jin et al., 2014).

One of the main advantages of this model is that the material's parameters for tensile and compression creep are completely independent, therefore they can be identified separately. This gives flexibility to fit the creep flow calculations to a large range of strain rates observed in experiments.

Nevertheless, the validity of the stress split hypothesis needs to be verified, 160 and this model presents the characteristic of making a sum between two terms 161 (positive and negative strain rates) that depend on the equivalent stress. The 162 consequence of this fact is that, if this model is used with identical properties 163 in tension and in compression, the resulting viscoplastic strain rate is not the 164 same as the one obtained with a symmetric model. Although this is not nec-165 essarily a limitation, in some cases it can be desirable to have a model that is 166 an interpolation of the tension and the compression material behaviors, without 167 this intrinsic orthotropy. 168

As remarked by Esposito and Bonora (2011), in many applications the primary creep of materials can't be neglected, since a considerable part of the allowable design strain occurs in this stage. This is the case of the alumina-spinel material studied in this work, as it is evident by the creep curves previously published by Samadi et al. (2020) and Teixeira et al. (2020).

¹⁷⁴ 3. Proposition of an asymmetric creep model considering primary

and secondary creep stages

176 3.1. Model description

In this work, to represent the primary and secondary creep behaviors of refractory materials, Equation 1 was adapted following the same principle of the split of the stress tensor in a positive and a negative part, as used by Blond et al. (2005).

The proposed model also differentiates from Blond's model in the way to consider the different contributions of the compression and tensile characteristics of the material. After the decomposition of the stress tensor, the deviatoric and equivalent stresses are calculated for each part (positive and negative) using Equations 4 and 5, respectively, such as in the model proposed by Blond et al. (2005).

¹⁸⁷ Nevertheless, instead of using $\underline{\underline{s}}^{\pm}$ and σ_{eq}^{\pm} to directly calculate the positive ¹⁸⁸ and negative viscoplastic strain rates (Equation 6), these values are used to ¹⁸⁹ calculate relative weights that each part of the stress tensor have on the total ¹⁹⁰ equivalent stress, using the relation:

$$w^{\pm} = \frac{\sigma_{eq}^{\pm}}{\sigma_{eq}} \tag{7}$$

Each portion of the viscoplastic strain rate is calculated as a function of the total deviatoric and equivalent stresses (using the full stress tensor, before the decomposition into positive and negative parts) and the respective material properties:

$$\underline{\dot{\underline{\varepsilon}}}^{cr^{\pm}} = f(\underline{\underline{s}}, \sigma_{eq}, A^{\pm}, n^{\pm}, m^{\pm}) \tag{8}$$

Later, each part of the viscoplastic strain rate is weighted by the values calculated using Equation 7. Therefore, the viscoplastic strain rate of the proposed asymmetric creep model is given by:

$$\underline{\underline{\dot{\varepsilon}}}^{cr} = w^+ \cdot \frac{3}{2} \frac{\underline{\underline{s}}}{\sigma_{eq}} A^+ \left\langle \sigma_{eq} - f_y^+ \right\rangle^{n^+} p^{m^+} - w^- \cdot \frac{3}{2} \frac{\underline{\underline{s}}}{\sigma_{eq}} A^- \left\langle \sigma_{eq} - f_y^- \right\rangle^{n^-} p^{m^-} \tag{9}$$

195 This model is also similar to the one developed by Mahnken (2003), in the sense that it proposes a weighted calculation of the creep strain rate. Never-196 theless, the model proposed here can be considered to be more straightforward, 197 and it also requires less parameters to characterize the creep behavior of a given 198 material, which is an important advantage. This model was implemented in an 199 Abaque UMAT subroutine, and examples of calculations are shown in the next 200 sections. The integration algorithm according to Benallal et al. (1988) was used 201 in this subroutine. 202

It should be noted that, when the material presents primary creep under 203 compression and secondary creep under tension, such as in the case of the 204 alumina-spinel brick, $m^- = 0$ in Equation 9. To summarize, Figure 3 presents 205 flow charts corresponding to the algorithm proposed by Blond et al. (2005) (full 206 lines) and the algorithm proposed in this work (dashed lines).

3.2. Numerical difficulties associated with primary creep 208

207

At the beginning of a structural simulation, when the load was not yet 209 applied, the strain in the simulated body is assumed to be zero, unless otherwise 210 stated. In this situation, it is reasonable to assume that the equivalent creep 211 strain is p = 0. Examining Equation 9 and taking into account that the variable 212 m can only take negative values, it is easy to deduce that: 213

$$\lim_{p \to 0} \underline{\dot{\varepsilon}}^{cr} = 0 \tag{10}$$

Therefore, the equivalent viscoplastic strain can't be defined as zero at time 214 t = 0, since this would result in the absence of creep strain in the subsequent 215 time step, independently of the applied load. 216

The first solution to this problem is to define a low non-zero initial value 217 for p. Nevertheless, it is not always clear which value should be used, since the 218 convergence of the simulation is highly dependent on it. For example, consider a 219 case where only compression creep is applied and $f_y = 0$. Equation 9 becomes: 220

$$\dot{\underline{\varepsilon}}_{=}^{cr} = \frac{3}{2} \underline{\underline{s}} A^{-} \sigma_{eq}^{(n^{-}-1)} p^{m^{-}}$$
(11)



Figure 3: Asymmetric creep model algorithms. Blond et al. (2005) (full lines) and the proposition of this work(dashed lines)

²²¹ Supposing $\underline{\underline{s}}$ and σ_{eq} to have an order of magnitude of 10, $A \approx 1 \times 10^{-10}$, ²²² $m \approx -2$, $n \approx 2$ and an initial approximation of $p = 1 \times 10^{-15}$, the resulting creep ²²³ strain rate would be $\underline{\underline{\dot{e}}}^{cr} \approx 1 \times 10^{22}$. A high value of initial creep strain rate is

expected from primary creep, but at this rate of strain, the integration algorithm would have to use an extremely low time step value in order to converge, which is not practical.

It can be concluded that an initial equivalent viscoplastic strain value should be used so that it is small enough to do not decrease the accuracy of the solution and large enough to allow the convergence in a reasonable time frame. This value depends on the material properties, the applied loads, the boundary conditions and on the time stepping used during the solution, and can be considerably difficult to estimate.

To improve the convergence of the model, the following actions can be taken:

1. Given the material properties and boundary conditions of the problem, an initial value for the equivalent creep strain $p_{t=0}$ needs to be determined. The convergence of the initial steps of the model is tested, using a small time increment. If necessary, $p_{t=0}$ should be increased.

2. The time step increment should be small during the first seconds of load-ing, since the primary creep curve can be steep at the beginning. It can be interesting to brake the initial steps into smaller ones, to allow a finer control of the time increment.

3. Even when the initial moments of primary creep are past, the maximum increment of time should be limited to reasonable values, since the automatic time stepping control present in some FEA software can attempt to increase it beyond the convergence limit of the integration algorithm. Even if the software is able to converge, it can take many iterations to finish a step, leading to slower computations.

Even if those measures are taken, the simulation can still diverge due to a rapid change in the creep strain rate, specially for the case where tensile loads are meaningful. To limit this problem, in the UMAT subroutine used to implement the creep models developed in this work, a variable was implemented to control the convergence of the integration algorithm. If the algorithm does not converge, the subroutine automatically returns to the previous converged iteration, and tries a time step that is half the previous attempt. This action is
only effective if the convergence problem is related to the integration algorithm
of the constitutive equations, not to the global Newton-Raphson integration
scheme.

258 4. Numerical simulations using the proposed asymmetric creep model

To evaluate the capabilities of the proposed asymmetric model, a set of 259 numerical simulations is presented, in increasing order of complexity. The goal 260 of these simulations is to verify if the model presents the expected behavior when 261 subjected to complex load cases. The material parameters used to perform these 262 tests are shown in Table 1. As it will become evident in the next sections, these 263 material parameters are close to the one corresponding to the alumina-spinel 26material. It was considered that the tensile behavior of the material could be 265 approximated by a secondary creep model, as discussed by Teixeira et al. (2020). 266

Parameter	Compression	Tension
E[MPa]	30000	30000
u[-]	0.2	0.2
$\log_{10} A[\mathrm{MPa}^{-n}s^{-1}]$	-14.16	-5.4
n[-]	3.96	1.5
m[-]	-2.74	_

Table 1: Material parameters used in the numerical simulation tests

²⁶⁷ In the simulations, four situations regarding the type of model and the ma-²⁶⁸ terial parameters were considered:

1. Abaque symmetric creep model using the compression properties of the

material. This configuration is commonly seen in publications related tothe creep of refractories, as previously cited.

272	2. Abaqus symmetric creep model using the tension properties of the mate-
273	rial, used as a reference to compare with the asymmetric model.
274	3. UMAT asymmetric creep model, but using the compression properties
275	of the material for compression and tension, to verify if the asymmetric
276	model specializes to a symmetric one when necessary.
277	4. UMAT asymmetric creep model, using the corresponding properties for
278	tension and compression.
279	4.1. Normal loads with stress reversal in two dimensions

The first model corresponds to a simple two-dimensional element (Figure 4a, where e is the thickness) subjected to a load path that varies in directions x and y, according to Figure 4b. The maximum tensile stress is $\sigma_t = 0.2$ MPa, and the minimum compression stress is $\sigma_c = -2.0$ MPa. Figures 4c and 4d show the time periods for which the stresses are kept in the sample.

Although this is a simple model, it represents a situation where, during 285 the loading cycle, there are moments where both principal stresses are positive 286 (Point B), both are negative (Point D), and when there is a composition of 287 positive and negative stresses at the same integration point (Points C and E). 288 Therefore, the simulation is useful to show the difference between a symmetric 289 and an asymmetric model, as well as the effect of the loading history. For this 290 model, an extra curve using the tension material parameters for both tension 29 and compression material laws is also presented. 292

Figure 5 shows the accumulated viscoplastic strains computed in each of the situation previously described. It is possible to observe that, when the same properties are used for tension and compression, the UMAT provides the same results as a symmetric model. More importantly, the asymmetric model presents an intermediate response between the tension and the compression symmetric models, as expected.

In Figure 5 it is clear that, until approximately 30 min, when only tension stresses are present in the element (Points A and B in Figure 4), the symmetric and asymmetric models give the same result. From that point further, when



Figure 4: Stress distribution – Normal loads. (a) Simulation model. (b) Stress path. (c) σ_x vs Time. (d) σ_y vs Time.

an asymmetry is included in the loading (Point C in Figure 4), the model re sponse changes, becoming an interpolation between the tension and compression
 behaviors.



Figure 5: Accumulated viscoplastic strain - Normal loads.

305 4.2. Brazilian test

Figure 6 shows the geometry, mesh and boundary conditions used to compare the symmetric and asymmetric models applied to a Brazilian test specimen. The sample was discretized using linear square elements with full integration, unless for a transition zone between the refined mesh in the contact region and the rest of the geometry, where linear triangular elements with full integration were used.

The sample was modeled with $\phi_s = 50$ mm diameter and e = 40 mm thick-312 ness. Analytic rigid surfaces representing the upper and lower jaws with diam-313 eters $\phi_j = 65 \text{ mm}$ were used to distribute the load more evenly on the sample 314 and to avoid excessive stress concentrations. The same strategy was used at the 315 bottom part of the model to restrict the vertical displacement of the sample. 316 Due to the geometry and force symmetries, only half of the sample was modeled. 317 A force of -400 N was applied on the model following a 30 s linear ramp, 318 and it was kept for two hours. This geometry and boundary conditions are the 319 same used for the mechanical tests at 1300 °C, presented in Section 5. 320 A comparison between vertical and horizontal displacements taken at the 321

³²² center of the sample for the four cases is shown in Figure 7. It is possible to



Figure 6: Brazilian test – Geometry, mesh and boundary conditions.

observe that, when compression curves are used in the asymmetric model, the
result is in high agreement with the symmetric model available on the software
Abaqus.



Figure 7: (a) Vertical and (b) horizontal displacements in the Brazilian test sample.

Figures 8 and 9 show the maximum principal stress and the minimum principal stress on the sample, respectively, for the cases where only tension or compression material parameters were considered (symmetric creep), and the asymmetric case. It can be observed that the maximum tensile stress value obtained on the sample was 0.35 MPa. Since refractory materials are less re-

sistant to tension, this stress limits the amount of force that can be applied 331 before the failure of the sample. The maximum compressive stresses observed 332 on the sample are in the range of -2.5 MPa to -3.8 MPa, although at the 333 center of the sample this value does not go higher than -1.8 MPa. This can be 334 considered as a limitation of the Brazilian test when compared to uniaxial tests 335 for the identification of material parameters, i.e., the tension and compression 336 loads are not decoupled, therefore the material can only be safely character-337 ized for a smaller stress range. Nevertheless, this only represents a problem if, 338 during operation, the actual compression stresses withstand by the material are considerably higher than the ones during the mechanical tests. 340

It should be noted that, in Figures 8 and 9, the contact zones were excluded 34: from the results plots. This was done because, in these regions, the model can 342 experiment stress concentrations, that makes the visualization of the plots more 343 difficult in the other regions of the model. In a real experiment, these regions suffer from a local failure, with the crash of the grains at the contact with 345 the jaws. This failure can normally be neglected in the experiment, but it can 346 influence the convergence of the numerical model. For instance, the elements 347 with higher stresses present, as a consequence, higher creep strain rate, and the 348 model takes longer to converge due to the need of low time steps, even if the bulk part of the sample is still under low stresses. To use the proposed asymmetric 350 model, attention should be payed to such stresses concentration areas, and they 35 should be removed from the model, if possible. 352

When compared to a symmetric model using the compression creep prop-353 erties, the asymmetric model has a considerably different behavior over time 354 regarding the strains. Figure 10 shows that, over the central line of the sam-355 ple, the total strain ε_x , that is mainly positive, is the same for both models at 356 the end of loading (30 s). Nevertheless, after one hour, the strain obtained by 357 the asymmetric model is approximately the double of the one obtained by the 358 symmetric model, and after two hours the ratio between them is around three. The same proportional difference is observed on Figure 11 for the total strain 360 ε_y , that is compressive. This fact is due to the averaged sum of the tension



Figure 8: Maximum principal stress distribution on a Brazilian test sample. (a) compression (b) tension and (c) asymmetric material properties.



Figure 9: Minimum principal stress distribution on a Brazilian test sample. (a) compression (b) tension and (c) asymmetric material properties.

³⁶² and compression creep strain rates applied by the asymmetric model, where the

³⁶³ tension part contributes to considerably increase the value of the strains.

Finally, Figure 12 shows the effect of the asymmetry in the accumulated viscoplastic strain on a Brazilian test. Once again, the proposed asymmetric model shows an intermediary behavior between the symmetric ones, and evidences the importance of the consideration of the different material properties in tension

368 and in compression.



Figure 10: Variation of the total strain in direction x with time – Symmetric and asymmetric models.



Figure 11: Variation of the total strain in direction y with time – Symmetric and asymmetric models.

369 4.2.1. Influence of the constitutive model parameters

To have a better idea on how each of the constitutive model parameters 370 influences the results obtained from a Brazilian test simulation, calculations 371 were made varying one parameter at a time around the absolute values of the 372 nominal properties presented on Table 1 by -10%, -20%, +10% and +20%. 373 These values are shown in Table 2. This method is not considered an accurate 374 sensitivity analysis, and the intention is to qualitatively understand how the 375 parameters influence the shape of the time-displacement curves, which is an 376 useful information when an inverse identification of the parameters using real 377



Figure 12: Accumulated viscoplastic strain distribution on a Brazilian test sample. (a) compression (b) tension and (c) asymmetric material properties.

³⁷⁸ experiments needs to be done. Figure 13 shows the variation of the vertical

 $_{379}$ displacement u_y of the top of the sample as a function of time, according to the

380 change of the parameters.

Parameter	+10%	+20%	-10%	-20%	
$\log_{10} A^-[\mathrm{MPa}^{-n}s^{-1}]$	-15.57	-16.99	-12.74	-11.32	
$n^{-}[-]$	4.35	4.75	3.56	3.16	
$m^{-}[-]$	-3.01	-3.28	-2.46	-2.19	
$\log_{10} A^+ [MPa^{-n}s^{-1}]$	-5.94	-6.48	-4.86	-4.32	
$n^{+}[-]$	1.65	1.8	1.35	1.2	

Table 2: Variation of the material parameters – Isotropic creep model

Figures 13a, 13b and 13c show the influence of the compression parameters A^- , n^- and m^- , respectively. The curve with the variation of -20% was not plotted, since the displacement was excessively high, due to the large influence of this parameter in the results. It is possible to observe that parameter n^- , that is an exponent of the stress, has a negligible influence on the results, due to the low values of stresses in this application. Parameters A^- and m^- influence mostly the beginning of the curve, changing its curvature radius, indicating that the compression stresses are predominant during the first hour, and the tensile stresses take over from this point further. This is an important conclusion, since it helps to decide in which part of the curve the identification procedure will focus, depending on the desired result. Similarly, A^+ has the higher influence among the tensile parameters, as can

Similarly, A^+ has the higher influence among the tensile parameters, as can be observed in Figures 13d and 13e, although n^+ starts to be influent after one hour of loading and can become more meaningful for longer periods. Contrary to the compressive parameters, the tensile parameters, specially A^+ , change mainly the slope of the curve after the initial curvature has passed. Again, this remarkable separation in time between the influence of the tensile and compressive parameters facilitates the inverse identifications.

³⁹⁹ 5. Experimental methodology

In this work, Brazilian tests were used to identify the creep parameters of 400 an alumina-spinel material. Traditionally, Brazilian tests have been applied to 401 study the tensile strength of concretes and geomaterials, as a replacement to 402 direct tensile tests. As pointed by several studies, the results of a Brazilian test 403 to calculate the tensile strength of a given material is only valid if the crack 404 initiation is at the center of the sample, and not at the stress concentrations 405 present at the contact points (Darvell, 1990; Fairhurst, 1964; García et al., 406 2017). For the applications in the current work, the position of the initiation of 407 the crack is not important, since no fracture is expected during the test. 408

The Brazilian test was chosen for the identification of the asymmetric creep parameters due to the complexity of the stress field developed in the sample during the application of the load. As shown in Figure 14, tensile stresses are developed at the center of the sample, while compressive stresses start to increase towards its borders and shear stresses are present in the regions in contact with the upper and lower jaws. A more detailed description of the



Figure 13: Change on the asymmetric creep model response due to variations of the material parameters – Brazilian test.

- 415 stress equations related to the Brazilian test can be found in Fahad (1996).
- $_{416}$ This stress distribution is adapted to the current case, since it allows the study
- 417 of the effect of the material properties under tension and compression using a
- single test, which considerably reduces the number of experiments that need to
 be performed.



Figure 14: Stress distribution in a Brazilian test sample

The application of Brazilian tests to refractory materials is still limited, spe-420 cially regarding the characterization of creep behavior. Belrhiti et al. (2017) used 421 this test in association to a DIC technique to characterize the mechanical be-422 havior of magnesia hercynite refractories used in cement rotary kilns, estimating 423 the elastic modulus, Poisson's ratio and fracture energy at room temperature. 424 Gazeau et al. (2015) used the Brazilian test associated with the integrated DIC 425 technique to identify the Young's modulus and the tensile strength of plane 426 membranes at temperatures up to 900 °C. These two applications demonstrate 427 the potential of this method to provide various information using a single ex-428 perimental setup, what can result in the reduction of the required number of 429 tests to characterize a material. This is specially important considering the cost 430 and time demand of high temperature tests. 431

432 5.1. Experimental setup

In this work, the Brazilian test was used in association with a DIC technique.
This technique is convenient for the current application, since the traditional
instrumentation of testing setups at high temperatures present significant difficulties.

Previous works have given detailed information about DIC applications at 437 high temperatures (Leplay et al., 2015; Leplay et al., 2010, 2012; Novak & Zok, 438 2011). When pictures at high temperatures need to be taken, the excessive 439 black body radiation coming from the furnace needs to be filtered, otherwise 440 the image is oversaturated and presents no contrast. This is frequently achieved 441 by using blue band pass filters, and sometimes neutral density filters depending 442 on the exposure time of the pictures, that only allows that the blue part of the 443 light spectrum arrives at the camera sensors. To increase the quality of the 444 pictures, it is also common to enlighten the sample using a blue light source. 445

Figure 15 shows the experimental setup used to perform the Brazilian tests and to take the pictures at high temperatures. This setup is composed of the following parts:



Figure 15: Experimental setup for the Brazilian tests at high temperature

449	1.	A water cooled custom made furnace with the capacity to heat up to
450		1300 °C.
451	2.	The furnace's door is equipped with a window made of a vitro ceramic ma-
452		terial, so that the sample can be photographed. This material was chosen
453		because of its adequate resistance to temperatures up to 1300 $^{\circ}\mathrm{C},$ its low
454		thermal expansion and its inexpensive price. The material is transparent
455		enough to allow that satisfactory pictures are taken.
456	3.	To increase the amount of blue light available when taking the pictures,
457		two blue light sources are used to enlighten the sample. In combination
458		with the blue filters, this increases significantly the contrast of the pictures,
459		to a point where the DIC analysis becomes possible. These lights are
460		mounted in a dedicated support that was designed to offers six degrees of
461		freedom, so they can be easily positioned to avoid glare in the pictures.
462	4.	A high resolution 12 bit CCD camera, AF Micro-Nikkor 200 mm lenses
463		and a blue band pass filter, to decrease the amount of light being captured
464		by the camera, since it blocks all parts of the optical spectrum that are
465		not blue, avoiding the saturation of the sensor.
466	5.	The camera and the lenses are cooled using two ventilators, since they
467		stand close to the furnace and can overheat. A third ventilator is used to
468		blow away the hot air between the window and the lenses, minimizing the
469		generation of heat hazes.
470	6.	A load cell with maximum capacity of 30 kN.
471	Т	he samples used in the Brazilian tests were cut from parallel refractory
472	brick	s. The first step was to cut slices 40 mm thick, to guarantee the parallelism
473	of th	e sample surface exposed to the camera. Second, 50 mm cylinders were
474	drille	d from these slices. This geometry was chosen to comply with a rule of
475	thum	b on the refractories field that requires the minimum dimension of the
476	samp	le to be at least 10 times the size of the largest grain in the material, that,
477	in th	e case of the alumina-spinel brick, is 3 mm.
478	Т	o improve the contrast of the samples surfaces, a SiC powder speckle pat-

tern was used, with grain sizes varying from 50 μ m to 100 μ m. The surfaces of 479 the sample were covered with a bonding agent, and the powder was deposited 480 using a sieve, similar to what was done by Archer et al. (2020), what allowed a 481 reasonable control over the particles dispersion. Using this method, the speckle 482 does not remain strongly attached to the sample, and, even if it does not detach 483 under simple gravitational action, direct contact with it should be avoided. Be-484 fore the test, the sample was let at rest for 12 hours, in order to dry the excess 485 of the bonding agent. Figure 17a shows an example of a sample coated with a 486 speckle pattern, already positioned at the testing machine. To guarantee the good quality of the pictures, a rigorous procedure was 488

followed at each test. This procedure was composed of:

The sample and the jaws were positioned at the testing machine at room
 temperature, and an initial pre-load of approximately 50 N was applied.

2. The furnace was closed, and the heating of the sample started. Figure 16 492 shows the heating curves used for the tests. Between room temperature 493 and 600 °C, a heating rate of 10 °C/min was used. From 600 °C to 1300 494 °C, this rate was reduced to 5 °C, to avoid thermal damage on the sample 495 and on the equipment. Before the beginning of the test, the furnace was 496 let in a dwell for 1 h, to stabilize the temperature of the testing setup. 407 The blue curve on Figure 16, obtained from one of the tests made at 1300 498 °C, shows that the displacement of the machine piston was constant before 499 the end of the dwell, indicating that there was no extra thermal expansion 500 of the equipment at the beginning of the test. 501

After the temperature homogenization time was passed, the camera lenses
 and the blue band pass filter were cleaned to remove any dust particles
 that could cause artifacts in the pictures and later mounted on the camera.
 To increase the images contrast, the blue lights were positioned to generate
 the maximum illumination of the sample as possible, avoiding glare, and
 the focus was manually adjusted in order to increase the sharpness. The
 exposure time was adjusted according to the lightning conditions to avoid

509 too bright or too dark images.

510

511

 The sample was loaded at a rate of 0.5 mm/min until the creep load was achieved, and the force was held constant until the end of the test.



Figure 16: Heating curve and evolution of machine displacements prior to the test.

Figure 17 shows the effect of the lightning and of the blue band pass filter 512 in the quality of the images. At room temperature (Figure 17a), the histogram 513 of the image, shown in Figure 18, is approximately normally distributed around 514 gray values from 75 to 200, and the image presents a high contrast. At 1200 °C, 515 without the use of the blue lights and of the blue band pass filter (Figure 17b), 516 the distribution of gray levels is restricted to the range between 105 and 115, 517 and the images looses most of its contrast. The use only of the blue band pass 518 filter, without the blue lights (Figure 17c), slightly decreases the sharpness of 519 the histogram, but not enough to guarantee enough contrast. Finally, using the 520 blue lights and filter (Figure 17d), the gray level distribution becomes closer to 521 that of the image at room temperature, and most of the contrast is recovered. 522



Figure 17: Influence of the blue light and the blue band pass filter in the quality of the image. (a) Image at room temperature. (b) No blue light and no filter. (c) No filter. (d) Use of blue light and blue band pass filter.



Figure 18: Histograms of the images at room and high temperature.

⁵²³ The open-source DIC software Ncorr (Blaber et al., 2015) was used to calcu-

 $_{\tt 524}$ $\,$ late the full field displacements of the samples. Ncorr uses the local subset-based

⁵²⁵ reliability-guided DIC method according to Pan (2009).

6. Identification of the asymmetric creep properties for the alumina spinel material at 1300 °C

Figure 19 shows the relative vertical displacement of the upper point of the Brazilian test samples at 1300 °C, calculated using DIC, that corresponds to the difference between the vertical displacements at the upper part and the vertical displacements at the lower part, to account for rigid body movements of the experimental setup. It is possible to observe that samples 1, 3, 5 and 6 are in good agreement, while samples 2 and 4 are lower and upper outliers, respectively.



Figure 19: Brazilian tests time vs displacement curves obtained by DIC.

Figure 20 shows an example of envelope for the material parameters at 1300 °C, obtained through a series of numerical simulations using the asymmetric creep model presented in Section 3. It can be expected that the material parameters will not highly deviate from the ones presented in the figure, even



⁵³⁹ if this analysis only considers a single displacement value, and not the full field.

Figure 20: Brazilian tests time vs displacement curves and parameters envelope.

Finally, taking into account the influence of the material parameters in each portion of the time vs displacement curve for Brazilian tests presented in Section 4.2.1, the parameters for the alumina-spinel material were identified in order to better approximate the values of the DIC calculations, and the resulting curve is presented in Figure 21. The identified parameters are shown in Table 3.

Parameter	Compression	Tension
$\log_{10} A[\mathrm{MPa}^{-n}s^{-1}]$	-14.86	-5.55
n[-]	3.96	1.5
m[-]	-2.74	_

Table 3: Identified material parameters

546 From Figure 21 it is possible to observe that the identified curve fits the

32

experimental values with good accuracy. The full DIC displacement field of
sample 6 was compared with the results of numerical simulations performed
using the identified material parameters, to verify the robustness of the identification.



Figure 21: Brazilian tests time vs displacement curves and inverse identification.

The vertical displacements fields of Points A, B and C represented in Fig-551 ure 21, corresponding to t = 0.6 h, t = 1.05 h and t = 1.45 h, respectively, are 552 shown in Figure 22. It is possible to see that there is a rigid body rotation of 553 the sample, since the displacements map does not correspond to the traditional 554 displacements field of Brazilian tests. To consider this effect, an extra identifica-555 tion calculation was made to determine what is the magnitude of the load that 556 caused this deviation. An horizontal load of -7 N was identified and applied 557 on the upper jaws in the simulation model. This corresponds to an error of 0.5° 558 in the application of the load, which shows that the experimental procedure is 559 sensitive to small deviations from the ideal boundary conditions. 560

Figure 23 shows the results of the numerical simulations using the previously identified material parameters and the horizontal load. It is possible to observe that the displacement maps of Figures 22 and 23 have a good equiv-

- $_{\rm 564}$ $\,$ alence, despite the experimental errors and the simplicity of the identification
- 565 procedure.



Figure 22: Brazilian tests: Vertical displacement in mm at 1300 °C – DIC sample 6. (a) Point A. (b) Point B. (c) Point C.



Figure 23: Brazilian tests: Vertical displacement in mm at 1300 °C – Simulation. (a) Point A. (b) Point B. (c) Point C.

It is interesting to observe how the vertical displacement of the central line 566 of the sample changes when the rigid body motions are included in the simula-567 tion, as shown in Figure 24. When the boundary conditions are symmetric, the 568 displacement increases monotonically and equally in both sides of the sample, 569 as expected. Nevertheless, a horizontal force in the negative x direction causes 570 an increase in the magnitude of the displacements at the right side of the sam-571 ple (point A in Figure 24), while the left side presents a positive displacement 572 during loading (point B in Figure 24). After approximately 1 h of creep defor-573

34



Figure 24: Brazilian tests: Vertical displacement of points A and B.

mations, the displacement of the right side becomes negative again, but always smaller than at the left. This non-symmetric behavior, caused by imperfections in the boundary and loading conditions, is frequently observed in mechanical experiments (de Melo et al., 2020) and has the potential to negatively influence the DIC identifications if not properly considered.

579 7. Conclusions

From previous publications available in the literature, it is clear that refrac-580 tory materials present a different creep behavior under tension and compression. 581 Therefore, there is a need to develop a dedicated model, considering that this 582 feature is not currently available in the main commercial Finite Element Anal-583 ysis software. In the present work, an asymmetric creep model was proposed, 584 that was especially designed to simulate refractory materials at high temper-585 atures. In comparison to previously published works, the proposed model has 586 the advantage to be able to represent the primary and secondary creep behavior 587 of the material, while keeping the possibility to have independent tests for the 588 identification of tension and compression parameters. 589

The main strategy employed in the development of the model was the split of the stress tensor into a positive and a negative part, similar to what have ⁵⁹² been done by Blond et al. (2005), but averaging the contributions of each stress
⁵⁹³ sign by the equivalent stresses. In this case, the model tends to its symmetric
⁵⁹⁴ version when the same material properties are used for both signs.

To identify the material parameters of an alumina-spinel material at 1300 °C, an experimental procedure composed of a Brazilian test in combination with a subset-based DIC technique was developed, that avoids the problems related to contact instrumentation at high temperatures. This technique showed to be successful for the determination of the model parameters.

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Highlights

- A creep model is proposed to represent the asymmetric behavior of refractories.
- The influences of each stress sign are weighted using a split of the stress tensor.
- The model can represent primary and secondary creep strain rates.
- A Brazilian test and a DIC analysis are used in the mechanical characterization.
- A refractory used in steel production is characterized using the proposed method.





(5)

3

Custom made furnace (water cooled)

Ventilator

Blue light source

Speckle pattern at 1300 'C



Digital Image Correlation analysis



-0.25 0.0

0.2 0.4 0.6 0.8

 $\overline{6}$

2

(4)

Load cell

Window

CCD camera 200mm lenses Blue bandpass filter

Numerical Simulation

Conclusion:

In this work, a new creep model to simulate refractory materials was proposed, that can represent the material's asymmetric behavior, i.e., different properties under tension and compression. The model parameter can be fully identified using a Brazilian test in combination with a Digital Image Correlation technique. An experimental setup to acquire high quality images at high temperatures is also proposed.

1.0 1.2

Time [h]

 \mathbf{C}

1.8 2.0

1.4 1.6 **Lucas Teixeira:** Conceptualization, Methodology, Software, Validation, Writing - Original Draft **Jean Gillibert**: Resources, Supervision. **Thomas Sayet**: Resources, Supervision, Writing - Review & Editing . *Eric Blond*: Resources, Supervision, Writing - Review & Editing.

Declaration of interests

 \Box The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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