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► To cite this version:

Jérôme Brulin, Amna Rekik, Laurent Josserand, Eric Blond, Alain Gasser, et al.. Characterization and modelling of a carbon ramming mix used in high-temperature industry. International Journal of Solids and Structures, 2011, 48, pp.854-864. 10.1016/j.ijsolstr.2010.11.024 . hal-00652709

HAL Id: hal-00652709

<https://hal.science/hal-00652709>

Submitted on 16 Dec 2011

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1 Characterization and modelling of a carbon ramming 2 mix used in high-temperature industry

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7 Abstract

This paper is devoted to the modelling of a specific ramming mix mainly used in the high-temperature industry due to its high-compacting behaviour. This material has the ability to absorb the deformation of parts submitted to high thermal loads. Triaxial and instrumented die compaction tests were carried out in order to identify the shear and hardening behaviours, respectively. Tests on the ramming mix were lead for a temperature range between 20°C and 80°C. The temperature effect is particularly observed on the material response when it is compacted. The main features of the behaviour of the ramming mix can be represented by the theoretical framework of the Modified Cam-Clay model. A single variable allows to accurately reproduce the hardening behaviour depending on the temperature. Moreover, an extension of the model for the hardening behaviour at high pressures is proposed. A good agreement between the experimental data and numerical tests is reached with this model.

8 *Keywords:* ramming mix, parameters identification, die compaction test,
9 triaxial test, temperature effect, FE method, Modified Cam-Clay model

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10 1. Introduction

11 The steel industry requires huge structures mainly composed of refractory
12 materials. The thermomechanical properties of these materials suit perfectly
13 high temperature applications. However, the expansion of these constituents
14 can damage some parts of the structure. To avoid this damage, ramming
15 mixes are often used to absorb the deformations. Indeed, these loose ma-
16 terials are well-known for their high-compacting behaviour. An appropriate
17 modelling of this material must at least be able to reproduce the compaction
18 behaviour sensitive to the temperature effect.

19 This paper investigates the case of a ramming mix composed of graphite
20 (80%) and coal tar (20%). Its aspect can be compared with a bituminous
21 sand. In the literature, many studies were carried out in order to predict
22 the behaviour of loose geological materials (Bousshine et al., 2001; Liu and
23 Carter, 2002), pharmaceutical powders (Wu et al., 2005; Han et al., 2008) or
24 metallic powders (Park et al., 1999; Chtourou et al., 2002). The modelling
25 of loose materials whose behaviour is highly dependent on the porosity rate
26 is classically based on micro-mechanical and macro-mechanical approaches.
27 The first one assumes that each particle is a sphere. It takes also into account
28 the contact interactions between the particles (Helle et al., 1985; Fleck et al.,
29 1992; Biba et al., 1993; Fleck, 1995). From a discrete approach, the macro-
30 scopic behaviour of a material can be evaluated by means of homogeniza-
31 tion methods (Piat et al., 2004; Le et al., 2008). Although micro-mechanical
32 models are devoted to understand the physical behaviour of the constituents,
33 macro-mechanical models (Shima and Oyane, 1976; Gurson, 1977; Haggblad,
34 1991) are well-adapted for engineering applications which often occur at a
35 large scale. The phenomenological models were mainly developed for applica-
36 tions in soil mechanics, such as the Drucker-Prager Cap model (Drucker and
37 Prager, 1952), the Modified Cam-Clay model (Roscoe and Burland, 1968)
38 and the Di Maggio-Sandler model (Di Maggio and Sandler, 1971). These
39 Cap models allow to reproduce the hardening behaviour in compaction of
40 initially loose powders. In the same approach, many extensions of these
41 models for porous materials were developed (Chtourou et al., 2002; Aubertin
42 and Li, 2004; Khoei and Azami, 2005; Park, 2007). The Drucker-Prager Cap
43 model remains one of the most used due to its ability to reproduce shearing
44 and compaction behaviours. The parameters of this model are simply iden-
45 tified by carrying classical tests. The hardening behaviour is described by a
46 Cap parameter dependent on the volumetric inelastic strain (Doremus et al.,

2001; Wu et al., 2005). In these studies, Young’s modulus and Poisson’s ratio defining the elastic part are assumed to be constant. In order to reproduce a possible nonlinear elastic behaviour, some authors have chosen to identify elastic and yield surface parameters evolving with the relative density (Kim et al., 2000; Sinka et al., 2003; Michrafy et al., 2004; Han et al., 2008). The influence of the temperature on the parameters of a Drucker-Prager Cap model was rarely taken into account (Piret et al., 2004). The thermal effects were mainly studied in the geotechnical field.

Models inspired on the Modified Cam-Clay model are often used to reproduce the behaviour of soils such as clays (Liu and Carter, 2002; Piccolroaz et al., 2006). It is an elastoplastic model with a yield surface which can grow or shrink according to the hardening rule. Triaxial and die compaction tests are widely used to identify the shearing and hardening behaviours. The hardening behaviour of the material is written in a specific space in which only two constant parameters are used to define nonlinear elastic and plastic parts. Furthermore, this model was extended in order to take into account the temperature effects (Hueckel and Borsetto, 1990; Hueckel and Baldi, 1990; Tanaka et al., 1995; Sultan et al., 2002) which mainly modify the size of the yield surface. In the Modified Cam-Clay model, the size of the yield surface follows the evolution of the yield stress under hydrostatic compression p_c . According to the extensions of the Modified Cam-Clay model, this evolution can be of exponential or polynomial form with various parameters to identify. For these reasons, the Modified Cam-Clay model was chosen in this work in order to reproduce the shearing and hardening behaviours of the ramming mix sensitive to the temperature effect. No tensile strength, nor cohesion are introduced in this model due to the context of the study mainly focused on the compacting behaviour.

This paper is organised as follows. Section 2 presents triaxial and die compaction tests performed on ramming mix samples with cylindrical shape allowing the investigation of the shearing and hardening behaviours at different temperatures. A specific calibration step is carried out to know the radial stress exerted on the sample during the die compaction test. Section 3 describes the adopted Modified Cam-Clay model. Only one parameter is used to modify the size of the yield surface in order to reproduce the temperature effect. An extension of this model to high-compressive pressures is also introduced. The various parameters of this model are identified from triaxial and die compaction tests data. Finally in section 4, the relevance of the Modified Cam-Clay model is first confirmed by comparison of the numerical

85 predictions to experimental data obtained from tests used for the identifi-
 86 cation of the model parameters. Finally, a hydrostatic compression test at
 87 room temperature and a die compaction test with transient heating validate
 88 the ability of this model to reproduce the behaviour of the studied ramming
 89 mix.

90 **2. Characterization of the ramming mix behaviour: triaxial and** 91 **die compaction tests**

92 In the high-temperature industry, the ramming mix is used to protect
 93 the steel shell from the expansion of the refractory bricks. Located between
 94 the shell and the bricks, the ramming mix is not directly subjected to the
 95 thermal load applied on the masonry. It is mainly stressed in compression at
 96 low strain rates for a temperature range between 20°C and 80°C. Triaxial and
 97 die compaction tests are carried out to identify the shearing and hardening
 98 behaviours at different temperatures and strain rates. The three-dimensional
 99 stress state is defined by two invariants: the equivalent pressure stress p and
 100 the von Mises equivalent stress q . They are defined as follows:

$$p = -\frac{1}{3}\text{trace}(\underline{\underline{\sigma}}) \quad (1)$$

101

$$q = \sqrt{\frac{3}{2}\underline{\underline{S}} : \underline{\underline{S}}} \quad (2)$$

102 where $\underline{\underline{S}}$ is the deviatoric part of the stress tensor $\underline{\underline{\sigma}}$, given by:

$$\underline{\underline{S}} = \underline{\underline{\sigma}} + p\underline{\underline{I}} \quad (3)$$

103 $\underline{\underline{I}}$ is the second order identity tensor.

104 In the case of a cylindrical sample submitted to compressive axial and radial
 105 stresses ($\sigma_A < 0$ and $\sigma_R < 0$, respectively), the equivalent pressure stress
 106 reads:

$$p = -\frac{\sigma_A + 2\sigma_R}{3} \quad (4)$$

107 and, the von Mises equivalent stress is:

$$q = |\sigma_A - \sigma_R| \quad (5)$$

2.1. Characterization of the shearing behaviour: triaxial tests

Hereafter, we will describe the testing system required to carry out triaxial tests on the ramming mix. The aim is to characterize the shearing behaviour of this material. Figure 1 depicts the triaxial compression apparatus used to perform the triaxial tests. This apparatus is composed of a cell of a 3.5 MPa capacity, a pressure controller, a 25 mm LVDT sensor and a 10 kN load cell. The cell is filled with water in order to apply a controlled radial stress on the sample. This cell is axially free. The axial displacement of the cell which is measured by the LVDT sensor corresponds to the axial displacement of the lower part of the sample. The upper part of the sample is motionless which yields to the axial compression of the sample. The axial force is measured by the load cell. This testing system ensures that cylindrical samples are submitted to axial loads under a constant radial pressure imposed by the water present inside the cell.

2.1.1. Triaxial test procedure

The tested samples are 50 mm in diameter and 95 mm in height. Each specimen is performed by a manual precompaction of 240 g of carbon ramming mix into a die, up to an average void ratio e of 0.42 ± 0.08 where e is given as follows:

$$e = \frac{\rho_r V}{m} - 1 \quad (6)$$

ρ_r is the real density of the material, m and V are the mass and volume of the sample, respectively. For this material, a gas pycnometer test would be the most appropriate for the measurement of the real density. Here using the Le Chatelier densimeter apparatus, the measured real density is $\rho_r = 1820 \text{ kg/m}^3$. This test consists in the immersion of the ramming mix in oil. Water was avoided to allow a better mixture with the coal tar. With this apparatus, it is impossible to apply a pressure which could favor the filling of all voids by the oil. The grain porosity is the most difficult to fill by the oil. Indeed, a mercury porosimetry on the graphite grains shows that there is a grain microporosity with an average void ratio of $0.02 \mu\text{m}$. The equilibrium of the meniscus of the liquid leads to a simple expression used in porosimetry tests relating the pressure P_l exerted on the liquid to fill the voids and the average diameter d of these voids:

$$P_l = \frac{|4\gamma \cos(\alpha)|}{d} \quad (7)$$

140 where γ is the interfacial tension and α is the wetting angle between the
 141 liquid and the surface of the sample. Considering oil for liquid in the den-
 142 simeter test with $\gamma=0.03$ N/m and $\alpha=30^\circ$, equation (7) shows that a pressure
 143 P_l of 5 MPa is required to fill the grain microporosity. Thereby, for the Le
 144 Chatelier densimeter test only macroporosities are filled and so e denotes a
 145 macro void ratio.
 146 In the case of the triaxial test, the initial macro void ratio of the sample is
 147 heterogeneous due to a manual compaction mode. This sample is set into an
 148 elastomer film to prevent the penetration of water. During the triaxial test,
 149 the sample is first submitted to a constant hydrostatic pressure $p = -\sigma_R$.
 150 Then, the axial displacement of the cell at constant velocity and radial pres-
 151 sure implies the increase of the axial stress on the sample following a stress
 152 path defined by the linear relation $q = 3p$ between the stress invariants.

153 *2.1.2. Triaxial test results: temperature and strain rate effects*

154 At room temperature, triaxial tests were carried out for confining pres-
 155 sures from 100 kPa to 800 kPa with velocities of 0.1 mm/min, 0.5 mm/min
 156 and 1 mm/min. To study the influence of the temperature on the shearing
 157 behaviour, a triaxial apparatus operating at high temperatures as used by
 158 Olmo et al. (1996) is necessary. The triaxial apparatus of this study has no
 159 available heating control, therefore a specific procedure was followed. The
 160 sample is first preheated during two hours, at for example 50°C , it is then set
 161 on the triaxial apparatus. In a second step, the cell is filled with preheated
 162 water at 50°C . In order to minimize the decrease of temperature during the
 163 test, a velocity of 1 mm/min was chosen for the triaxial tests at 50°C and
 164 80°C . The strain rate effect is so not considered at elevated temperatures.
 165 For each triaxial test, a critical shearing stress is reached when the increase
 166 of the von Mises equivalent stress stops. As presented in figure 2 for triaxial
 167 tests with a confining pressure of 200 kPa, there is no effect of the strain rate
 168 at room temperature. Only the temperature influences the critical shearing
 169 stress which decreases.

170 *2.2. Characterization of the hardening behaviour: die compaction tests*

171 A hydrostatic compression test is classically used to identify the hardening
 172 behaviour of porous materials. An oedometer test on a confined cylindrical
 173 sample constitutes also an acceptable solution but requires to be instru-
 174 mented in order to measure both axial and radial stresses applied on the
 175 sample. The axial stress is easy to get due to a load cell located above the

176 sample. The radial stress is applied by the confinement of a hollow cylinder.
 177 In some cases, it is possible to dispose of a load sensor located in the die
 178 in order to have a direct measure (Mesbah et al., 1999; Sinka et al., 2003).
 179 Otherwise, simple relations can be established between the external circum-
 180 ferential strain of the hollow cylinder and the radial stress applied on the
 181 sample. The identification of this calibration relation is detailed hereafter.
 182 An instrumented die compaction test on a static press INSTRON 5800R was
 183 developed as presented in figure 3. Axial displacement and axial load are
 184 acquired with a 50 mm LVDT sensor and a 250 kN load cell, respectively.
 185 Thanks to a heating control system, compaction tests at elevated tempera-
 186 tures are possible. A heating collar with an integrated K-type thermocouple
 187 linked to the thermal control is set around the free cylinder. A water-cooling
 188 system is added between the load cell and the upper piston in order to avoid
 189 the expansion of the load cell during these tests which could affect the mea-
 190 surements. Moreover, a double-effect compaction is chosen (free die in the
 191 axial direction) to compress equally the sample at the top and the bottom.
 192 The die/sample interface is lubricated with a silicon grease to homogenize
 193 the radial stresses applied on the height of the sample. The friction coeffi-
 194 cient between the final compacted sample and the lubricated die was taken
 195 to be 0.22. This value was measured with an inclined plane apparatus. It
 196 represents an estimation of the friction coefficient which evolves (decreases
 197 globally) when the sample is compacted due to the decrease of the roughness
 198 of the lateral area of the ramming mix sample. Finally, 8 circumferential
 199 gauges are set around the die in order to evaluate its average circumferen-
 200 tial strain. These gauges are located at mid-height of the die with 70 mm
 201 between the lowest and the highest ones. They allow to record strains of the
 202 die for samples higher than 70 mm.

203 *2.2.1. Calibration test on a rubbery sample*

204 The calibration relation can be identified experimentally from a die com-
 205 paction test on an incompressible rubbery sample (Mosbah et al., 1997; Gein-
 206 dreau et al., 1999; Hong et al., 2008; Michrafy et al., 2009) or numerically
 207 when the circumferential strain of the hollow cylinder is known (Bier et al.,
 208 2007). In this study, we choose to carry out a calibration test with an in-
 209 compressible sample. It is worth noting that for an incompressible rubbery
 210 sample submitted to a confined load, there is an equality between the axial
 211 and radial stresses. In this case, it is possible to identify a linear relation
 212 between the applied hydrostatic pressure P_h and the average of the circum-

ferential strains of the die $\varepsilon_{\theta\theta}^{die}$ measured by gauges as follows:

$$C(H_s) = \frac{P_h}{\varepsilon_{\theta\theta}^{die}} \quad (8)$$

H_s is the height of the sample. $C(H_s)$ is the function to identify which can depend on the height of the sample. Figure 4 illustrates the evolution of $\frac{P_h}{\varepsilon_{\theta\theta}^{die}}$ as a function of the height of the sample. Eight die compaction tests show a constant value for the C parameter when the contact between the die and the sample is established. These tests carried out on rubbery samples of 102 mm in height (6 tests) and 92 mm in height (2 tests) allow to identify the following value for C :

$$C_{test} = 51 \text{ GPa} \quad (9)$$

This experimental value can be compared with a theoretical one obtained for the case of a hollow cylinder submitted to an internal pressure p_1 . This cylinder has a height L , an internal and an external radius R_1 and R_2 , respectively. The behaviour of the cylinder is assumed to be linear elastic with a Young's modulus E and a Poisson's ratio ν . For this cylinder, the local stress field (Salençon, 2007) is given by:

$$\sigma_{rr} = \frac{p_1 R_1^2}{R_2^2 - R_1^2} \left(1 - \left(\frac{R_2}{r} \right)^2 \right) \quad (10)$$

$$\sigma_{\theta\theta} = \frac{p_1 R_1^2}{R_2^2 - R_1^2} \left(1 + \left(\frac{R_2}{r} \right)^2 \right) \quad (11)$$

where r is the local radius ($R_1 \leq r \leq R_2$). Then, taking into account the boundary conditions:

$$\sigma_{rr}(r = R_2) = 0 \quad (12)$$

$$\sigma_{\theta\theta}(r = R_2) = \frac{2p_1 R_1^2}{R_2^2 - R_1^2} \quad (13)$$

and the constitutive local Hooke's law:

$$\varepsilon_{\theta\theta} = \frac{1}{E} \sigma_{\theta\theta} - \frac{\nu}{E} (\sigma_{rr} + \sigma_{zz}) \quad (14)$$

232 it is possible to deduce from equation (14) the expression of $\sigma_{\theta\theta}(r = R_2)$ as
 233 follows:

$$\sigma_{\theta\theta}(r = R_2) = E\varepsilon_{\theta\theta}(r = R_2) + \nu\sigma_{zz}(r = R_2) \quad (15)$$

234 The friction between the sample and the cylinder induces a shear stress at
 235 the sample/cylinder interface and consequently an axial stress on the cylinder
 236 which is not taken into account in the theoretical result. For the carried tests,
 237 the upper and lower sections of the steel cylinder are free and thus (Salençon,
 238 2007):

$$\sigma_{zz}(r = R_2) = 0 \quad (16)$$

239 Equation (15) reads then:

$$\sigma_{\theta\theta}(r = R_2) = E\varepsilon_{\theta\theta}(r = R_2) \quad (17)$$

240 Finally, combining equations (13) and (17), we deduce a theoretical estima-
 241 tion for the C parameter:

$$C_{theory} = \frac{p_1}{\varepsilon_{\theta\theta}(r = R_2)} = \frac{E}{2} \left(\left(\frac{R_2}{R_1} \right)^2 - 1 \right) \quad (18)$$

242 For the considered die: $R_1 = 40.6$ mm, $R_2 = 50$ mm and $E = 210$ GPa.
 243 From equation (18), the theoretical constant is set to:

$$C_{theory} = 54 \text{ GPa} \quad (19)$$

244 The 6% deviation with the experimental result is due to the neglected friction.
 245 The above theoretical study allows to validate the identified experimental
 246 value even if it neglects the friction at the sample/cylinder interface. As a
 247 conclusion, this calibration step provides an estimation for the average radial
 248 stress ($\sigma_{rr} = C_{test} \varepsilon_{\theta\theta}^{die}$) of the ramming mix sample during a die compaction
 249 test.

250 2.2.2. Die compaction tests at different temperatures and strain rates

251 For the compaction tests, 700 g of carbon ramming mix are poured into
 252 a die and precompacted with the apparatus up to an initial macro void ratio
 253 of 0.37 ± 0.08 . The precompaction step induces an axial force close to 1 kN
 254 and a radial stress allowing to maintain the mid-height of the free cylinder

aligned with the mid-height of the initial sample. The initial sample is 81 mm in diameter and 102 mm in height which gives an aspect ratio higher than 1 as mentioned in Doremus et al. (2001) allowing to minimize the stress gradients in the sample. This aspect ratio and the double-effect compaction system allow to assume that the initial macro void ratio is homogeneous. For tests at elevated temperature, strains are recorded during heating and dwell time in order to take the expansion into account. Load/unload cycles are programmed to identify both elastic and plastic parts of the hardening behaviour. Results of these cycles at different temperatures (20°C, 50°C and 80°C) and velocities (0.1 mm/min and 1 mm/min) are presented in figure 5. The two curves corresponding to the tests at room temperature at rates of 0.1 mm/min and 1 mm/min are very close. In the same way, the two curves at a temperature of 50°C merge. The strain rate has no effect on the hardening behaviour. The repeatability of the die compaction test is checked at 80°C and 1 mm/min. It is observed that only the temperature influences the hardening behaviour. For the same consolidation state, the axial load decreases with respect to the temperature. This phenomenon is observed in figure 6 where the hardening behaviour with heating is compared with the one at room temperature. For this test carried out under a velocity of 0.4 mm/min, the sample has an initial macro void ratio of 0.34 ± 0.08 . During this test, a first heating from 20°C to 50°C and a second one from 50°C to 80°C were imposed at 3°C/h on the external part of the steel cylinder. The viscous part of the ramming mix induces a slight softening of the axial stress with the increase of the temperature during a die compaction test.

2.2.3. Die compaction tests at high pressures

Figure 7 presents the results of a die compaction test carried out up to high pressures (40 MPa) at room temperature. The first part of the curve showing the "load-displacement" diagram is similar to the plastic part of the hardening behaviour at room temperature presented in figure 5. In fact, until an axial displacement of 27 mm, the macro void ratio is positive and all macro voids are progressively filled. Afterwards due to pressures higher than 4 MPa, the microporosity of the graphite grains is filled by the coal tar. Considering equation (7) with $\gamma=0.03$ N/m and $\alpha=130^\circ$ which are the parameters for a bituminous, a pressure close to 4 MPa is obtained in order to fill the microporosity of the graphite grains. The macro void ratio is negative because the real density does not account for this microporosity.

291

292 As done for the Modified Cam-Clay model, the hardening behaviour is
 293 written in the $(\ln(p), e)$ plane. In figure 8 are depicted the results of the
 294 die compaction tests carried out at room temperature under low and high
 295 pressures. It allows to illustrate the ability of a compaction law with a sole
 296 elastic slope and two plastic slopes to describe the hardening behaviour of the
 297 ramming mix. The formulation of an extended Modified Cam-Clay model is
 298 presented in the following section.

299 **3. Modelling of the ramming mix**

300 *3.1. Modified Cam-Clay formulation*

301 The Modified Cam-Clay model, which is an extension of the original
 302 model developed by Roscoe and Burland (1968) introducing the critical state
 303 concept, is relevant to reproduce the elastic and plastic parts of the hardening
 304 behaviour of the ramming mix. It is also able to take into account the
 305 temperature effect. The proposed model is based on the Modified Cam-
 306 Clay model implemented in the finite element software ABAQUS/Standard
 307 (Abaqus, 2007).

308 *3.1.1. Yield surface of the model*

309 The Modified Cam-Clay model described in figure 9 is based on a yield
 310 surface with an associated flow rule:

$$F = \frac{1}{\beta^2} \left(\frac{p}{a} - 1 \right)^2 + \left(\frac{q}{Ma} \right)^2 - 1 = 0 \quad (20)$$

311 where M is a constant that defines the slope of the critical state line, a is
 312 the size of the yield surface and β is a constant which can modify the shape
 313 of the cap. In this work, this parameter is used to take into account the
 314 temperature effect on the yield surface of the hardening behaviour. It is
 315 worth noting that for a hydrostatic compression p_c , the yield stress is given
 316 by:

$$p_c(T) = a(1 + \beta(T)) \quad (21)$$

317 The yield surface (equation (20)) is written in terms of two stress invariants:
 318 the equivalent pressure stress p and the von Mises equivalent stress q . In this
 319 section, we introduce some notations. The strain tensor $\underline{\underline{\varepsilon}}$ is decomposed in
 320 two parts as follows:

$$\underline{\underline{\varepsilon}} = \underline{\underline{\varepsilon}}^{el} + \underline{\underline{\varepsilon}}^{pl} \quad (22)$$

321 $\underline{\underline{\varepsilon}}^{el}$ denotes the elastic part of the strain field and $\underline{\underline{\varepsilon}}^{pl}$ represents its plastic
 322 part. Each of these strains can be divided into deviatoric and volumetric
 323 parts. The strain field (equation (22)) reads then:

$$\underline{\underline{\varepsilon}} = (\underline{\underline{\varepsilon}}_{dev}^{el} + \underline{\underline{\varepsilon}}_{dev}^{pl}) - (\varepsilon_{vol}^{el} + \varepsilon_{vol}^{pl})\underline{\underline{I}} \quad (23)$$

324 Moreover, the volumetric part of the strain field ε_{vol} can be expressed as a
 325 function of the macro void ratio e and the initial macro void ratio e_0 :

$$\varepsilon_{vol} = \varepsilon_{vol}^{el} + \varepsilon_{vol}^{pl} = \ln \left(\frac{1+e}{1+e_0} \right) \quad (24)$$

326 The strain field evolves then following a hardening behaviour for high p values
 327 and a shearing behaviour for high q values.

328 3.1.2. *Shearing behaviour*

329 The critical state line is the key point of the model because it allows to
 330 define hardening and softening behaviours of the material. In fact, when
 331 submitted to an increasing deviatoric stress for a constant value of p lower
 332 than a , the material exhibits a deviatoric elastic behaviour defined by:

$$d\underline{\underline{S}} = 2Gd\underline{\underline{\varepsilon}}_{dev}^{el} \quad (25)$$

333 where G is the shear modulus whose expression is defined by:

$$G = \frac{3(1-2\nu)(1+e_0)}{2(1+\nu)\kappa} p \exp(\varepsilon_{vol}^{el}) \quad (26)$$

334 κ is the logarithmic bulk modulus. Equation (26) demonstrates that G in-
 335 creases with the compaction rate of the material. Then, the yield surface is
 336 reached for values of q upper than the product Mp leading to a softening
 337 behaviour. The yield stress curve decreases and reaches the critical state
 338 line.

339 3.1.3. *Hardening behaviour*

340 When submitted to hydrostatic pressures, the elastic part of the material
 341 is governed by its volumetric behaviour:

$$\exp(\varepsilon_{vol}^{el}) = 1 + \frac{\kappa}{1+e_0} \ln \frac{p_0}{p} \quad (27)$$

where p_0 is the initial value of the equivalent pressure stress. If yielding occurs, the material exhibits a hardening behaviour described in figure 10 and defined by:

$$de = -\lambda d(\ln p) \quad (28)$$

λ is the logarithmic hardening constant for the plasticity. Hereafter, this slope takes the value of λ_1 for positive macro void ratios and λ_2 for negative macro void ratios. The hardening behaviour was implemented in the FEA code ABAQUS thanks to a user defined field (USDFLD) subroutine which computes the macro void ratio of each element constituting the mesh. Then, two specific logarithmic hardening constants function of the macro void ratio are defined.

The hardening behaviour is characterized by the growth of the yield surface size defined by:

$$a = a_0 \exp \left[(1 + e_0) \frac{1 - \exp(\varepsilon_{vol}^{pl})}{\lambda - \kappa \exp(\varepsilon_{vol}^{pl})} \right] \quad (29)$$

where a_0 is the initial position of a . This initial overconsolidation is computed as follows:

$$a_0 = \frac{1}{2} \exp \left(\frac{e_1 - e_0 - \kappa \ln p_0}{\lambda - \kappa} \right) \quad (30)$$

e_1 denotes the macro void ratio of the ramming mix for a hydrostatic pressure of 1 MPa. If the critical state line is reached after the yielding, the material then distorts without any changes in shear stress or volume.

Finally, the use of the Modified Cam-Clay model requires 9 parameters:

- ν and κ for the elastic behaviour,
- λ_1 and λ_2 for the plastic behaviour,
- M for the critical state line,
- β for the yield surface evolving with the temperature,
- e_0 , e_1 and p_0 defining the initial state of the material.

3.2. Parameters identification

For different temperatures, the shearing parameter M is identified from triaxial tests described in section 2.1.2. Results of the die compaction tests (section 2.2.2), written in the $(\ln(p), e)$ plane, allow to obtain the parameters of the hardening behaviour. The temperature effect on the parameters of this model is particularly observed.

3.2.1. The critical state line

From the results of the triaxial tests mentioned in section 2.1.2, the critical shearing stresses permit to plot the critical state line. Considering no effect of the velocity, a single value of M was identified at room temperature: $M_{20^\circ C} = 1.18$ as presented in figure 11. Results of tests carried out at $50^\circ C$ and $80^\circ C$ are depicted in the same figure. All of them are located under the critical state line identified at room temperature. Since there is no thermal control during these tests, we can only conclude the global influence of the temperature on the slope of the critical state line but nothing on the real value of the parameter M . For this reason, a slight decrease of the slope was assumed for this range of temperatures: $M_{50-80^\circ C} = 1.1$.

3.2.2. Parameters of the hardening behaviour

Parameters identification is done in the $(\ln(p), e)$ plane as previously explained. Results of the carried die compaction tests at different temperatures are exploited as in figure 8. The parameters of the hardening behaviour are reported in table 1. Temperature has no effect on the elastic and plastic slopes. As shown in figure 12, only the parameter e_1 decreases with the increase of the temperature. It means that for the same hydrostatic pressure (1 MPa), the sample is more compacted at $80^\circ C$ than at $20^\circ C$. This result is directly related to a phenomenon often observed (Hueckel and Baldi, 1990; Sultan et al., 2002): the decrease of the yield surface with respect to the temperature. As written in equation (30), e_1 is only used to compute the initial consolidation state at the corresponding temperature. In this paper, the temperature effect is reproduced with the parameter β which can modify the shape of the cap part. A unit value is set at room temperature: $\beta(20^\circ C) = 1$. In order to assess $\beta(50^\circ C)$ and $\beta(80^\circ C)$, we consider a hydrostatically compacted sample heated from the room temperature up to a temperature T without any volume change. It means that during this heating step the consolidation state a and the void ratio e remain constant. From equation (21),

400 we can write:

$$\beta(T) = \frac{p_c(T)}{a(T)} - 1 \quad (31)$$

401 At room temperature:

$$\beta(20^\circ C) = \frac{p_c(20^\circ C)}{a(20^\circ C)} - 1 = 1 \quad (32)$$

402 Considering the constant consolidation state, it comes at higher tempera-
403 tures:

$$\beta(T) = \frac{p_c(T)}{a(20^\circ C)} - 1 = 2 \frac{p_c(T)}{p_c(20^\circ C)} - 1 \quad (33)$$

404 According to equation (28) and the parameters e_1 , λ_1 , λ_2 reported in table 1,
405 it is possible to compute the values $p_c(50^\circ C)$ and $p_c(80^\circ C)$ from a chosen
406 value of $p_c(20^\circ C)$ after heating without any volume change. For $T = 50^\circ C$
407 and $T = 80^\circ C$, equation (33) leads to the following values:

$$\beta(50^\circ C) = 0.56 \quad \text{and} \quad \beta(80^\circ C) = 0.21 \quad (34)$$

408 All the parameters needed for the computation of the Modified Cam-Clay
409 model for the ramming mix are summarized in table 2. The Poisson's ratio
410 ν is set equal to 0.25. The obtained results are in agreement with the ten-
411 dencies of the evolution of the Cam-Clay parameters with the temperature
412 as mentioned in Graham et al. (2001). In fact, heating produces variations
413 of the yield surface size leading to a decrease of the elastic zone.

414 4. Numerical results

415 An axisymmetric numerical analysis of triaxial and die compaction tests
416 used for parameters identification is carried out with the finite element code
417 ABAQUS/Standard 6.7. Two additional tests independent from the pre-
418 vious ones are also modelled in order to validate the model. For all these
419 simulations, the ramming mix behaviour is computed with the parameters
420 previously identified.

421 4.1. Modelling of identification tests

422 To simulate triaxial and compaction tests, the ramming mix sample is
423 meshed with four-node axisymmetric continuum elements. Upper and lower
424 pistons are modelled with rigid supports.

4.1.1. Triaxial tests

For the triaxial test, a hard-contact condition in normal behaviour and a friction coefficient of 0.3 (measured with an inclined plane apparatus between the filter paper and the ramming mix) in tangential behaviour are defined between the sample and pistons. Due to the manual compaction mode, an initial heterogeneous macro void ratio is considered for the sample with an average value of 0.42. For all samples, the heterogeneous distribution of the macro void ratio is assumed to be linear with $e = 0.52$ at the bottom of the sample and $e = 0.32$ at its top. In fact, the initial sample is more compacted in its upper part in order to respect the chosen initial height. This distribution was chosen due to its efficiency to reproduce accurately the shearing behaviour under a radial stress of 100 kPa. As shown in figure 13(a), the sample is first hydrostatically stressed on its upper and lateral areas. Then, an axial displacement is imposed on the lower piston. The axial load is read on the upper piston which is locked. This load corresponds to the one measured by the load cell in figure 1. In fact for the simulation, the axial pressure exerted by water is directly applied on the sample and not on the upper piston. When compacted, the sample takes a convex shape (see figure 13(b)) and is mainly stressed in the center and the corners. Numerical and experimental results compare the evolution of the axial load with respect to the axial displacement. They are shown in figure 14 at room temperature and in figure 15 at higher temperatures (50°C and 80°C). A good agreement is reached between tests and simulations for the shearing behaviour.

4.1.2. Die compaction tests

The die compaction test modelling is reported in figure 16(a), the steel die is meshed with four-node axisymmetric continuum elements. Its behaviour is assumed to be isotropic elastic linear with a Young's modulus $E_{die} = 210$ GPa and a Poisson's ratio $\nu_{die} = 0.3$. Contact conditions are defined between the steel parts and the sample: a hard-contact condition in normal behaviour and a friction coefficient of 0.22 in tangential behaviour as explained in section 2.2. Due to the double-effect precompaction step, a homogeneous macro void ratio of 0.37 describes the initial state of the sample. It is submitted to an axial displacement imposed by the lower piston. The upper piston is locked and provides the axial load applied on the sample. As shown in figure 16(b), the radial stress distribution in the sample is globally homogeneous. Moreover, the steel die which axially moves up due to the friction, is radially stressed in compression where it is in contact with the sample and in

462 tensile elsewhere. The axial load response to the axial imposed displacement
 463 is studied. As shown in figure 17, elastic and plastic parts of the hardening
 464 behaviour accurately fit with the proposed model at room temperature. The
 465 same agreement between experimental and numerical results is obtained as
 466 well at 50°C and 80°C. Figure 18 shows the necessity to allow for a second
 467 plastic slope λ_2 to define the hardening behaviour at high pressures. In order
 468 to avoid numerical problems, a sweet transition is defined between the two
 469 plastic slopes.

470 *4.2. Validation of the model*

471 *4.2.1. Die compaction test with loads/unloads and heating*

472 A die compaction test with load/unload cycles and heating during the
 473 test is first used for the validation of the model. A sample with an initial
 474 homogeneous macro void ratio of 0.37 is submitted to a load/unload cycle
 475 at a velocity of 0.2 mm/min and an increase of the temperature in two
 476 steps: first from 20°C to 50°C and then from 50°C to 80°C as shown in
 477 figure 19. The thermal field was measured by a thermocouple during the
 478 test and applied in the simulation on the external part of the free cylinder.
 479 Thanks to a preliminary thermal computation in transient state with the
 480 steel and ramming mix thermal properties defined in table 3, the evolution
 481 of the temperature in the ramming mix sample is known. Numerical and
 482 experimental results for this test are depicted in figure 20. The efficiency of
 483 the model to reproduce accurately the hardening behaviour of the material
 484 evolving with the temperature is validated.

485 *4.2.2. Hydrostatic compression test*

486 A second test using the triaxial apparatus is developed as well. The idea
 487 is to carry out a hydrostatic compression test with loads and unloads, with
 488 the only use of the pressure exerted by water. For the ramming mix sam-
 489 ple preparation, a similar procedure to that described for triaxial tests was
 490 adopted. As shown in figure 21, the cell is axially locked like the upper piston
 491 which has no contact with the sample. So no axial displacement is applied
 492 on the sample and no axial load is measured by the load cell. The sample is
 493 put on the lower locked piston and a cylindrical disk is set above in order to
 494 attach the elastomer film. The sample is then hydrostatically stressed on the
 495 lateral and upper areas. In order to evaluate the distortion of the sample,
 496 the axial displacement of the center of the cylindrical disk above the sample

497 is investigated. The sample being in the cell filled with water, a direct mea-
 498 surement is impossible. For this reason a marker tracking method (Nugent
 499 et al., 2000; Lee et al., 2009) is used. Markers are drawn on the cylindrical
 500 disk. A 8-bit CCD camera allows to grab images of the marked area for each
 501 hydrostatic stress increment of 50 kPa. The displacement field of the center
 502 of the cylindrical disk can be computed. We assume that this displacement
 503 corresponds to the one of the upper part of the sample. Figure 22 shows
 504 the experimental and numerical evolutions of the axial displacement of the
 505 upper part of the sample with respect to the hydrostatic stress. Unlike the
 506 plastic part which is well-reproduced, the elastic part does not seem to be ac-
 507 curately reproduced by the model. It can be explained by the loss of contact
 508 between the disk and the sample during the unload step. Indeed during the
 509 unload, a pump-effect leads to the filling of the space between the cylindrical
 510 disk and the sample with air as shown in figure 23. Accordingly, the axial
 511 displacement of the cylindrical disk differs from the axial displacement of the
 512 upper part of the sample at the end of the unload. During the reload, the
 513 air is first evacuated and then the sample is loaded. This phenomenon is
 514 not numerically taken into account which leads to slight differences for the
 515 elastic part.

516 5. Conclusion

517 In the present paper, a specific ramming mix of the steel industry was
 518 studied in order to select an appropriate model reproducing its behaviour.
 519 A macroscopic model which is well adapted to engineering applications was
 520 retained: the Modified Cam-Clay model which is often used in the geotechni-
 521 cal field. It is known for its ability to reproduce the shearing and hardening
 522 behaviours. In the proposed model, a single parameter β permits to take
 523 into account the temperature effect on the hardening behaviour. Moreover,
 524 the hardening behaviour was completed for high pressures defining a second
 525 plastic slope. This second slope is related to the microporosity filling. An
 526 experimental campaign allowed to determine the material parameters of the
 527 proposed model. Triaxial tests allowed to identify the slope of the critical
 528 state line of the model and an instrumented die compaction test was con-
 529 ceived to characterize the hardening behaviour. For all these tests, no influ-
 530 ence of the strain rate was established for strain rates lower than $2 \times 10^{-4} \text{ s}^{-1}$.
 531 Only the temperature effect on the critical state line and the yield surface
 532 was clearly observed. In fact, the size of the yield surface decreases while

533 the temperature increases. In order to validate the well agreement between
534 experimental and numerical results, two additional tests were carried out.
535 A die compaction test with heating and a hydrostatic compression test at
536 room temperature coupled with a marker tracking method have confirmed
537 the relevance of the model to reproduce the behaviour of the ramming mix.

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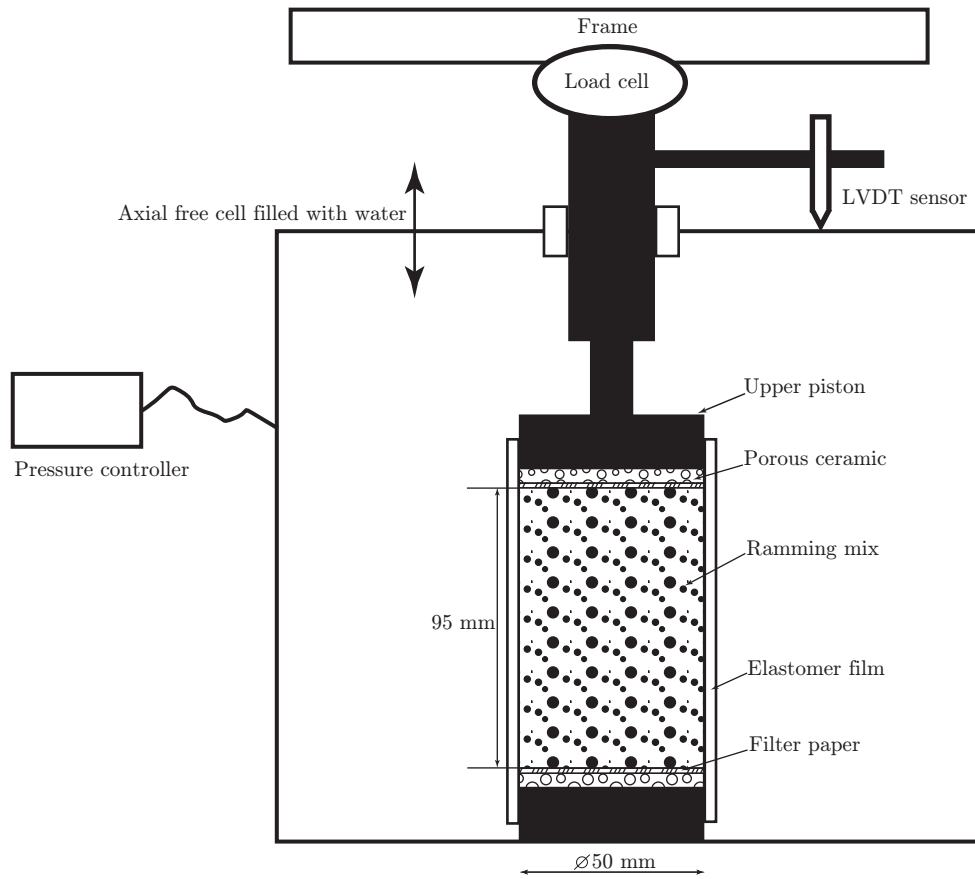


Figure 1: Triaxial compression test.

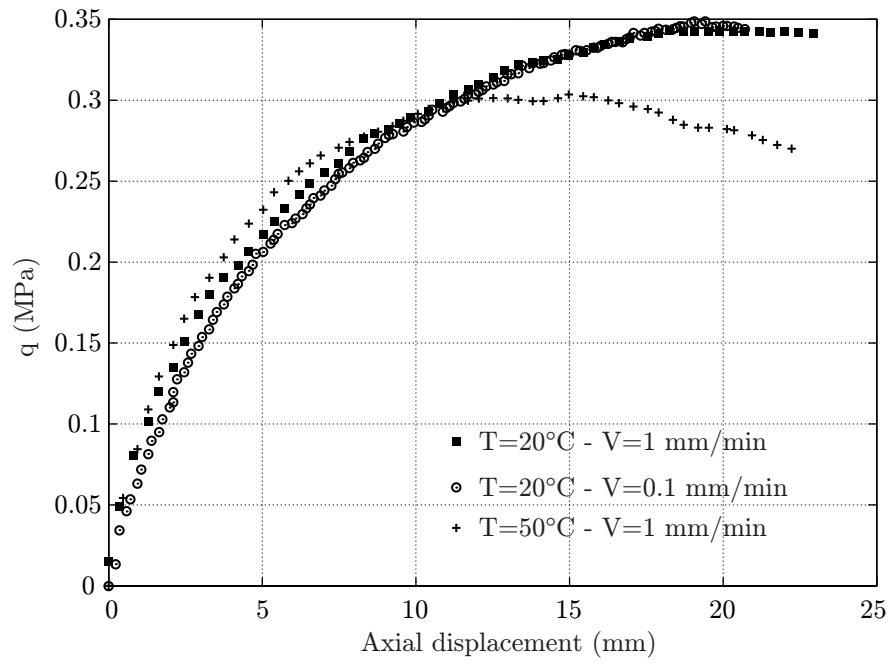


Figure 2: Triaxial tests with a confining pressure of 200 kPa at different temperatures and velocities.

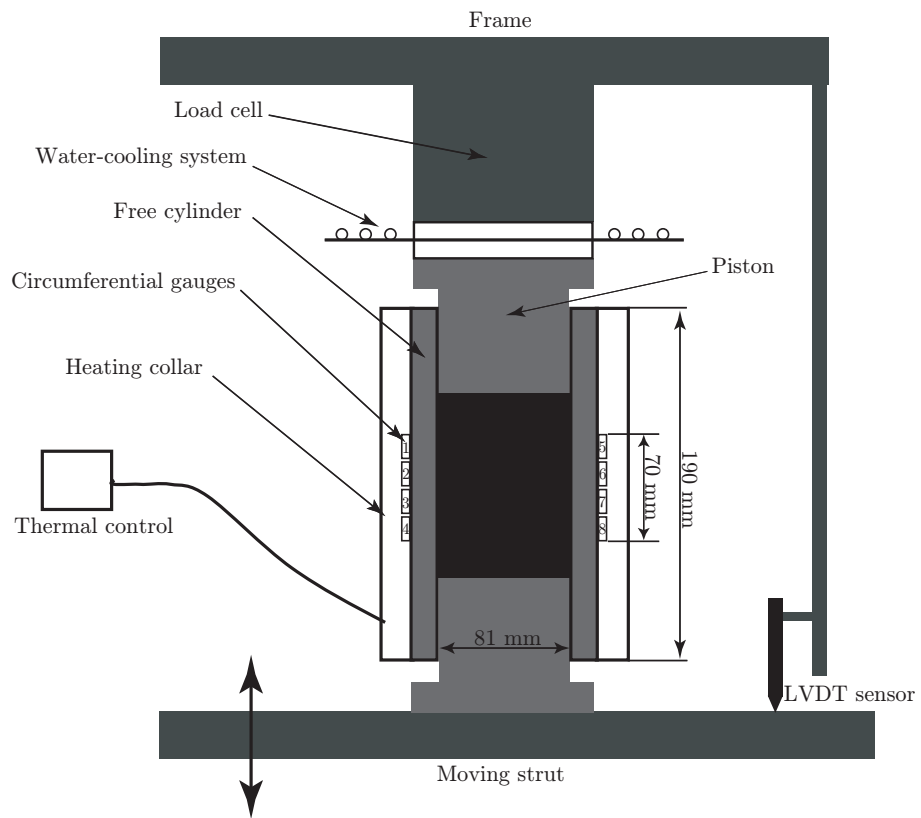


Figure 3: Instrumented die compaction test.

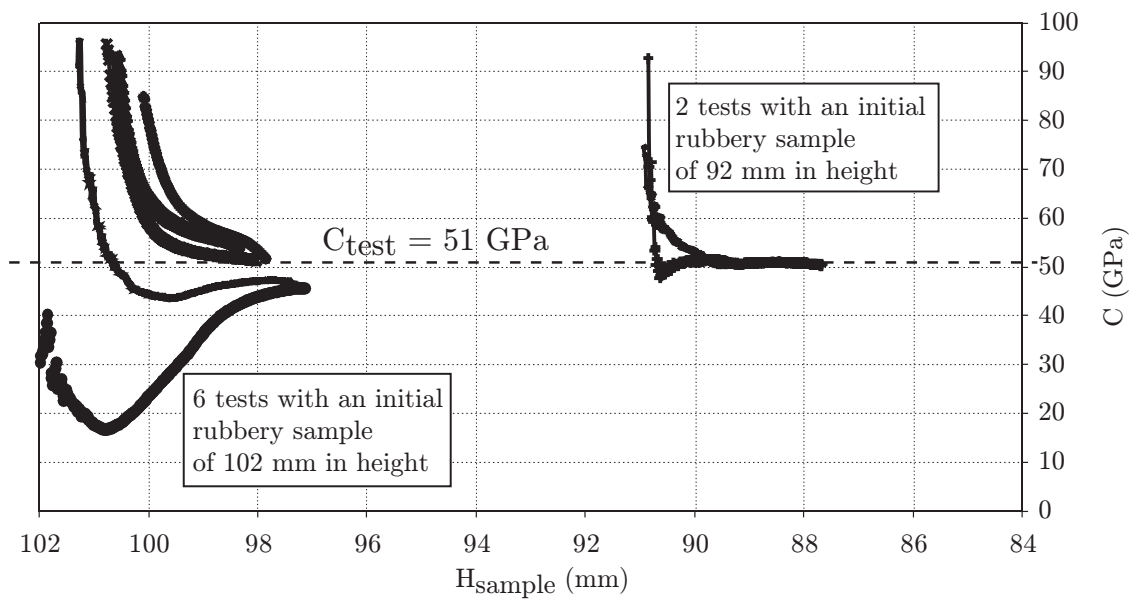


Figure 4: Identification of the calibration relation.

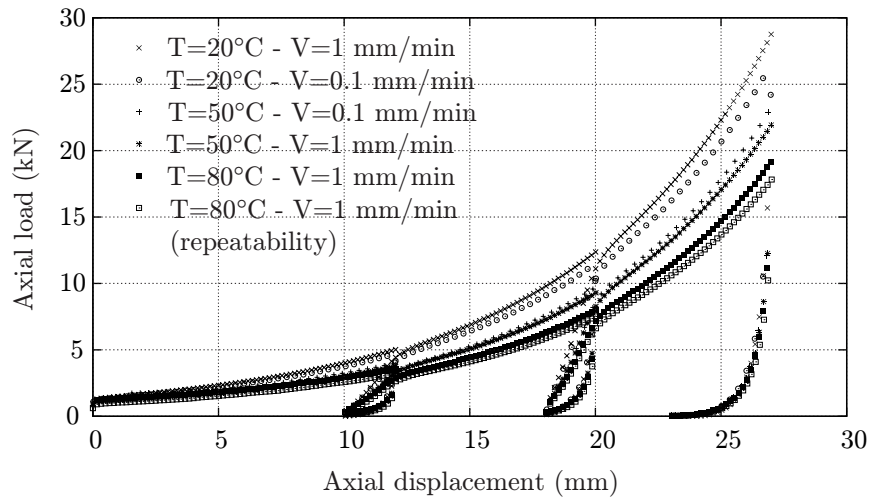


Figure 5: Results of die compaction tests at different velocities and temperatures.

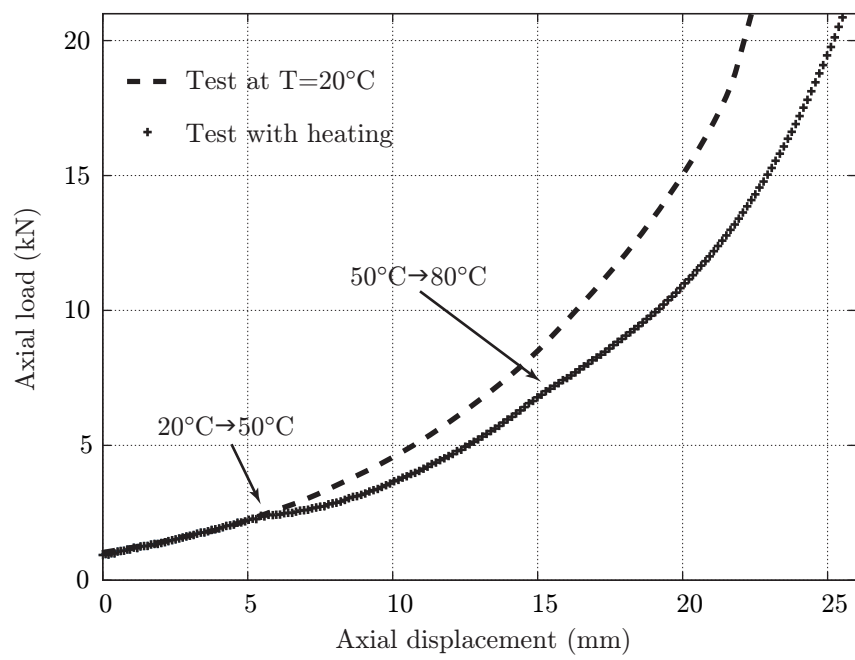


Figure 6: Temperature effect on the hardening behaviour.

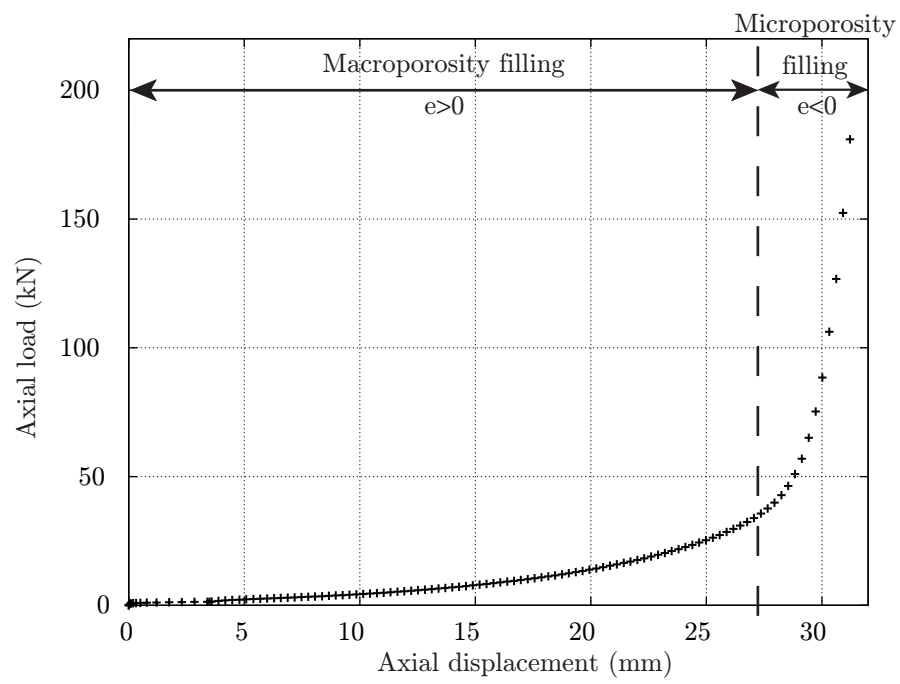


Figure 7: Die compaction test under high pressures.

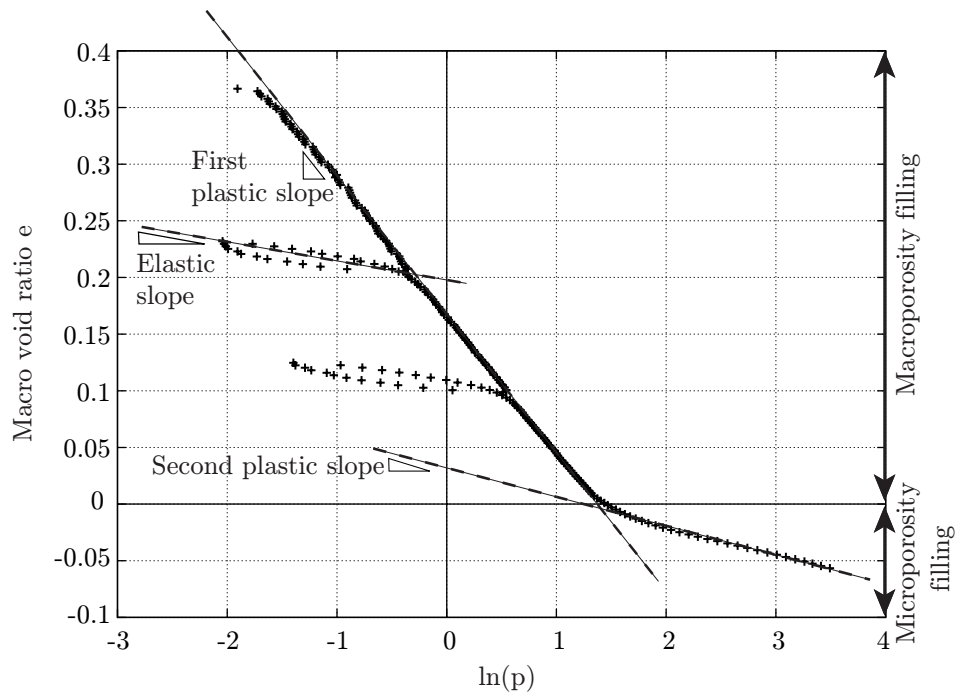


Figure 8: Hardening behaviour in the $(\ln(p), e)$ plane.

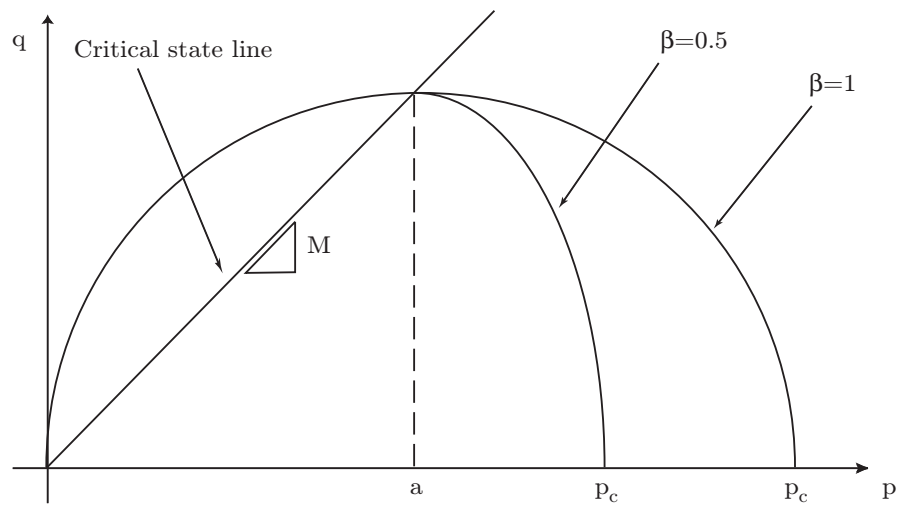


Figure 9: Yield surface and critical state line of the Modified Cam-Clay model.

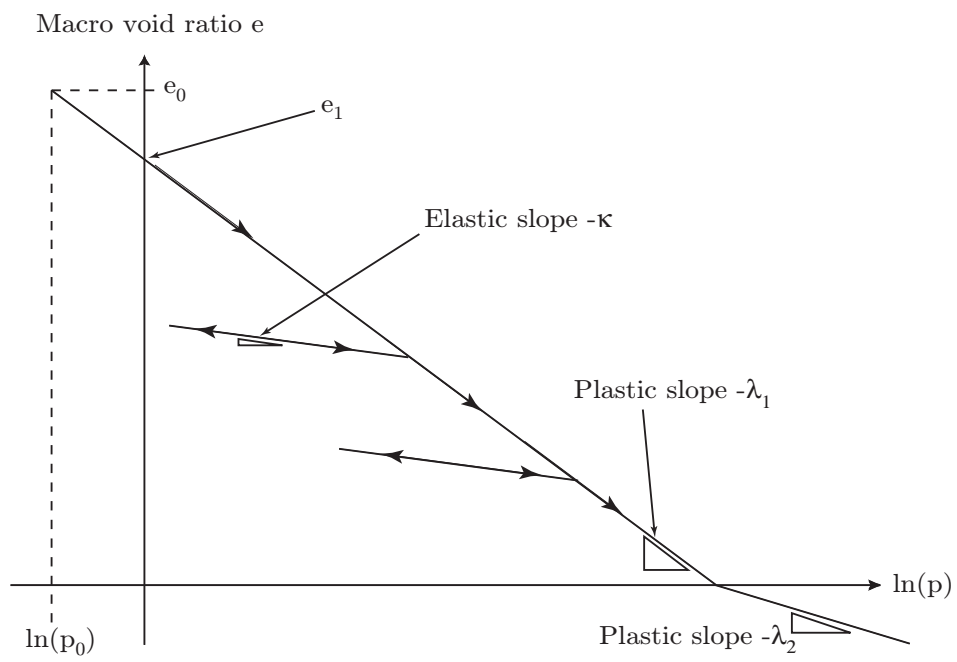


Figure 10: Compaction behaviour of the Modified Cam-Clay model.

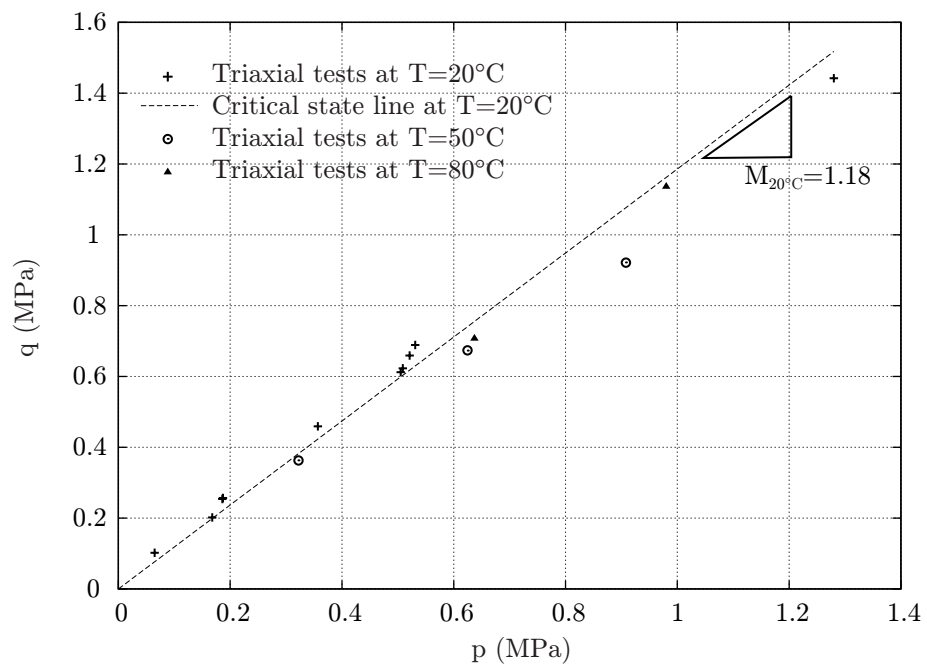


Figure 11: Results of triaxial tests at 3 different temperatures.

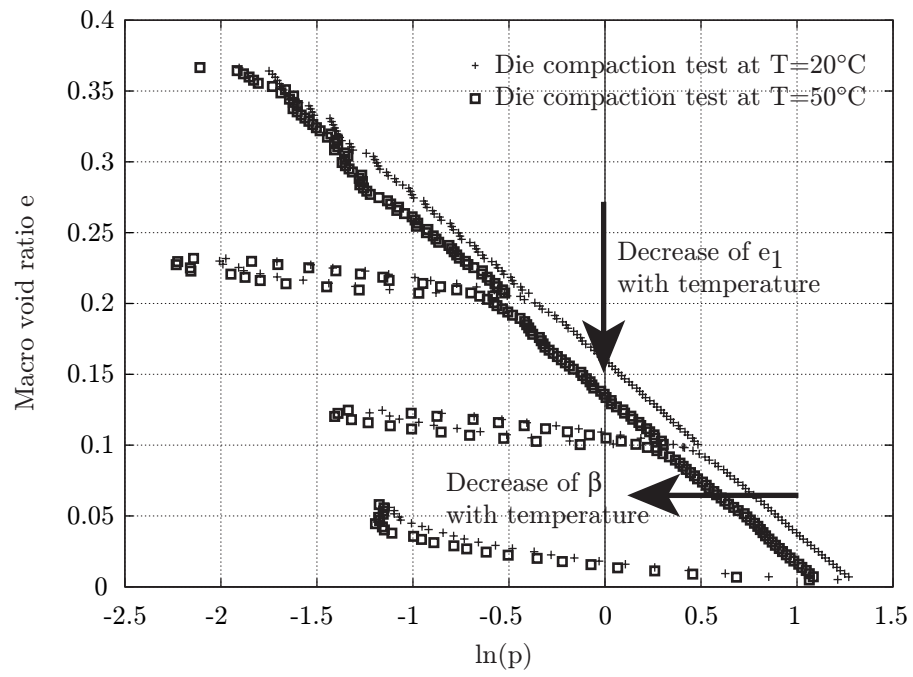


Figure 12: Influence of the temperature on the hardening behaviour.

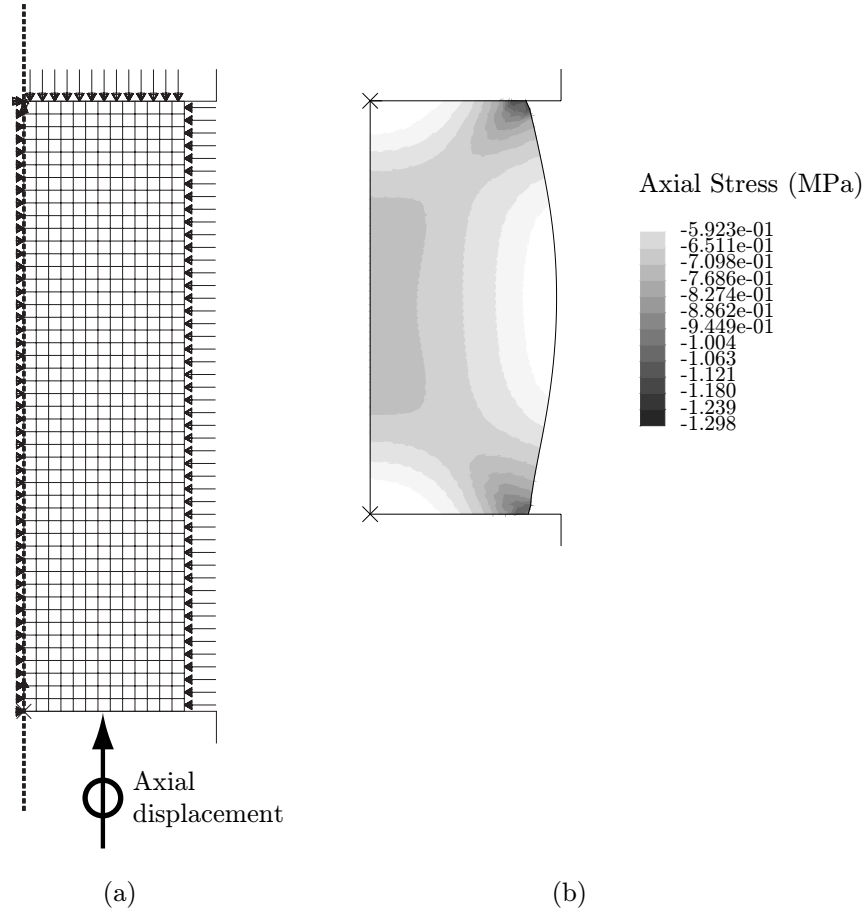


Figure 13: Simulation of the triaxial test: (a) Mesh and boundary conditions, (b) Axial stress distribution.

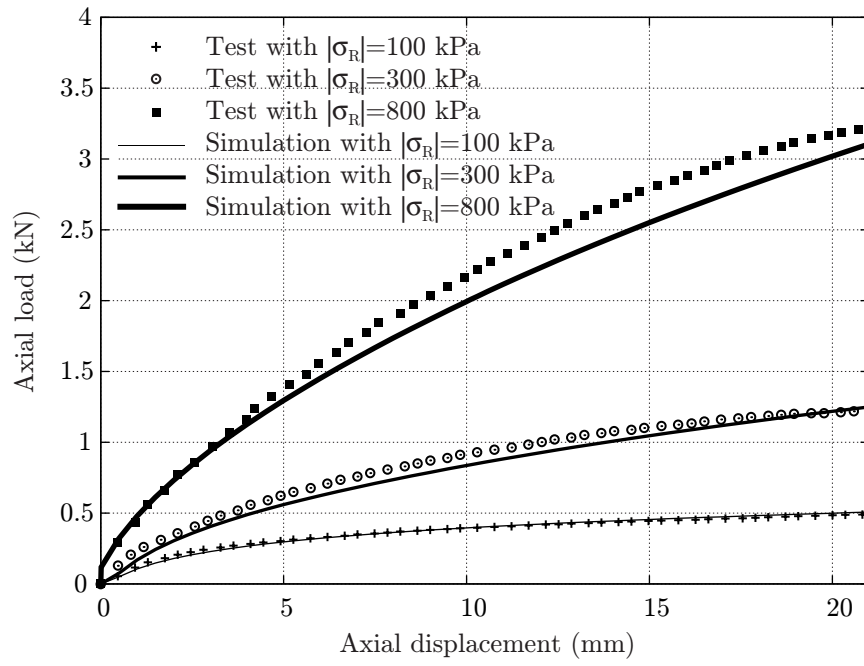


Figure 14: Triaxial tests with different radial stresses at $T=20^{\circ}\text{C}$.

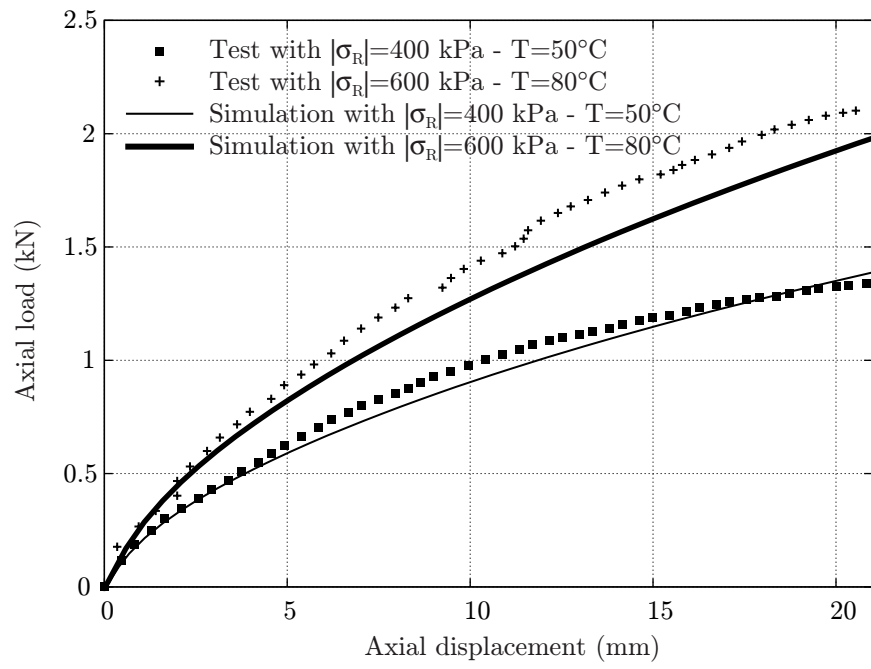


Figure 15: Triaxial tests at $T=50^\circ\text{C}$ and $T=80^\circ\text{C}$.

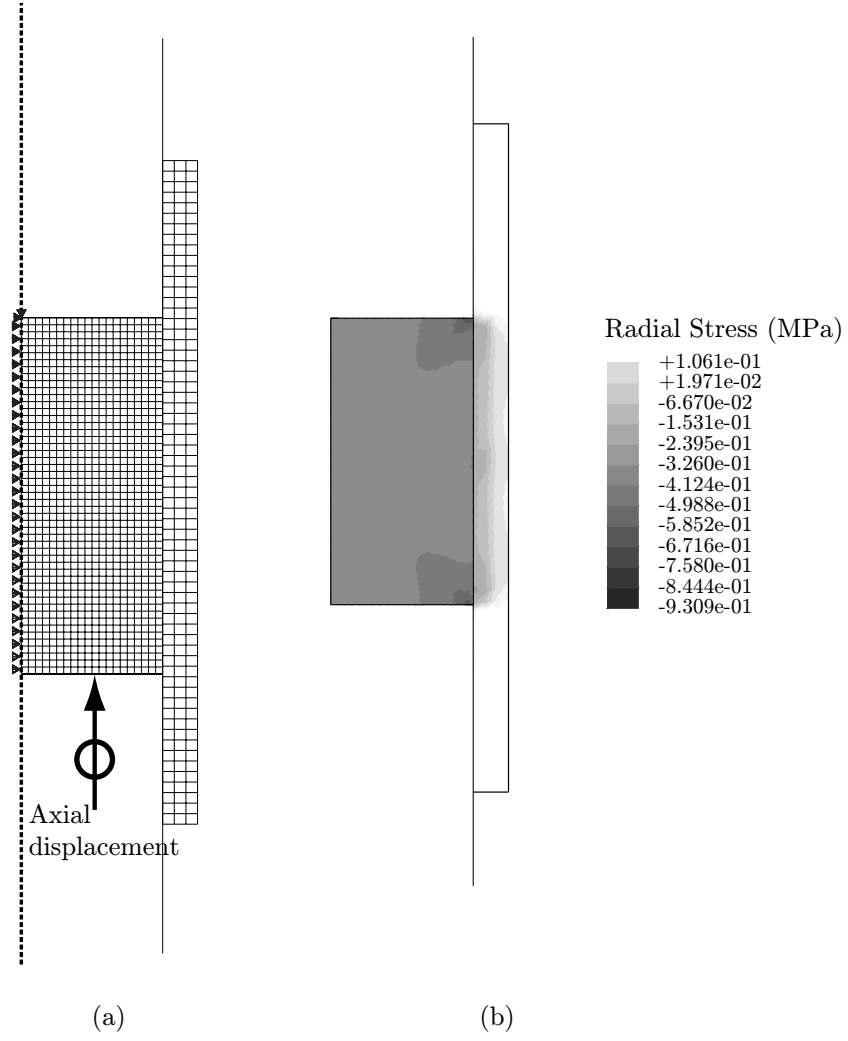


Figure 16: Simulation of the die compaction test: (a) Mesh and boundary conditions, (b) Radial stress distribution.

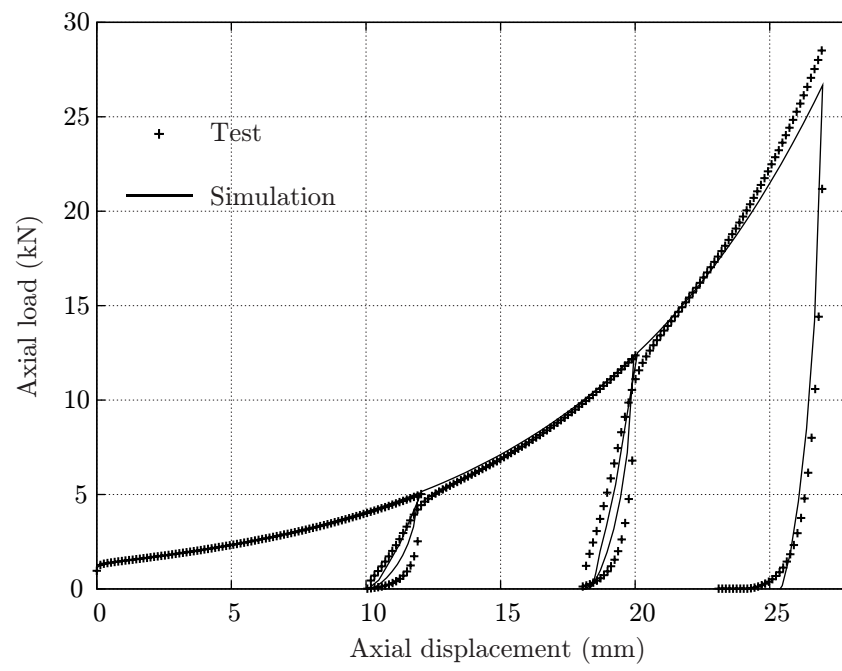


Figure 17: Die compaction test at $T=20^{\circ}\text{C}$.

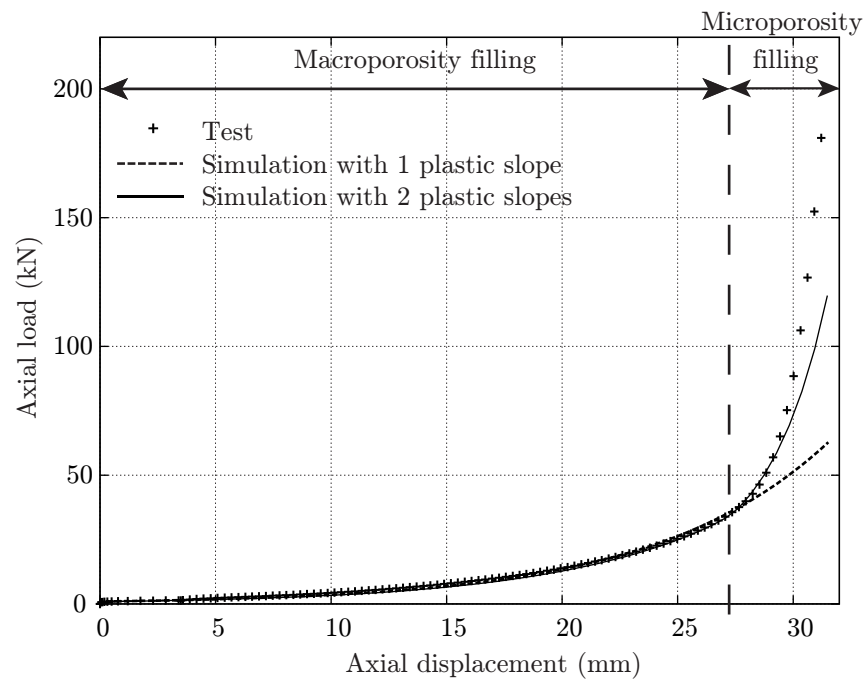


Figure 18: Experimental and numerical results for high pressures.

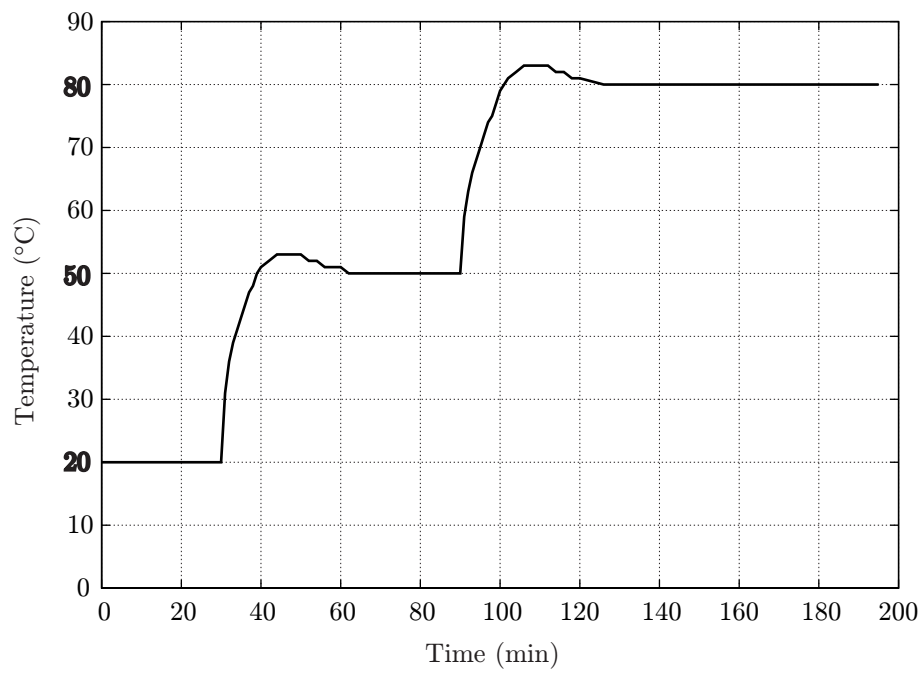


Figure 19: Temperature evolution during the die compaction test.

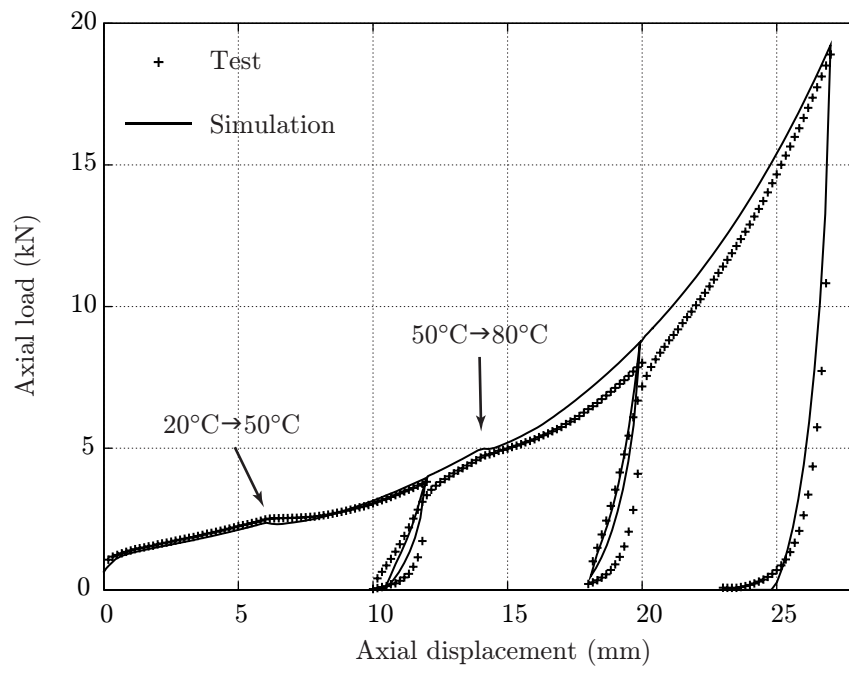


Figure 20: Die compaction test with loads/unloads and heating.

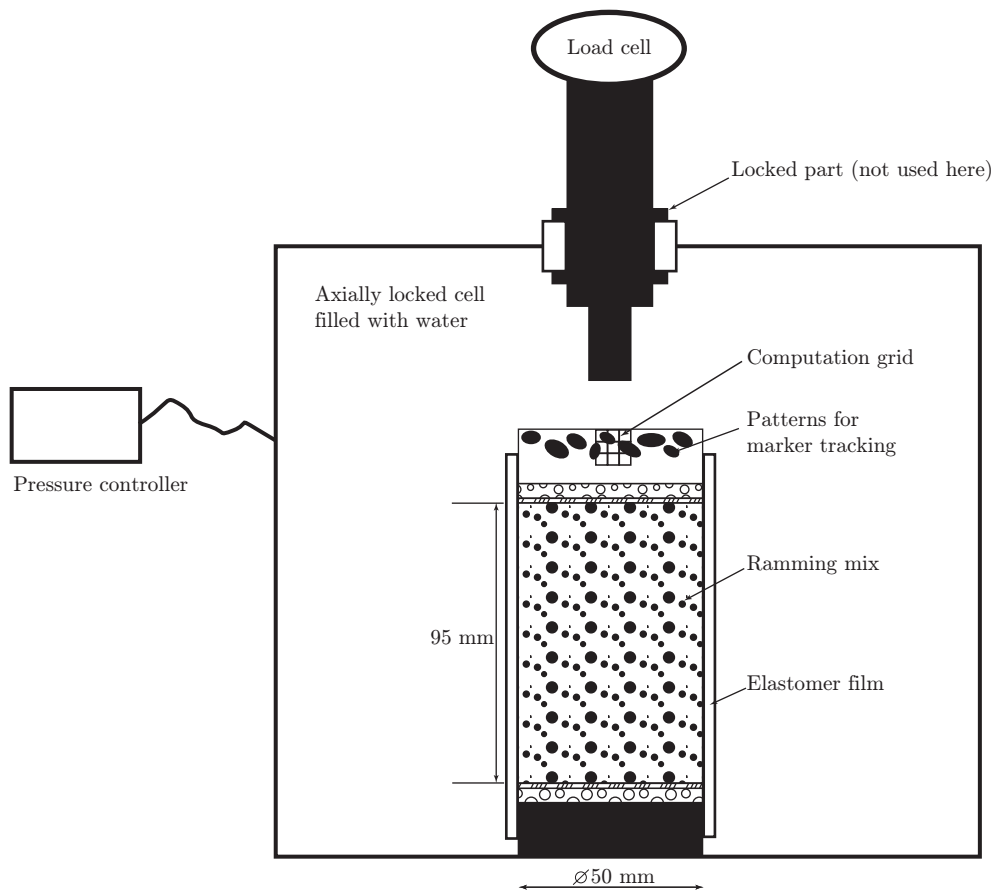


Figure 21: Hydrostatic compression test.

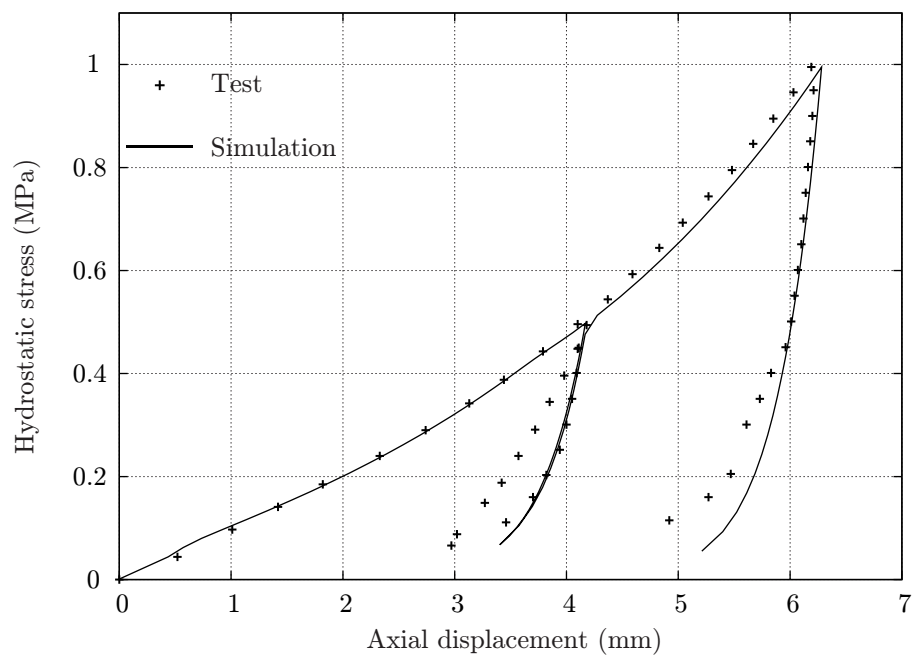


Figure 22: Hydrostatic compression test with loads/unloads.

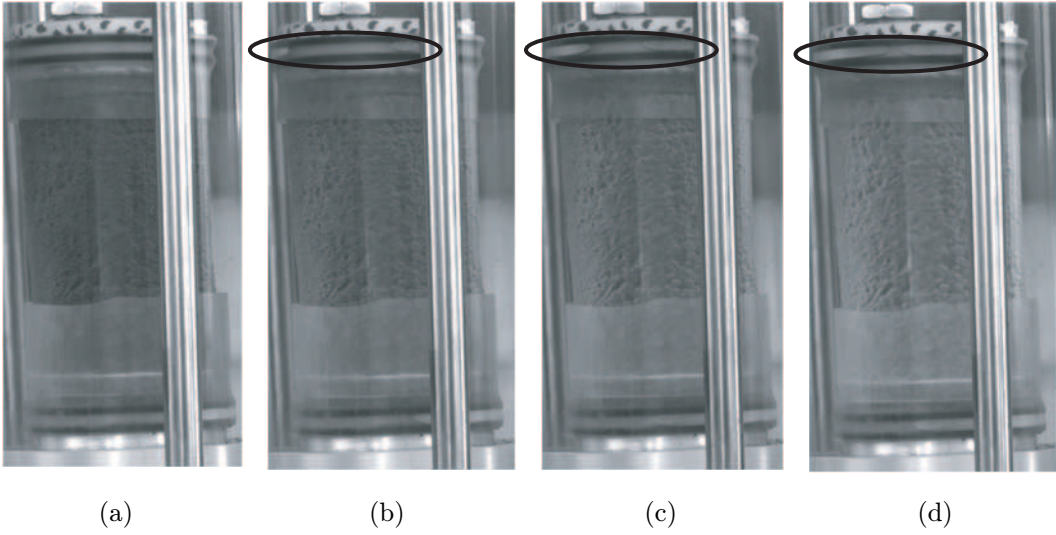


Figure 23: Air pump-effect during the unload for different pressures: (a) 400 kPa, (b) 300 kPa, (c) 150 kPa, (d) 100 kPa.

Table 1: Hardening behaviour parameters for the ramming mix.

Temperature (°C)	20	50	80
κ	0.017	0.017	0.017
λ_1	0.12	0.12	0.12
λ_2	0.024	0.024	0.024
e_1	0.16	0.13	0.1

Table 2: Modified Cam-Clay model parameters for the ramming mix.

Temperature (°C)	20	50	80
ν		0.25	
κ		0.017	
M	1.18	1.1	1.1
λ_1		0.12	
λ_2		0.024	
e_1	0.16	0.13	0.1
β	1	0.56	0.21

Table 3: Thermal properties for the steel and the ramming mix.

	Steel	Ramming mix
Density (kg.mm^{-3})	7.85×10^{-6}	$[1.33 \times 10^{-6}; 1.73 \times 10^{-6}]$
Conductivity ($\text{W.mm}^{-1}.\text{K}^{-1}$)	0.047	0.025
Specific heat ($\text{J.kg}^{-1}.\text{K}^{-1}$)	450	700
Expansion (K^{-1})	1.2×10^{-5}	3.3×10^{-6}