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1 **Investigation of the multiple-fragmentation process and *post*-fragmentation**  
2 **behaviour of dense and nacre-like alumina ceramics by means of tandem**  
3 **impact experiments and tomographic analysis**

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8 **ABSTRACT**

9 During the impact by an armour-piercing projectile against a bilayer ceramic/backing  
10 protective system, dynamic tensile stresses are generated leading to the inception and the  
11 propagation of numerous and oriented cracks. This intense tensile damage, called  
12 fragmentation, affects the performances of the shielding and its capacity to resist to multiple  
13 impact. The residual confined strength of a fragmented ceramic is relatively weak compared  
14 to its undamaged compressive strength, however not negligible. Characterising the dynamic  
15 behaviour of a pre-fragmented ceramic is then of interest to design improved armour  
16 solutions. In this work, an impact configuration called tandem test, which consists in a normal  
17 impact followed by a penetrating impact, is applied to two alumina ceramics with different  
18 microstructures: a conventional 98% pure alumina compared to a bioinspired brick-and-  
19 mortar ceramic called “MAINa” (a nacre-like alumina) which exhibits higher flexural strength  
20 and fracture-toughness. Following the normal impact tests, a fragments size analysis is  
21 conducted by means of X-ray micro-computed tomography distribution, which reveals that  
22 the mean fragments size is significantly larger in the nacre-like alumina, thus demonstrating a  
23 correlation between the fragments size and the penetrating resistance of fragmented alumina  
24 ceramic. Finally, the strength of both alumina ceramics in their fragmented state is identified

25 based on an inverse approach in which the penetrating tests are numerically simulated  
26 considering the Drucker-Prager model.

27 **Keywords:** *Armour, damage, X-ray tomography, fragments size, Drucker-Prager model.*

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## 28 **1 Introduction**

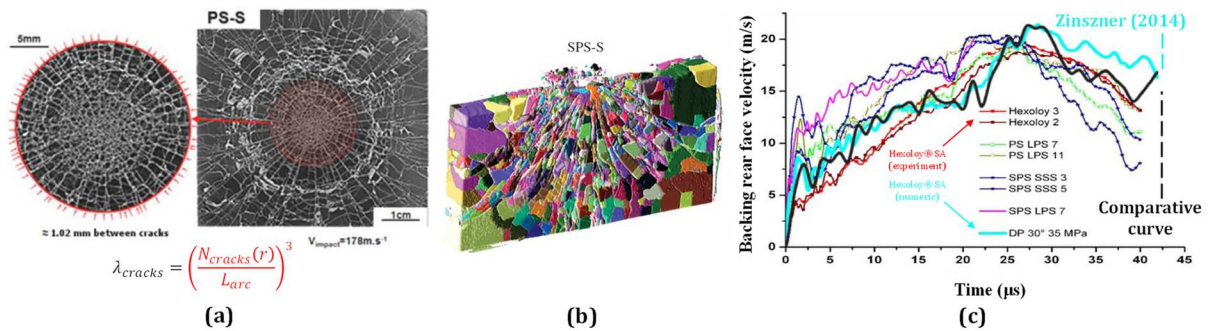
29 In Vietnam War (1955 – 1975) and later on, advanced ceramics have been largely used  
30 in armour systems to create lightweight body and vehicle protection to resist against bullet  
31 projectiles, such as the well-known Armour-Piercing (AP) projectiles [1]. Alumina ( $\text{Al}_2\text{O}_3$ )  
32 ceramics are common ceramic materials used in ballistics, along with titanium diboride  
33 ( $\text{TiB}_2$ ), boron carbide ( $\text{B}_4\text{C}$ ) and silicon carbide ( $\text{SiC}$ ). Among these four ceramics,  $\text{Al}_2\text{O}_3$  has  
34 the lowest ballistic efficiency and the highest density, but the total cost is much lower that  
35 makes  $\text{Al}_2\text{O}_3$  very attractive [2]. Due to their compression strength, ceramic materials are used  
36 in bilayer shielding, which constitutes among the most efficient protective configuration, to  
37 shatter and break the impacting threat of the projectile core by erosion and fragmentation [3–  
38 5]. However, ceramics exhibit *a contrario* a brittle behaviour and their inherent low tensile  
39 properties make necessary the use of a ductile plate (aluminium alloy, steel, composites such  
40 as fibre-reinforced polymer) as backing, in order to convert the debris' kinetic energy and  
41 mechanical momentum into deformation and delamination [4,6]. For instance, it was observed  
42 that the ballistic limit velocity of a 11.4-mm thick alumina plate (AD-85) was only 390 m/s  
43 compared to 650 m/s for a 6.35-mm alumina (AD-85) backed with aluminium of same  
44 thickness [7]. The impact scenario of an AP projectile against a ceramic plate can be divided  
45 into three phases [8–10]. For  $\sim 1 \mu\text{s}$ , a triaxial compression under uniaxial strain is generated,  
46 inducing damage mechanisms such as micro-plasticity (with pore collapse) and micro-  
47 cracking [8,11], under strain-rates exceeding  $10,000 \text{ s}^{-1}$ . In the second loading phase ( $1 - 5$   
48  $\mu\text{s}$ ), a dynamic fragmentation due to high strain-rate tensile loading occurs. Finally, the third

49 stage, which is the longest one (up to 30 [3] or 56  $\mu\text{s}$  [5]), corresponds to the penetration of  
50 the projectile within the fragmented – partially or fully damaged – ceramic for which the size  
51 and the mobility of fragments play a key role on the resistance of the ceramic [10]. Thanks to  
52 X-ray radiography analysis, it was observed that the ceramic (for a boron carbide ( $\text{B}_4\text{C}$ )) is  
53 actually stopped 56  $\mu\text{s}$  after impact [5]. During the penetration of a ceramic armour system by  
54 an AP projectile, less than 1% of the total impact energy is estimated to be absorbed by the  
55 fracturing of the ceramic [12], and so debris ejecta do participate to a large proportion of  
56 removal of the total energy. It appears that the contribution of fragments resistance in the  
57 penetration stage plays a major role, although the ceramic has already been strongly cracked  
58 upon impact. Thus, the penetration resistance of the fragmented media needs to be considered  
59 as well as the material parameters of the ceramic prior fragmentation (such as Hugoniot  
60 Elastic Limit (HEL), tensile strength, *etc.*) to design improved shielding.

61 The resistance of a fragmented ceramic under impact loading was investigated in the  
62 2010s in the PhD works of Rossiquet [13] and Zinszner [10] with a new “tandem  
63 configuration” developed by Zinszner, Forquin and Rossiquet [14,15]. In this test, the ceramic  
64 is fragmented upon normal impact of a flat-end projectile, as a first step. *Post-mortem*  
65 observations of the fragmented SiC ceramic are conducted to analyse the cracks density  
66  $\lambda_{cracks}$  (example in Figure 1 (a)) as studied by Zinszner, Forquin and Rossiquet [15] and  
67 Forquin *et al.* [16]. Circles of given radius  $r$  were drawn from the impact point and the  
68 number of cracks  $N_{cracks}$  crossing each circle was counted. This number was divided by the  
69 arc length ( $L_{arc} = r \cdot \theta$  where the radius  $r$  is the distance from the impact point and  $\theta$  the arc  
70 angle in radian), all of which being raised at the power of three. Indeed, the fragmentation  
71 process is a 3D process and cracks are supposed to be triggered from volume defects (*i.e.*  
72 defects randomly distributed in the volume). In Forquin and Andò [17], the fragments  
73 distribution of a target subjected to an Edge-On-Impact (EOI) test was investigated by means

74 of X-ray micro-computed tomography, Figure 1 (b). Following the normal impact, a  
75 perforating (or rather penetrating) impact of a penetrating projectile is conducted as a second  
76 step, in order to evaluate the resistance of the pre-fragmented ceramic. The velocity profile  
77 measured on the rear face of the backing is compared to the data of numerical simulation with  
78 the aim to identify Drucker-Prager (DP) plasticity model [18], which provides a describing of  
79 the dynamic behaviour of the fragmented ceramic. The DP model was formerly considered to  
80 numerically model the mechanical response of a fragmented ceramic in the modified Wilkins'  
81 model [19]. This latter model [20,21] also includes a tensile damage model to simulate the  
82 growth of fracture conoid (Hertzian cracks) [20,21]. DP is a pressure-dependant model  
83 defined in 1952, usually applied to granular medias which exhibit a small or nil cohesion  
84 strength. Anderson Jr., Chocron and Behner [22] postulated that the DP constitutive model  
85 could represent the penetration response of a SiC, pre-damaged by thermal shock and load-  
86 unload cycles. The three model parameters of the DP model were determined by inverse  
87 approach and a good correlation with experimental results was obtained with several sets of  
88 parameters. However, it was pointed out by Anderson Jr. *et al.* [23] that a projectile  
89 necessarily penetrates a damaged fragmented media whatever the penetration velocity as the  
90 damage front propagates faster than the front of the impacting rod. More recently, an  
91 identification of the DP model parameters was performed by Zinszner [10] based on the  
92 numerical simulation of the penetration impact of a conical end projectile (in tandem  
93 configuration) through a pre-fragmented SiC ceramic. The numerical velocity profile matches  
94 with experimental results, which validates the numerical model, Figure 1 (c). The  
95 development of ceramic materials aiming to mitigate cracks propagation under impact loading  
96 is something that attracted global attention in ballistics. Since 2003, Forquin *et al.* [24] have  
97 investigated the use of a SiC ceramic infiltrated with an aluminium alloy for reducing the  
98 mobility of fragments and increasing the resistance towards a second impact. It is the reason

99 why, ceramics having high fracture-toughness could be good candidates as front plate of  
 100 bilayered configurations.



101 (a) (b) (c)  
 102 **Figure 1.** Estimation of the density of cracks in a fragmented SiC ceramic subjected to normal impact (adapted  
 103 from: [15]). (b) 3D X-ray and *post*-treated rendering of a fragmented SiC grade after EOI [17]. (c) Numerical  
 104 simulation of a penetrating impact test according to [10] curve and as obtained in this work (curve in black  
 105 colour), compared to the experimental velocity profile (adapted from [10]). [2-column fitting]

106 Biological materials (such as: mollusc shells and seashells, arthropod exoskeleton, antlers,  
 107 tusks and teeth, bird beaks, bones, coral, ... [25]) adopt microstructures that have drawn  
 108 attention to researchers because they exhibit exceptional mechanical properties *i.e.* high  
 109 stiffness, high strength and high toughness, while keeping a moderate density [25]. Their  
 110 components are organised in complex hierarchical structures although they are made of basic  
 111 natural polymers and minerals that are relatively weak when taken individually [25]. The  
 112 reason of their superior performances exceeding what can be achieved using the same  
 113 synthetic materials comes indeed from the complex microstructure they adopt  
 114 (organic/inorganic laminar structures, presence of porous and fibrous elements) and the  
 115 interaction between each constituent [25]. A material which brought attention to researchers  
 116 working in materials science is the nacre that covers the surface layer of many seashell  
 117 species (*e.g.* abalone Mollusca): it is a platelet-reinforced composite made of calcium  
 118 carbonate CaCO<sub>3</sub> (calcite or aragonite minerals [26]) with an organic matrix made of  $\alpha$ -amino  
 119 acids (containing aspartic acid, cysteine, glycine, alanine [26]) and polysaccharide [27]. As  
 120 noted by Chen *et al.* [25], nacre, like other biological materials, adopts an increasing

121 complexity of its hierarchical structure as the dimension becomes smaller. The nacreous  
122 architecture is built with “bricks” of stacked platelets (~ 500 nm thick in abalone [25]) glued  
123 at their interfaces with an organic “mortar” matrix (20–50 nm thick in abalone [25]), hence  
124 the so-called “brick and mortar” (BM) microstructure name. In addition, inter-tile mineral  
125 bridges within the “mortar” help to stick the platelets together as well as providing toughness  
126 resistance [25]. This anisotropic, or rather orthotropic [28], type of material is in majority  
127 composed of CaCO<sub>3</sub> ceramic (95 wt.% inorganic, 5 wt.% organic in abalone [25]) and can  
128 exhibit a fracture energy 3000 times higher than the ceramic alone [27]. Damage-resistance is  
129 achieved through different failure mechanisms depending on the loading direction. Menig *et*  
130 *al.* [29] characterised abalone shells under quasi-static and dynamic loadings, and Chen *et al.*  
131 [25] described the influence of the abalone shell microstructure in a review, dedicated to  
132 mechanisms. When loaded perpendicularly to the platelets, failure modes correspond to axial  
133 splitting in compression (compressive strength  $\sigma_c = 540 \text{ MPa}$ ), bridging fracture and  
134 extension of the organic layer in tension (ultimate tensile strength  $\sigma_t = 5 \text{ MPa}$ ). When loaded  
135 in parallel to the platelets, failure modes consist in axial splitting mode and plastic  
136 microbuckling (kinking) in compression ( $\sigma_c = 235 \text{ MPa}$ ) and tiles sliding with brittle inter-  
137 shear failure in tension ( $\sigma_t = 170 \text{ MPa}$ ) [25,29]. The dynamic compression strength of  
138 abalone BM microstructure was found to be 50% higher than its quasi-static strength [29].  
139 The organic layer plays a significant role in controlling damage since it gives viscoplastic  
140 deformation as well as crack deflection [61]. Radi *et al.* [30] developed a series of mesoscopic  
141 numerical simulations of nacre using Discrete Element Method (DEM) and concluded that the  
142 ratio of interface strength to tablet drives the regime of failure. Increasing the interface  
143 strength compared to the matrix strength (ratio tending to 1) should lead to a brittle and  
144 catastrophic failure and should be avoided. More details upon the role of the polymer (or even  
145 metallic [31]) phase can be found in other works ([32–35]). Several processing strategies were

146 attempted to replicate the layering of nacre. Bouville *et al.* [36] engineered a BM artificial  
147 nacre entirely mineral, of composition 98.5 vol.% Al<sub>2</sub>O<sub>3</sub>, 1.3 vol.% SiO<sub>2</sub> and 0.2 vol.% CaO.  
148 This synthetic nacre is not a ceramic/polymer composite but a ceramic/ceramic one, mostly  
149 composed of alumina since both the platelets and the matrix are made of alumina material. In  
150 fact, the presence of a polymeric phase brings toughness but decreases strength [37] and high-  
151 temperature capabilities [36]. Freeze-casting and-drying processes were used on the green  
152 body during the preparation, known as ice-templating. The latter technique is a directional  
153 freezing under a flow method of the suspension allowing obtaining long-range order of  
154 parallel ice crystals. The growth of ice crystals acts as a driving force for the local self-  
155 assembly of the anisotropic platelets. These crystals of ice are then removed by vacuum  
156 freeze-drying. Supplementary details on such process can be found in [38,39] and more  
157 details are available in the patent WO 2015/189659 “Ceramic product with oriented particles  
158 and method for the production thereof” [40]. The sintering (of the 86%-porous sample) is  
159 realised by Spark Plasma Sintering (SPS) at 1500 °C with pressure assistance (100 MPa). The  
160 nacre-like alumina in Bouville *et al.* [36] was tested in 3-point bending tests to point the  
161 importance of the synergetic effects of the nacre constituents (platelets + glass phase +  
162 bridging nanoparticles). This material was compared to a first “partially”-like nacre without  
163 nanoparticles (platelets + glass phase) which exhibits a classical brittle response even if a high  
164 fracture-toughness is achieved compared to a conventional alumina ceramic (6.1 MPa.m<sup>1/2</sup> vs.  
165 3.5 MPa.m<sup>1/2</sup>). This nacre-like alumina was also compared to a second “partially”-like nacre,  
166 without glass phase (platelets + nanoparticles), which exhibits a significant toughening stage  
167 with high failure strain, but with a lower flexural strength  $\sigma_w$  (~ 275 MPa, against ~ 325 MPa  
168 for a reference alumina, ~ 350 MPa for the material without nanoparticles but with platelets +  
169 nanoparticles and ~ 450 MPa for the nacre from Bouville *et al.* [36]). The latter authors  
170 distinguish the “intrinsic” and “extrinsic” toughening phenomena. Intrinsic phenomena



171 include the inherent strengths of the matrix, of the platelets and of the bridges, the visco-  
172 plasticity of the matrix and the crack deflection at the platelet interface. Extrinsic phenomena  
173 (“no true ductility”) that results in stable crack growth, are explained by the contribution of  
174 each nacre constituent, including visco-elastic deformation of the matrix, bridge rupture, and  
175 inelastic shearing and frictional sliding during platelet pull-out. At the crack tip, crack  
176 branching, crack bridging, multiple cracking and platelet delamination release the locally high  
177 stresses. Numerical model of a BM armour consisting of B<sub>4</sub>C tablets and polyurea  
178 tablet/tablet interfaces was investigated in [41]. Another numerical model using layer  
179 waviness and cohesive interface in an aluminium alloy 7075 inspired by nacre was developed  
180 in [42]. Both studies concluded that better ballistic performances were achieved compared to  
181 their monolithic counterpart, but no experimental data that permits to confirm or infirm these  
182 results. To the best of authors knowledge, no fragmentation testing technique has ever been  
183 applied to nacre, to the exception of EOI tests applied to nanolayered Ti<sub>3</sub>SiC<sub>2</sub> (MAX phase)  
184 [43].

185 The two materials considered in the present study named 98% Al<sub>2</sub>O<sub>3</sub> and MAINa (which  
186 means Nacre-Inspired Alumina Material or *Matériau Aluminé Inspiré de la Nacre* in French)  
187 are presented in the second section, while tandem tests are presented in the third section.  
188 Normal impact test results are presented in the fourth section. Next, the penetrating impact is  
189 numerically simulated in the fifth section in order to identify the Drucker-Prager parameters  
190 by means of a numerical inverse approach, thus simulating the fully damaged material.  
191 Finally, the microstructure influence of both alumina ceramics on their fractured (damaged)  
192 strength is discussed.

## 193 **2 Ceramic samples**

### 194 2.1 Microstructures

#### 195 *2.1.1 Alumina ( $Al_2O_3$ ) ceramic*

196 The reference ceramic (Figure 2, left) is a dense alumina, 98% pure in mass, that is  
197 produced with a classical route. The production of the ceramic starts with the selection of the  
198 raw powder that strongly influence the performances of the material [13]. The green-body  
199 (mixture of ceramic with organic and inorganic additives before sintering) is shaped by  
200 uniaxial-pressing. The sintering step consists in consolidating the material by heat action  
201 without (solid-state) or with partial (liquid-state) melting of one or several constituents in a  
202 kiln. During this step, grains merge at their boundaries (coalescence) and the porosity content  
203 is considerably reduced (densification, dimensional shrinkage) [44,45]. The sintering is  
204 performed pressure-less (PS), that constitutes a traditional and economical technique to sinter  
205 large quantities of ceramic products [13].

#### 206 *2.1.2 Nacre-like alumina (MAINa) ceramic*

207 The MAINa is described in the patent “Dense sintered product” (WO 2018/141736)  
208 [46]. The platelets (Ronaflair® White Sapphire powder from Merck group) are the first  
209 particulate fraction (or platelet fraction) of approximate size 5–10  $\mu m$  with 500–1000 nm in  
210 thickness. The second particulate fraction (size < 1  $\mu m$ ) is made of three constituents. First,  
211 mineral ceramic bonds form bridges between the platelets that correspond to nanoparticles of  
212 YSZ (Yttria ( $Y_2O_3$ )-partially Stabilised Zirconia ( $ZrO_2$ )) from TZ-3Y-E® (3 mol.%  $Y_2O_3$ )  
213 powder and of approximate average size 40 nm [47] according to the supplier (from Tosoh).  
214 Second and third, additional smaller nanoparticles (~ 20 nm) of silica ( $SiO_2$ ) and calcia (CaO)  
215 of liquid-phase precursors are present as glass or glass-ceramic particles to foster filling the  
216 remaining gaps during the sintering stage. Some of these nanoparticles deposit on the surface  
217 of platelets and form nano-asperities responsible of part of the energy dissipation by frictional

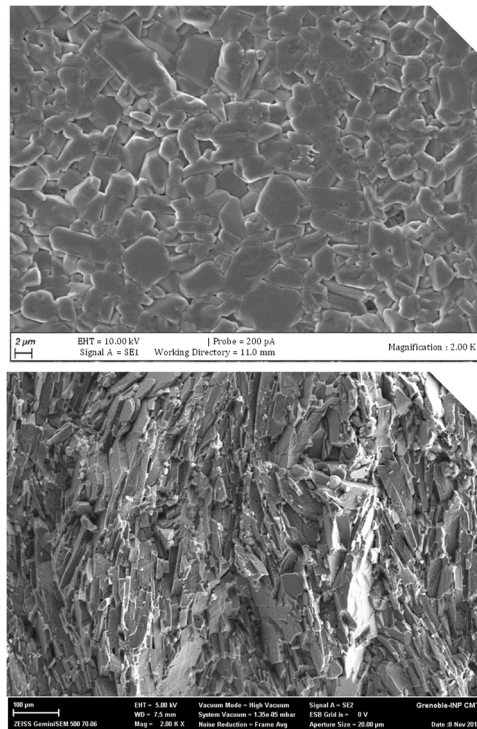
218 sliding during platelet pull-out and breaking of the bridges. In terms of composition, the  
219 material is made of 94.7 wt.%  $\text{Al}_2\text{O}_3$ , 4 wt.%  $\text{ZrO}_2$ , 0.75 wt.%  $\text{SiO}_2$ , 0.25 wt.%  $\text{CaO}$  and 0.3  
220 wt.% of other elements (of which less than 0.1 wt.% of  $\text{MgO}$ ). The processing strategy and  
221 the manufacturing processes can be divided into the three following steps.

222 Powder preparation of an aqueous colloid: An anionic dispersant (Darvan® 7NS from  
223 Vanderbilt Minerals, sodium methyl methacrylate (Na-MMA)) and additives are mixed  
224 together in de-ionised water with a suspension of nanometre powders ( $\text{ZrO}_2$ ,  $\text{SiO}_2$ ,  $\text{CaCO}_3$ ),  
225 then agitated (ball-milled with  $\text{Al}_2\text{O}_3$  balls) overnight (at least 12 hours) to ensure a proper  
226 deagglomeration.  $\text{Al}_2\text{O}_3$  platelets are then added 3 hours before the end of the cycle (to avoid  
227 any excessive breakage by the milling media) with a thickener (Carbopol® EDT 2691 from  
228 Lubrizol Corporation).

229 Freezing of the green body: The obtained green body is poured in a stainless steel  
230 before the recipient be plunged in liquid nitrogen for rapid freezing (around 20 minutes). Ice  
231 crystals are created. The frozen green body is frozen-dried for about 5 days. The intermediary  
232 product, a dried bloc with low mechanical properties, is ground with a hand press before  
233 being sieved at 1 mm to eliminate the biggest agglomerates.

234 Thermal treatment then pressure-assisted and Field-Assisted Sintering Technique  
235 (FAST): The ground powder is thermally treated at 450 °C for 1 hour (50 °C/h of temperature  
236 ramp) and fired until 900 °C (100 °C/h) then cooled (-300 °C/h), so that the organic  
237 constituents are removed (debinding) and the nanoparticles pre-sinter on the platelets. The  
238 nanoparticles of the glass phase sinter at lower temperatures than platelets due to their higher  
239 surface area and curvature. The  $\text{ZrO}_2$  nanoparticles are not completely dissolved in the glass  
240 phase thanks to short processing times during the densification. The precursor particles ( $\text{SiO}_2$   
241 and  $\text{CaO}$ ) may delay the densification of the  $\text{ZrO}_2$  nanoparticles by preventing their contacts  
242 with the platelets. Following this, the powder is placed in a cylindrical graphite matrix to be

243 pressed at 5 MPa. Afterwards, the die is placed within a kiln and sintered via pressure-assisted  
244 FAST (or pressure-assisted SPS technique) at around 1500 °C (100 °C/min) with 50 MPa.  
245 Compared to conventional sintering, SPS technique produces a more homogeneous heating,  
246 higher heating rate, finer grains and higher relative densities. The elevation of temperature is  
247 due to the Joule effect of a graphite die travelled by a Direct Current (DC). After sintering, the  
248 platelets are aligned to each other.



249 **Figure 2.** SEM micrographs of Al<sub>2</sub>O<sub>3</sub> (courtesy of Saint-Gobain, at the top) and MAINa fracture surface (3SR, at  
250 the bottom) showing its BM microstructure. [single-column fitting]  
251

## 252 2.2 Materials properties

253 Some physical properties of both materials are reported in Table 1. The densities  $\rho$   
254 come from material data sheets to be compared to the theoretical densities  $\rho_{th}$  of 3890 kg.m<sup>-3</sup>  
255 for Al<sub>2</sub>O<sub>3</sub> and 3933 kg.m<sup>-3</sup> for MAINa. The porosity  $p$  of Al<sub>2</sub>O<sub>3</sub> is deduced from its purity, the  
256 MAINa's is estimated by comparing its theoretical density (~ 4032 kg.m<sup>-3</sup>) and the measured  
257 Archimede's density (~ 3933 kg.m<sup>-3</sup>). The grain size (for Al<sub>2</sub>O<sub>3</sub>) and the platelet size (for  
258 MAINa) are provided by Saint-Gobain thanks to SEM images.

Grade	$\rho$ (kg.m <sup>-3</sup> )	$p$ (%)	Grain (Al <sub>2</sub> O <sub>3</sub> ) / Platelet (MAINa) size ( $\mu$ m)
Al <sub>2</sub> O <sub>3</sub>	3900	~ 2	~ 2.6
MAINa	3930	~ 3	~ 5 – 10 (diameter) ~ 0.5 – 1 (thickness)

**Table 1.** Physical properties of the two Al<sub>2</sub>O<sub>3</sub> grades (Saint-Gobain data).

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Some basic mechanical properties are reported in Table 2. Relative to MAINa, which is an anisotropic material, data of the latter refer to its characterisation when platelets are oriented in a perpendicular way to the direction of loading. The hardness  $H_V$  was measured by Vickers indentation by using a Buehler 6030 micro-durometer with a diamond squared-base pyramid in accordance with the NF and EN 843-4 standard [48]. The samples were beforehand embedded in a resin then polished. The hardness of Al<sub>2</sub>O<sub>3</sub> was tested with a 0.2-kg load ( $H_{V_{0.2}}(Al_2O_3) = 1735$ ) and the nacre's with a 0.1-kg load ( $H_{V_{0.1}}(nacre) = 1600$ ). Hardness data are expressed in Pascal unit of pressure (GPa) later on in Table 2. The Young's modulus  $E$  of Al<sub>2</sub>O<sub>3</sub> was calculated using a resonance measurement method in accordance with the ASTM C1259 standard [49]. Subject to a mechanical impulsion, the mechanical vibration is detected and converted into an electric signal, which is analysed to isolate fundamental frequency. Using flexural specimens, the flexural vibration mode provides the elastic modulus. Using rectangular specimens, both the flexural and the torsional vibration modes provide both the Young's and the shear moduli. The two Young's moduli obtained with both types of specimen are almost identical. The value delivered by the rectangular specimen reported in Table 1 will be kept as this geometry also delivers the shear modulus. The Young's modulus  $E$  of MAINa was calculated using bending test results (Table 1). Considering the Young's modulus and the shear modulus (obtained from the resonance frequency in torsional vibration mode), the Poisson's ratio  $\nu$  can be deduced according to ASTM C1259 [49]. The Modulus Of Rupture (MOR) is measured by 3-point bending (or

280 flexural) tests performed with  $\sim 45 \times 4 \times 3 \text{ mm}^3$  (rectangular cross section) polished and  
 281 chamfered test beams, in accordance with the NF EN 843-1 standard [50]. Tests were  
 282 conducted with a 10-kN Shimadzu loading press. Thanks to the MOR value, the mean failure  
 283 stress  $\sigma_w$  is calculated with a Weibull's analysis [51,52]. The stress data displayed in Table 2  
 284 are given as function of an effective volume  $V_{eff}$  ( $1 \text{ mm}^3$ ) based on equation (1) [53].

$$V_{eff} = \int_{\Omega} \left( \frac{\langle \sigma(\underline{x}) \rangle}{\sigma_{max}} \right)^m . d\omega, \quad (1)$$

285 where  $m$  is the Weibull's modulus,  $\sigma(\underline{x})$  is the longitudinal stress in the tested sample at a  
 286 point of coordinates,  $\sigma_{max}$  is the maximum principal stress, and  $\langle . \rangle$  are the Macaulay brackets  
 287 that take the positive (tensile) value of stress. In 3-point bending, the equation (1) becomes  
 288 (2):

$$V_{eff} = \frac{V}{2. (m + 1)^2} \quad (2)$$

289 Given the Weibull's modulus  $m$ , the mean strength  $\sigma_{w,ref}$  associated to a reference effective  
 290 volume ( $V_{eff,ref} = 1 \text{ mm}^3$ ) can be calculated in (3):

$$\sigma_{w,ref} = \sigma_{w,bending} \cdot \left( \frac{V_{eff,bending}}{V_{eff,ref}} \right)^{\frac{1}{m}} \quad (3)$$

291 where:  $\sigma_{w,bending}$  is the mean tensile strength (also called Modulus Of Rupture or MOR)  
 292 equal to 367 MPa ( $V_{eff,bending} \sim 3.0 \text{ mm}^3$ ) and 581 MPa ( $V_{eff,bending} \sim 1.2 \text{ mm}^3$ ) for  
 293  $\text{Al}_2\text{O}_3$  and MAINa, respectively. The larger number of specimens tested in bending ( $\geq 20$ )  
 294 enables to deduce the Weibull's modulus [51,52] of  $\text{Al}_2\text{O}_3$  (Table 2). As regards MAINa, the  
 295 Weibull's modulus reported in Table 2 originates from the nacre-like alumina material.  
 296 Finally, the fracture-toughness  $K_{Ic}$  of the two ceramics was determined in accordance with the  
 297 ASTM C1421 standard [54] using the SENB (Single-Edge Notched-Beam) method, which  
 298 consists in a 4-point bending test performed on a pre-notched sample. The notch was

299 machined with a diamond saw and was ended with pre-crack of around 0.3 mm in length with  
 300 a razor blade coated with a diamond paste to create a stress concentration at the notch tip. In  
 301 MAINa,  $K_{Ic}$  was equal to  $6.15 \pm 0.26 \text{ MPa}\cdot\text{m}^{1/2}$ . The interest of the BM microstructure is that  
 302 the “apparent” fracture-toughness increases as crack propagates due to energy dissipation.  
 303 This enhanced toughening resistance is known as R-curve effect and is characterised by the  
 304 apparent fracture-toughness parameter  $K_J$  in  $\text{MPa}\cdot\text{m}^{1/2}$ . The measured  $K_J$  was  $9.84 \pm 0.34$   
 305  $\text{MPa}\cdot\text{m}^{1/2}$ . It can be noted that compared to  $\text{Al}_2\text{O}_3$ ,  $K_{Ic}$  is 26.54% higher and  $K_J$  is even  
 306 102.5% higher.

Grade	$H_v$ (GPa)	$E$ (GPa)	$\nu$ (-)	$m$ (-)	$\sigma_w$ (MPa)	$K_{Ic}$ ( $\text{MPa}\cdot\text{m}^{1/2}$ )
$\text{Al}_2\text{O}_3$	17.0	369	0.20	8	421**	4.86 ( $\pm 0.50$ )
MAINa	15.7	344	0.26	14*	589**	6.15 ( $\pm 0.26$ )

307 \* Another nacre-like alumina material / \*\* For  $V_{eff,ref} = 1 \text{ mm}^3$

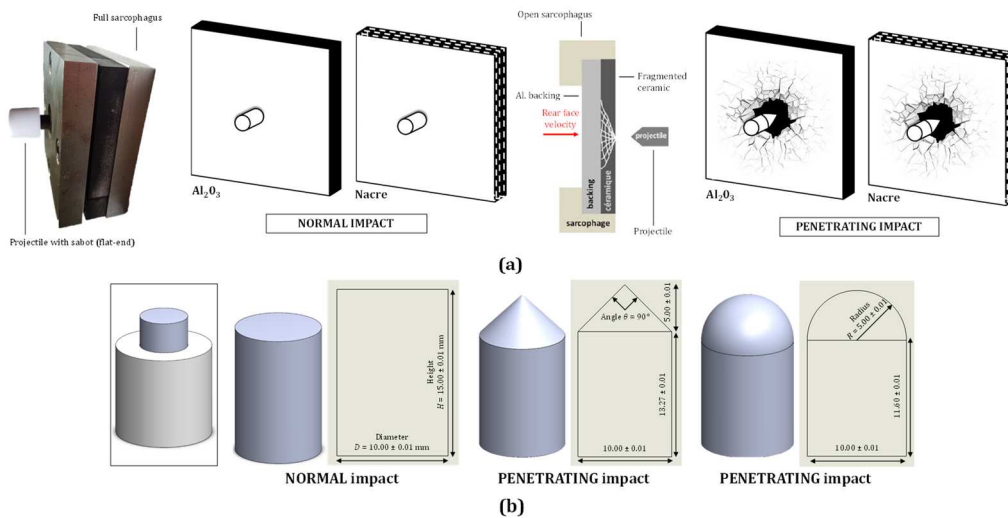
308 **Table 2.** Mechanical properties of the two  $\text{Al}_2\text{O}_3$  grades (Saint-Gobain data).

### 309 **3 Tandem test: normal and penetrating impact test**

#### 310 3.1 Introduction to tandem test

311 The tandem test represented in Figure 3 (a) is a two-step experimental testing  
 312 technique. It was developed in previous research works [13,10,15] to evaluate the behaviour  
 313 of a fragmented ceramic under impact. The first test, called “normal impact test”, is conducted  
 314 with a flat-end projectile (Figure 3 (b)) and uses a confining system to prevent any loss of  
 315 fragments after impact. The second test, called “penetrating impact test”, aims to analyse the  
 316 residual penetration resistance of the fragmented ceramic. It consists in impacting the  
 317 damaged target with a conical end or hemispheric end projectile as shown in Figure 3 (b). The  
 318 mechanical behaviour of the fragmented ceramic is deduced from particle velocity  
 319 measurement on the rear face of the backing.

320 The dimensions (length ( $L$ )  $\times$  width ( $W$ )  $\times$  thickness ( $t$ )) of the ceramic plates are  $50 \times$   
 321  $50 \times 7 \text{ mm}^3$ . Finish required rectification to ensure a strict parallelism of the surfaces (0.05  
 322 mm max. gap); no polishing was required. The exact dimensions and densities of the ceramic  
 323 plates, the impact velocities  $V_{impact}$  and the projectile mass  $m$  are reported in Table 3.  
 324 Projectile impact speed is set to 175 m/s in case of normal impact and penetrating impact with  
 325 a conical-head projectile, whereas an impact speed of 145 m/s is set in the case of penetrating  
 326 impact conducted with a hemispherical-head projectile. Tests "1" and "1b" correspond to  
 327 distinct targets subjected to tandem tests. Tests "2" refer to targets subjected to normal impact  
 328 and X-ray tomographic evaluations. The projectiles are made of steel. The three projectile  
 329 geometries own the same approximate mass (9.10 g) and are placed inside a Teflon® sabot  
 330 having a diameter adjusted with the inner diameter of the gas launcher tube ( $\sim 20.10 \text{ mm}$ ). No  
 331 visible deformation of the projectile in any test was observed after both stages of impact.



332  
 333 **Figure 3.** (a) Tandem test (normal impact view, penetrating impact sketch (adapted from: [10])) with  
 334 orientations of interest. (b) Projectile shapes and dimensions. [1.5-column fitting]

Sample name	Dimensions (mm)	$\rho_{exp}$ ( $\text{kg}\cdot\text{m}^{-3}$ )	Impact test	$V_{impact}$ (m/s)	Projectile $m$ (g)
Tandem-Al <sub>2</sub> O <sub>3</sub> - 1	50.41 $\times$ 50.30 $\times$ 7.03	3896	Normal	175.05	9.11
			Penetrating	175.05	9.13



<b>Tandem-Al<sub>2</sub>O<sub>3</sub>- 1b</b>	50.65 × 50.49 × 7.14	3805	Normal	188.20	9.09
			Penetrating	184.21	9.16
<b>Normal-Al<sub>2</sub>O<sub>3</sub>- 2</b>	50.50 × 50.32 × 7.17	3814	Normal	181.39	8.95
<b>Tandem-MAINA- 1</b>	50.20 × 50.30 × 6.27	3926	Normal	184.21	9.10
			Penetrating	178.16	9.11
<b>Tandem-MAINA- 1b</b>	50.18 × 50.15 × 6.30	3861	Normal	172.31	9.10
			Penetrating	178.46	9.14
<b>Tandem-MAINA- 1b Hemi</b>	50.11 × 50.01 × 6.28	3935	Normal	172.32	9.11
			Penetrating	142.39	9.07
<b>Normal-MAINA- 2</b>	49.62 × 49.44 × 5.63	3902	Normal	181.70	9.02

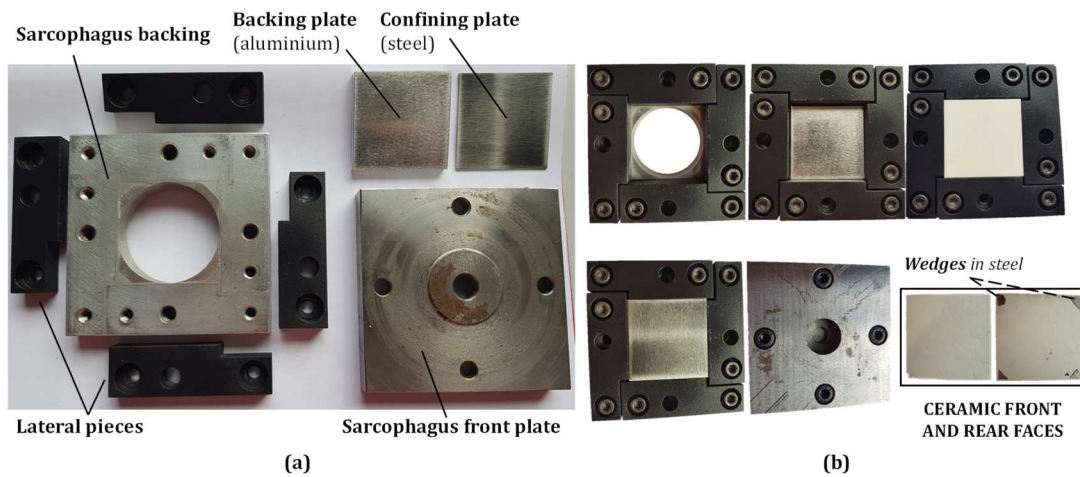
335 **Table 3.** Dimensions and experimental densities ( $\rho_{exp}$ ) of the ceramic samples, type of impact (normal or  
336 penetrating), velocities and mass of the projectiles.

### 337 3.2 Normal impact test

338 In the first stage, a normal impact is performed against an aluminium-ceramic-steel  
339 sandwich configuration. The numerical design and optimisation of this configuration was  
340 studied in [14]. The different pieces of the sarcophagus device are visible in the Figure 4 (a).  
341 An aluminium alloy plate of same size ( $49.40 \times 49.40 \text{ mm}^2$ ) but of smaller thickness (6 mm)  
342 is used as backing (properties in Table 4). Steel wedges of few  $\text{mm}^2$  (*cf.* Figure 4 (b)) are  
343 glued on the rear face corners of the ceramic to impose a 0.1-mm gap, which ensures an  
344 impedance discontinuity that promotes the fragmentation process in the ceramic. At the top of  
345 the ceramic, a thin (1 mm thick) steel plate ( $49.40 \times 49.40 \text{ mm}^2$ ) is placed to prevent any  
346 fragments ejection. The ceramic sandwiched between the aluminium backing and the steel  
347 front sheet is placed in a sarcophagus configuration. The assembly steps are visible in the  
348 Figure 4 (b).

349 3.3 Penetrating impact test

350 The second stage of tandem test consists in impacting the fragmented ceramic backed  
 351 with the same aluminium plate as in normal impact test but using a hemispherical or conical  
 352 end projectile. A stop-sabot device is placed to prevent the impact of the sabot against the  
 353 target during the recording time. The sarcophagus front plate and the thin steel plate are  
 354 removed prior the second impact.



355 (a) (b)  
 356 **Figure 4.** (a) Exploded view of the tandem sarcophagus with (b) successive assembly steps of the tandem  
 357 sarcophagus: screw of the aluminium lateral pieces, positioning of the aluminium backing, positioning of the  
 358 ceramic plate (with wedges), positioning of the steel front plate and closing of the assembly. [2-column fitting]

Grade	$\rho$ (kg.m <sup>-3</sup> )	$E$ (GPa)	$\nu$ (-)	$R_e$ (GPa)
Aluminium 7075	2800	72	0.33	0.45
High-strength steel	7785	210	0.30	1.35

359 **Table 4.** Physical and mechanical properties of the aluminium alloy and of the steel alloy (applies for the steel  
 360 projectiles too).

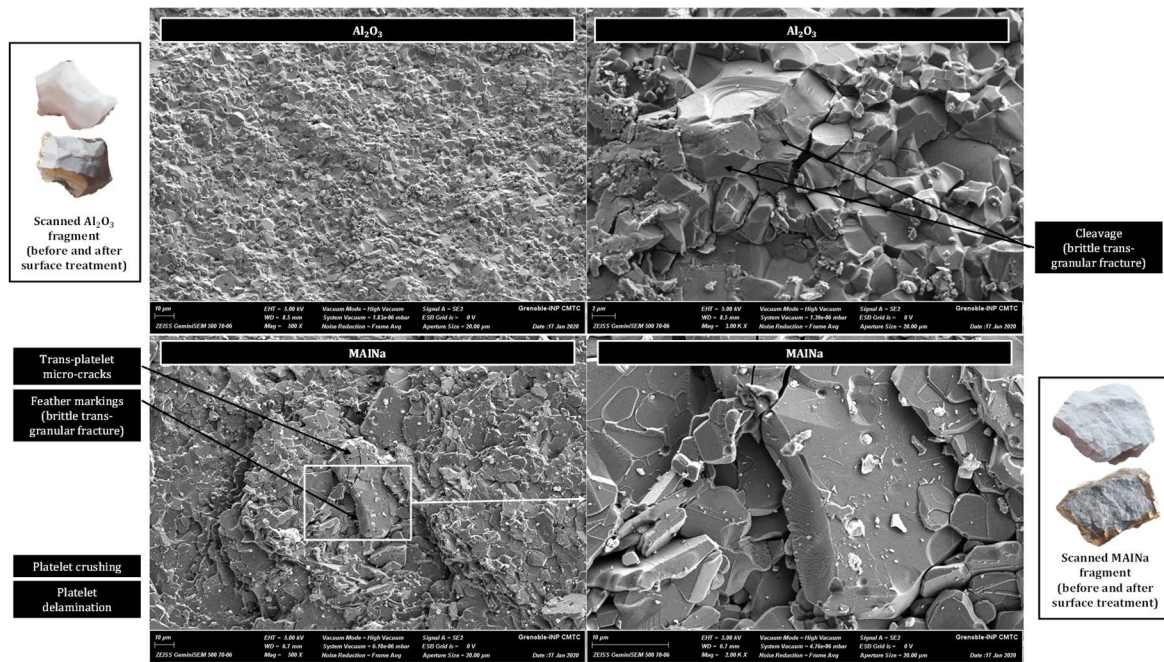
361 The front face of the target is visualised with an ultra-high-speed camera (Kirana®, 5  
 362 Mfps capacity, 924×768 px, 180 images), set to 500 kfps with 2  $\mu$ s exposure time, except for  
 363 “Tandem-Al<sub>2</sub>O<sub>3</sub>-1” and “Tandem-MAINa-1” recorded using 200 kfps. The triggering is made  
 364 with a laser interferometer (1.5 MHz of bandwidth, maximum measured velocity 20 m/s)  
 365 pointed toward the rear surface of the aluminium backing plate. The measured velocity cannot

366 exceed 23 m/s due to the limited range of measured velocity with this equipment. In addition,  
367 two high-power flash lights are placed close to the zone of visualisation to deliver enough  
368 light during recording. The triggering of the flash is performed when the projectile crosses  
369 one of the three velocity barriers that are used to determinate the impact velocity.

#### 370 3.4 Fractography after penetrating impact tests

371 Next to penetrating impact, fragments of ceramics were analysed through SEM using  
372 secondary electrons to investigate the topology information of the fracture surface. The  
373 detector is placed in a grazing position.

374 The fracture surfaces for  $\text{Al}_2\text{O}_3$  exhibits an intergranular-dominated fracturing mode  
375 (as observed in Chen *et al.* (2006) [55] on alumina subjected to plate-impact below the HEL),  
376 although some transgranular modes are also visible (cleavage, river patterns, feather  
377 markings). Images of MAINa fragments put into evidence additional mechanisms of energy  
378 dissipation, such as frictional sliding during platelet pull-out, delamination of platelets and  
379 crack deflection at the platelet interfaces.



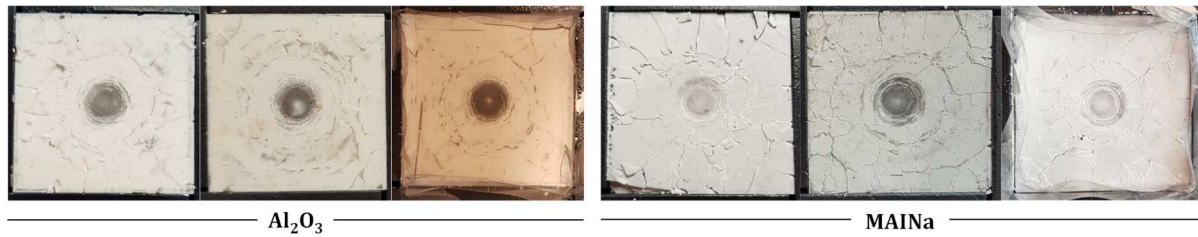
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381 **Figure 5.** SEM micrography on recovered fragments from Tandem- $\text{Al}_2\text{O}_3$ -1b and Tandem-MAINa-1b (1 to 1.5  
 382 mm approximate size along the major axis) on which silver lacquer and graphite were deposited to allow for  
 383 conductivity and evacuating electric charges. [2-column fitting]

384 **4. Fragmentation under normal impact experiment**

385 4.1 Visualisation of cracking pattern

386 When the sarcophagus front plate and the thin steel sheet are taken off, the  
 387 fragmentation pattern for each sample is revealed (Figure 6). The failure pattern of the  
 388 samples with the same microstructure shows a good repeatability. Such “deterministic  
 389 multiple-fragmentation feature” was already noted in previous works (the reader is referred to  
 390 [16]). Since the samples are not polished, it is difficult to count the cracks by direct visual  
 391 observation, especially for  $\text{Al}_2\text{O}_3$  for which crack opening seems to be smaller than MAINa  
 392 (the lower the toughness, the lower the crack opening). Concentric radial circles of cracks are  
 393 found in abundance.



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**Figure 6.** *Post-mortem* observations after normal impact of  $\text{Al}_2\text{O}_3$  and MAINa (from left to right: Tandem- $\text{Al}_2\text{O}_3$ -1, Tandem- $\text{Al}_2\text{O}_3$ -1b, Normal- $\text{Al}_2\text{O}_3$ -2, Tandem-MAINa-1, Tandem-MAINa-1b and Normal-MAINa-2).

[2-column fitting]

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#### 4.2 Evaluation of cracking density

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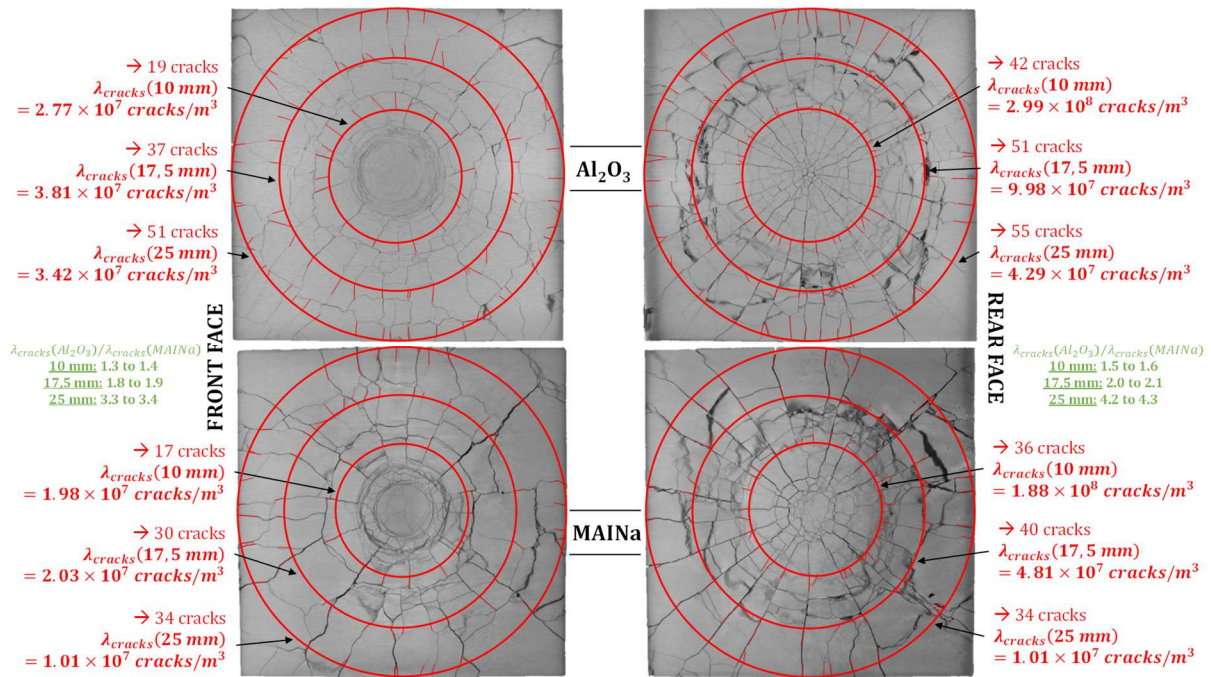
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The samples named Normal- $\text{Al}_2\text{O}_3$ -2 and Normal-MAINa-2 were analysed through tomography scanning. Prior to impact, a plastic sheet was put to encompass the ceramic and the steel plate so that the ceramic target can be vacuum infiltrated (equipment: VACUMET 52® from Mekton Instruments Inc.) with a hyper-fluid resin after impact. After normal impact, the impacted steel plate was removed before infiltration of the fragmented ceramic with vacuum set down to 600 – 650 mmHg (0.80 – 0.87 bar). The infiltrated sample is then cured for 24 hours. Next, 3D field X-ray micro-tomography is performed within the tomographic platform in 3SR. An X-ray beam is generated with a 100-kV voltage and a 250- $\mu\text{A}$  current (tube power of 25 W). The duration of scanning is 112 min producing a set of 1800 slices (400 pixels through the thickness, 1800 pixels through the length and width) with 30- $\mu\text{m}$  of voxel size. The source consists in an electron beam accelerated against a tungsten (W) target. An aluminium filter (0.5 mm thick) is placed in front of the source in order to pre-harden the signal by attenuating low-energy photons. Ring artefacts, caused by shifts of the sample on the rotation axis after reconstruction, are decreased thanks to a ring filter. Three different slices from Computed Tomography (CT) are shown in Figure 7.



414

415 **Figure 7.** Post-mortem evaluation of cracks density based on tomographic images. [2-column fitting]

416 When looking at the Figure 7, MAINa clearly exhibits fewer cracks than the Al<sub>2</sub>O<sub>3</sub>

417 target, so possibly larger fragments. On the front and rear faces from the tomographic images,

418 different circles centred on the impact point were defined considering radius of 10, 17.5 and

419 25 mm, which correspond to different levels of strain-rates (*cf.* next sub-section). The cracks

420 density  $\lambda_{cracks}$  is calculated and marked down. It appears to be about 2 to 3 times smaller in

421 MAINa sample than in Al<sub>2</sub>O<sub>3</sub> on both front and rear faces. Thus, larger fragments are

422 expected in MAINa than in the Al<sub>2</sub>O<sub>3</sub> ceramic sample. More specifically, fragments in

423 MAINa that are far from the impact seem to be drastically larger. For instance, on the front

424 face, the ratio of the cracks density measured in MAINa to the one of Al<sub>2</sub>O<sub>3</sub> is equal to 1.3 to

425 1.4 at 10 mm from the impact point, while it increases to 3.3 to 3.4 at 25 mm.

#### 426 4.3 Estimation of the stress-rate in the ceramic plate

427 The normal impact, that is the first stage of tandem test, is simulated with the

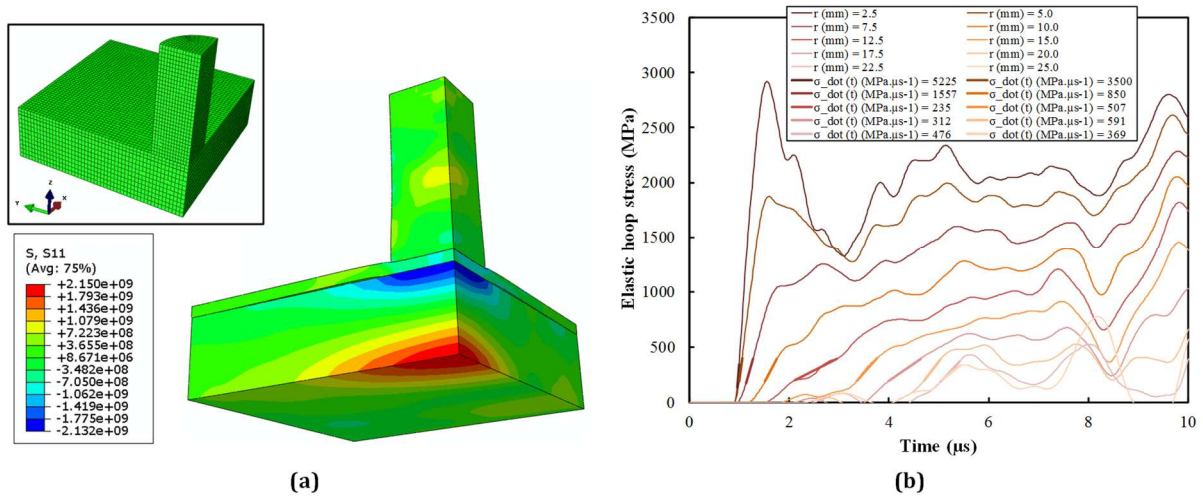
428 ABAQUS Explicit Finite Element (FE) software. The impact velocity with the flat end

429 impactor is set to 175 m/s. Two symmetry planes are considered. The mesh is made of ~ 0.5-

430 mm C3D8R elements (3D 8-node linear brick, reduced integration, hourglass control). The  
431 material parameters considered for the steel projectile and the steel confining plate on the top  
432 of the ceramic are reported in the Table 4. An elasto-plastic model with an elastic limit  $R_e$  is  
433 considered for the projectile and the confining plate whereas the behaviour of the ceramic is  
434 modelled with the elastic parameters provided in Table 2. Surface-to-surface contact  
435 algorithm applies with a friction coefficient of 0.2.

436         In the wake of the compressive wave, fragmentation process develops on the rear face  
437 due to tensile hoop stresses induced by the radial motion of the matter. The evolution of hoop  
438 stresses  $\sigma_{\theta\theta}$  as function of time is plotted considering different radii defining a cylindrical  
439 coordinate system with the opposite face of the point of impact as origin, Figure 8. The stress-  
440 rate  $\dot{\sigma}$  then is deduced considering the slope between 200 MPa and 400 MPa, 400 MPa  
441 corresponding approximately to the tensile strength of alumina measured in 3-point bending.  
442 The change of hoop stress-rate with the radius is plotted as function of the radius in the Figure  
443 9. It is observed that the hoop stress-rate rapidly decreases with the radius until 12.5 mm and  
444 is fluctuating in between 300 and 600 MPa/ $\mu$ s beyond 12.5 mm. Accordingly, the stress-rate  
445 at  $r = 10$  mm ( $\dot{\sigma} = 850$  MPa/ $\mu$ s) is much higher than the stress-rate at  $r = 17.5$  mm ( $\dot{\sigma} = 312$   
446 MPa/ $\mu$ s), which explains the higher cracking density noted at  $r = 10$  mm compared to  $r =$   
447 17.5 mm, as observed in the previous 4.2 section.

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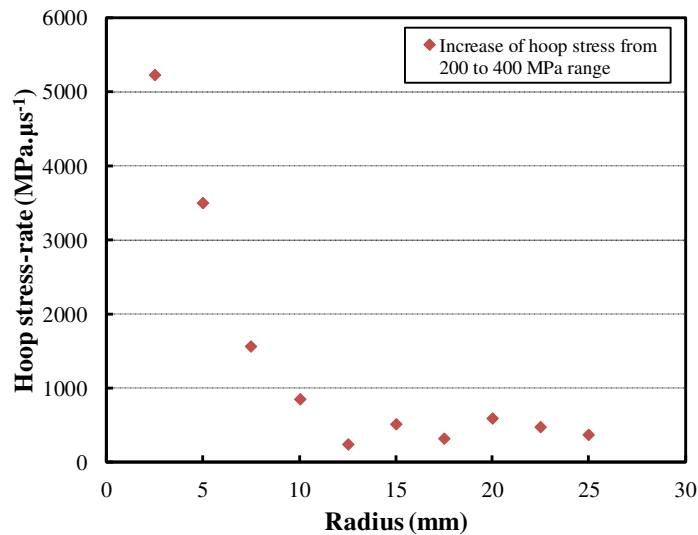
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**Figure 8.** Numerical simulation of normal impact considering an  $\text{Al}_2\text{O}_3$  target impacted at 175 m/s. (a)  $S_{11}$  stress field at 5  $\mu\text{s}$  after impact. (b) Profile of hoop stresses on the rear face of the ceramic as function of time.

[1.5-column fitting]



453

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**Figure 9.** Change of the hoop stress-rate with the radius in the rear face of the  $\text{Al}_2\text{O}_3$  ceramic. [single-column fitting]

#### 4.4 Evaluation of fragments size by tomography

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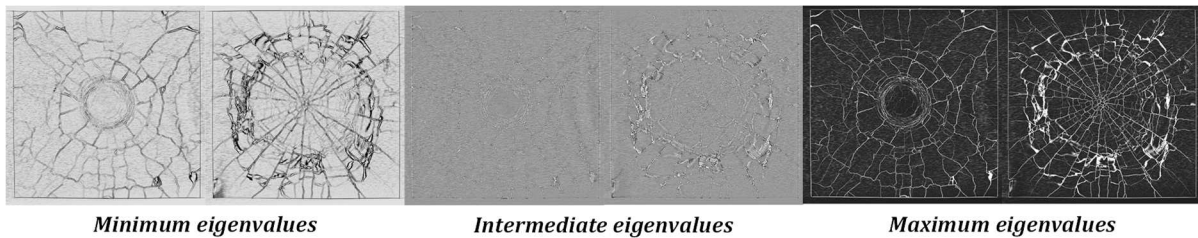
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In this part, the fragments distribution is analysed. The tomographic greyscale images are rescaled in Fiji (an open-source software program for image analysis) using a bilinear interpolation to reduce the size and so the processing time of the multi-image, passing from  $1800 \times 1800 \times 400 \text{ px}^3$  to  $900 \times 900 \times 200 \text{ px}^3$ . Then, the images are loaded in a Python



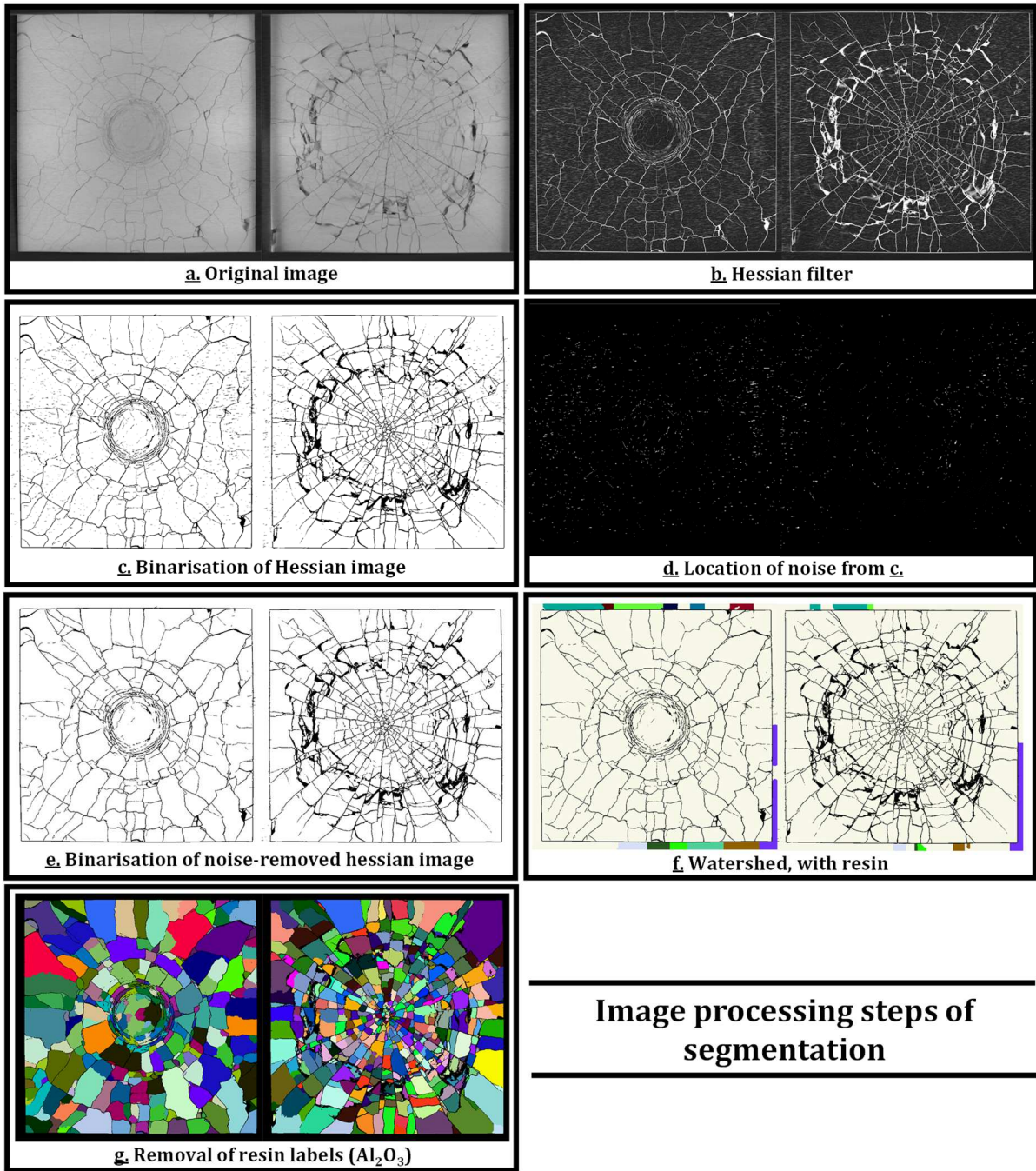
461 environment to be treated with the imported SPAM [56] package. The processing steps  
 462 applied to the greyscale images as already used in Forquin and Andò [17] are the following:  
 463 first, a 3D Sobel operator is employed for the detection of cracks. Next, a 3D median filter  
 464 (reduces noise), a thresholding (“black-and-white” two-value binarised image), a 3D marker-  
 465 based watershed algorithm on the Euclidean distance (segmentation of fragments) followed  
 466 by a labelling (assigning a value for each separated fragments), and a manual label merging to  
 467 join non-physically separated fragments and compensate over-segmentation, are applied. The  
 468 same *post-treatment* methodology as in [17] was considered in the present work with the  
 469 following change: a hessian filter is rather applied first and foremost to allow a proper  
 470 separation of the different fragments. As shown in Figure 10, the maximum hessian filter  
 471 better discriminates all the cracks.



472 *Minimum eigenvalues* *Intermediate eigenvalues* *Maximum eigenvalues*  
 473 **Figure 10.** Hessian-filtered images of  $\text{Al}_2\text{O}_3$  for different eigenvalues components. [2-column fitting]

474 Then, the filtered images are binarised with threshold parameter, but the resulting  
 475 image being noisy, a function is used to detect the smallest labels (considered as noise) in  
 476 order to delete them. Finally, SPAM owns a label toolkit to rapidly create labelled images  
 477 thanks to a watershed for separating the single interconnected solid phase into individual  
 478 particles [49]. However, in the watershed process, the glue gets also labelled into different  
 479 fragments, so a manual operation is required to delete them. To give an idea to the reader,  
 480 after 7 (9 for MAINa) manual iterative operations including at maximum 5 to 10 labels, all the  
 481 glue was managed to be removed. The different steps are reported in Figure 11. The  
 482 fragments are coloured at the end with a colour panel (“glasbey inverted”) plugin. While the  
 483 code seems to be satisfactory for  $\text{Al}_2\text{O}_3$ , a visual inspection in Figure 12 shows slight over-

484 segmentation of the largest fragments in MAINa, as pointed out by red arrows on some  
485 fragments. This issue was already encountered by Forquin and Andò [17]. To counter-act this,  
486 an additional step is added that consists in a manual step of label merging that can be  
487 automatized with a SPAM function named  
488 “label.contacts.detectAndFixOversegmentation(*lab*, *nVoxThreshold*)” (with *lab* the multi-  
489 array matrix image after the “g” step and *nVoxThreshold* the number of voxel that defines a  
490 contact). The new comparison is shown in Figure 13.

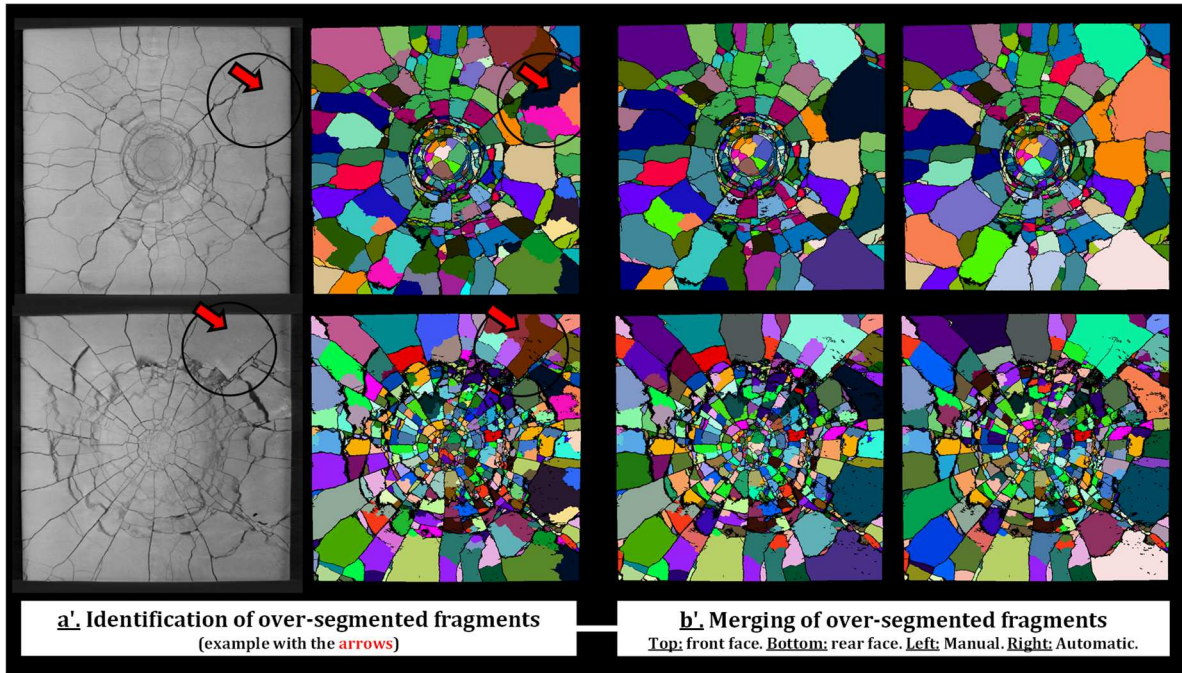


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**Figure 11.** Processing steps of segmentation, example of  $\text{Al}_2\text{O}_3$  ceramic target. [2-column fitting OR 1-page fitting]

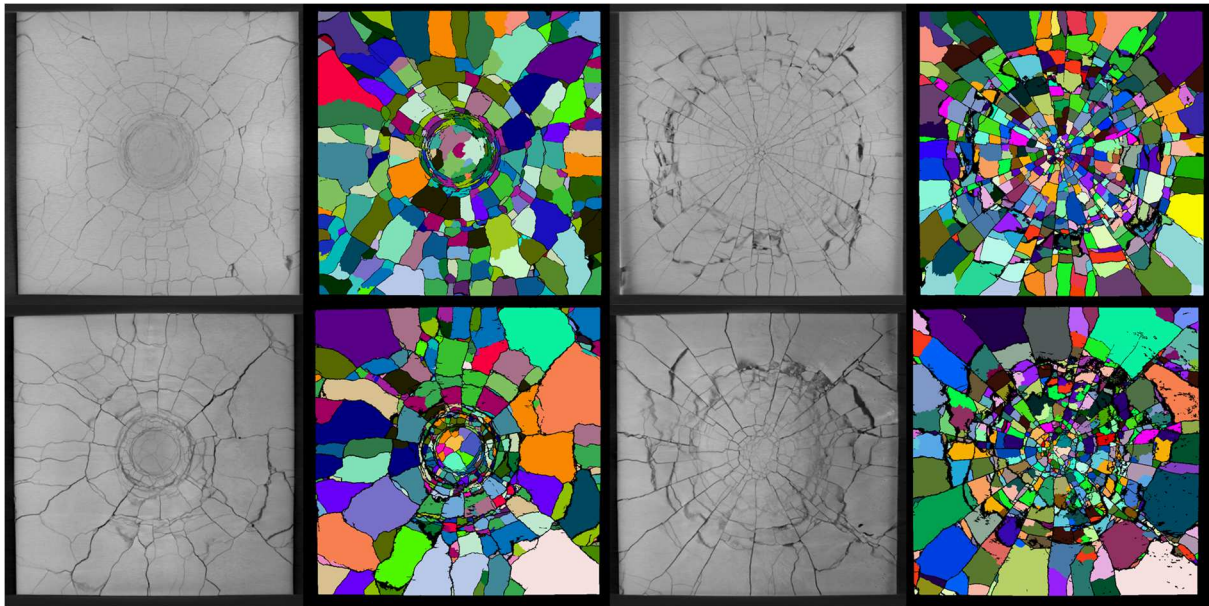


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**Figure 12.** Comparison between tomographic and segmented images, for MAINa ceramic target. [2-column fitting]



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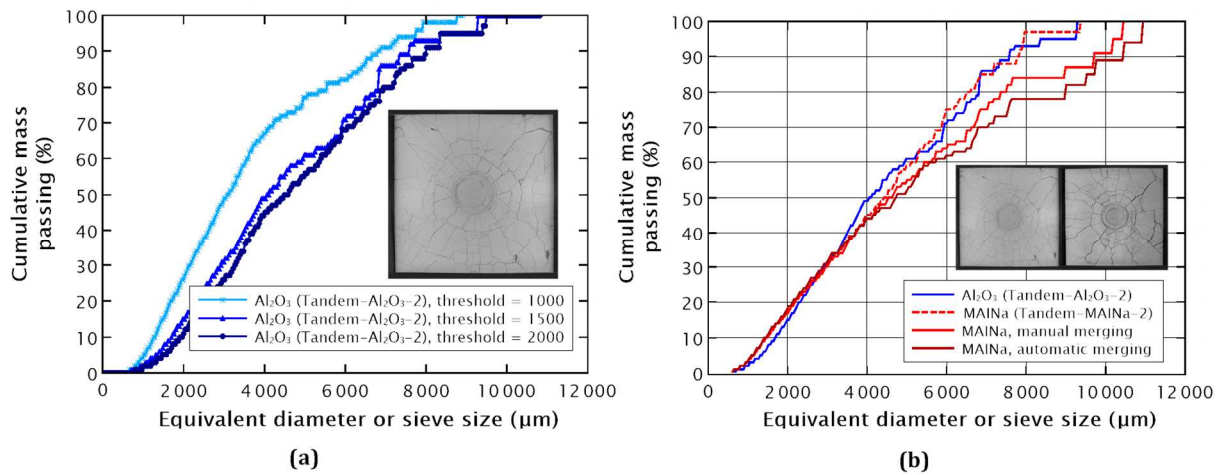
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**Figure 13.** Final comparison between tomographic and segmented images for  $\text{Al}_2\text{O}_3$  (top) and MAINa (bottom, automatic merging), after treating the over-segmentation of MAINa with a Python code. [2-column fitting]

Three threshold values from the hessian images were considered to investigate the influence of this parameter on the fragments distribution. As it can be seen in Figure 14 (a), the 1000 cut-off level provides too much noise to represent correctly the fragments

503 distribution. On the other hand, cut-off levels of the 1500 and 2000 lead to similar trends;  
504 finally, the value 1500 was chosen. The cumulative distribution of fragments, classified by  
505 ascending size just as sieving as illustrated in Figure 14 (b), gives an indication on the  
506 fragmentation intensity in both  $\text{Al}_2\text{O}_3$  and MAINa ceramic tiles. In average, the fragments  
507 generated in MAINa seem to be bigger than in  $\text{Al}_2\text{O}_3$  in an appreciable way. Below 4 – 6 mm,  
508 which corresponds to the fragmentation produced at the highest strain-rates (central area of  
509 the target), the two curves are very closed. On the other hand, above 4 – 6 mm, which  
510 corresponds to the fragments generated at the lower strain-rate (peripheral area of the target),  
511 the difference can be considered as significant. Figure 14 (b) confirms the visual inspection  
512 and the quantitative estimation of cracking density carried out on the tomographic images  
513 (Figure 7). Indeed, the main difference between  $\text{Al}_2\text{O}_3$  and MAINa concerns the larger  
514 fragments, which can be explained by the difference of fracture-toughness between both  
515 materials. This result supports the interest of MAINa in the case of multi-hit configurations.  
516 Indeed, according to STANAG 4569 (edition 3) [57] (a Standardization Agreement defined  
517 by the North Atlantic Treaty Organization (NATO)), in multi-hit configurations applied to  
518 non-transparent armour, the second impact is located at a distance of 25 millimetres away  
519 from the first impact. Conversely, in the case of single impact, the interest of MAINa would  
520 still need to be confirmed.



521 (a) Cumulative mass distribution of fragments as function of three values of threshold for Al<sub>2</sub>O<sub>3</sub>. (b)

522 Particles mass distributions deduced from *post-mortem* tomography analysis. [2-column fitting]

523

524 Two cross sectional views (Figure 15 (a)) of the ceramics are exposed in Figure 15 (b),

525 showing the middle part of the targets (medium slices). In the lateral views, an excess of resin

526 at the top of each sample is noticed and corresponds to the low intensity due to the lower

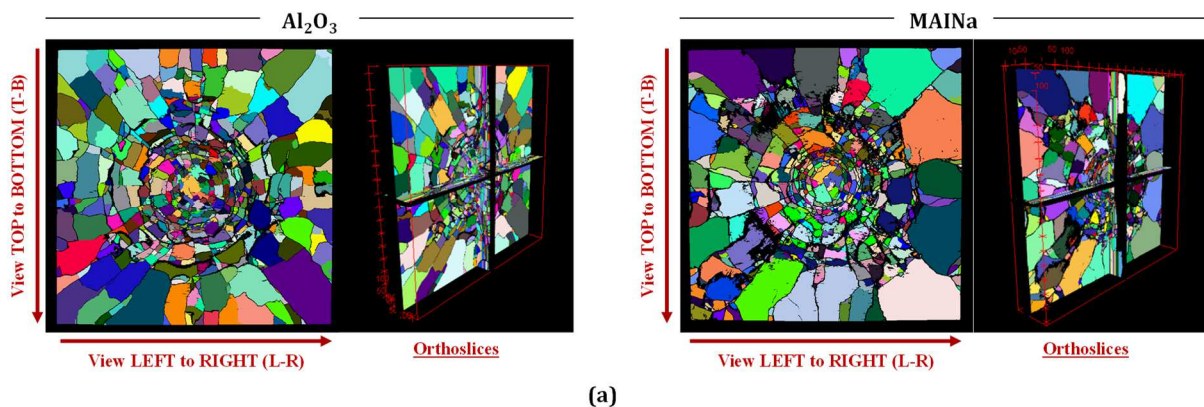
527 density of the resin compared to that of ceramics. In addition, a slight bending deformation of

528 the fragmented ceramic plate is noted and seems to be more important in nacre-like ceramic.

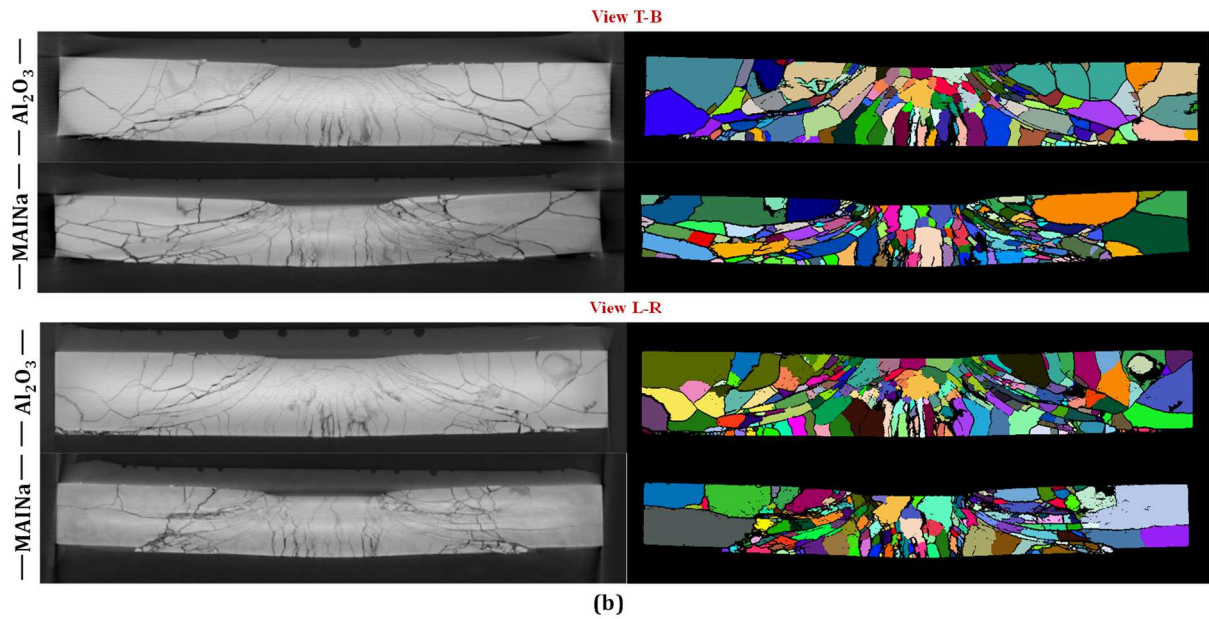
529 The most important point is that fragments in MAINa seem to be more distributed with a

530 horizontal way (following platelets which are distributed horizontally) whereas those in Al<sub>2</sub>O<sub>3</sub>

531 seem to be more distributed along a longitudinal or inclined orientation.



532



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**Figure 15.** (a) Definition of cross-section views. (b) *Post-mortem* visualisation of two cross-section views of the  $\text{Al}_2\text{O}_3$  and of the MAINa, from tomographic images next to their segmented images. [2-column fitting]

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## 5. Results of penetrating impact experiment

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### 5.1 Visualisation of fragmented ceramics subjected to penetrating impact

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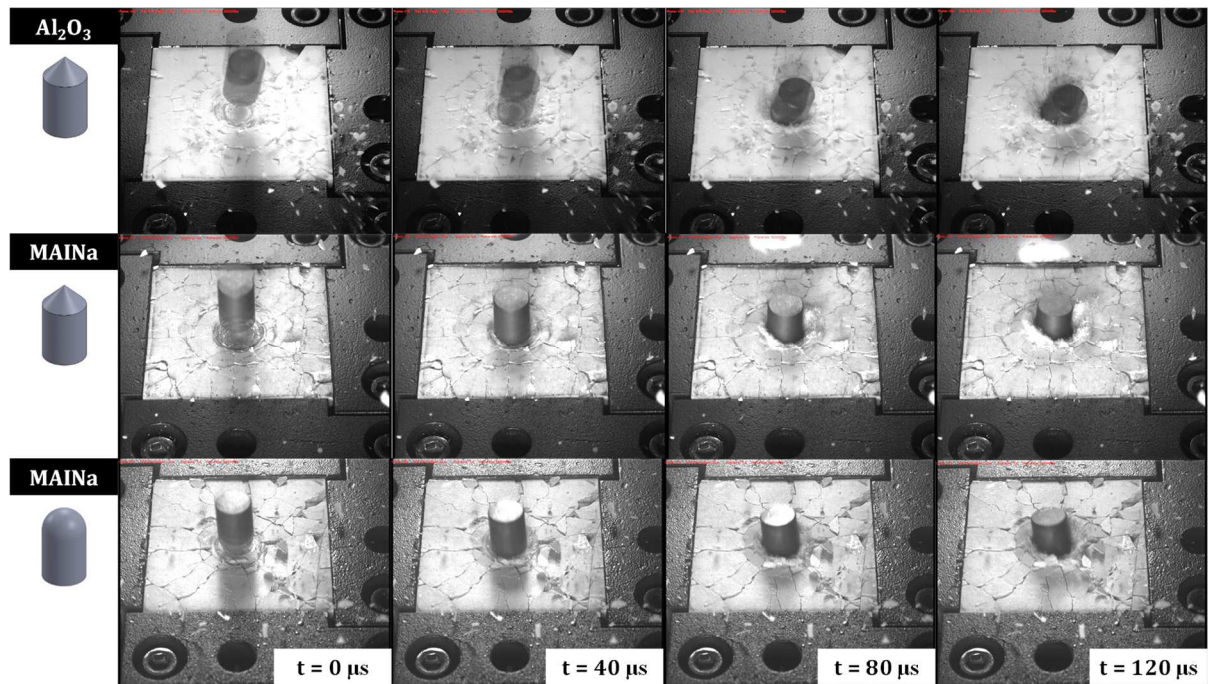
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Several pictures captured at different times with the Kirana® ultra-high-speed camera are presented in the Figure 16. The time  $t = 0$  is established when the projectile is about to reach the ceramic target although the exact position is difficult to define because of the angle of the camera. For better visualisation, the luminosity is amplified by 40% for  $\text{Al}_2\text{O}_3$  and by 20% for both MAINa samples. The tilt angle of the projectile before impact is relatively small. During the first 120 microseconds of the selected time interval, the fragments around the point of impact are ejected while fragments farther from the point of impact remain in their initial position in the recording time.



546

547 **Figure 16.** Penetrating test visualised with ultra-high-speed imaging at four different times, impacted at 175 m/s  
 548 with the conic end projectile ( $\text{Al}_2\text{O}_3$ , MAINa) and impacted at 145 m/s with the hemispheric end projectile  
 549 (MAINa). Samples, from top to bottom, are: Tandem- $\text{Al}_2\text{O}_3$ -1, Tandem-MAINa-1b, Tandem-MAINa-1b Hemi.

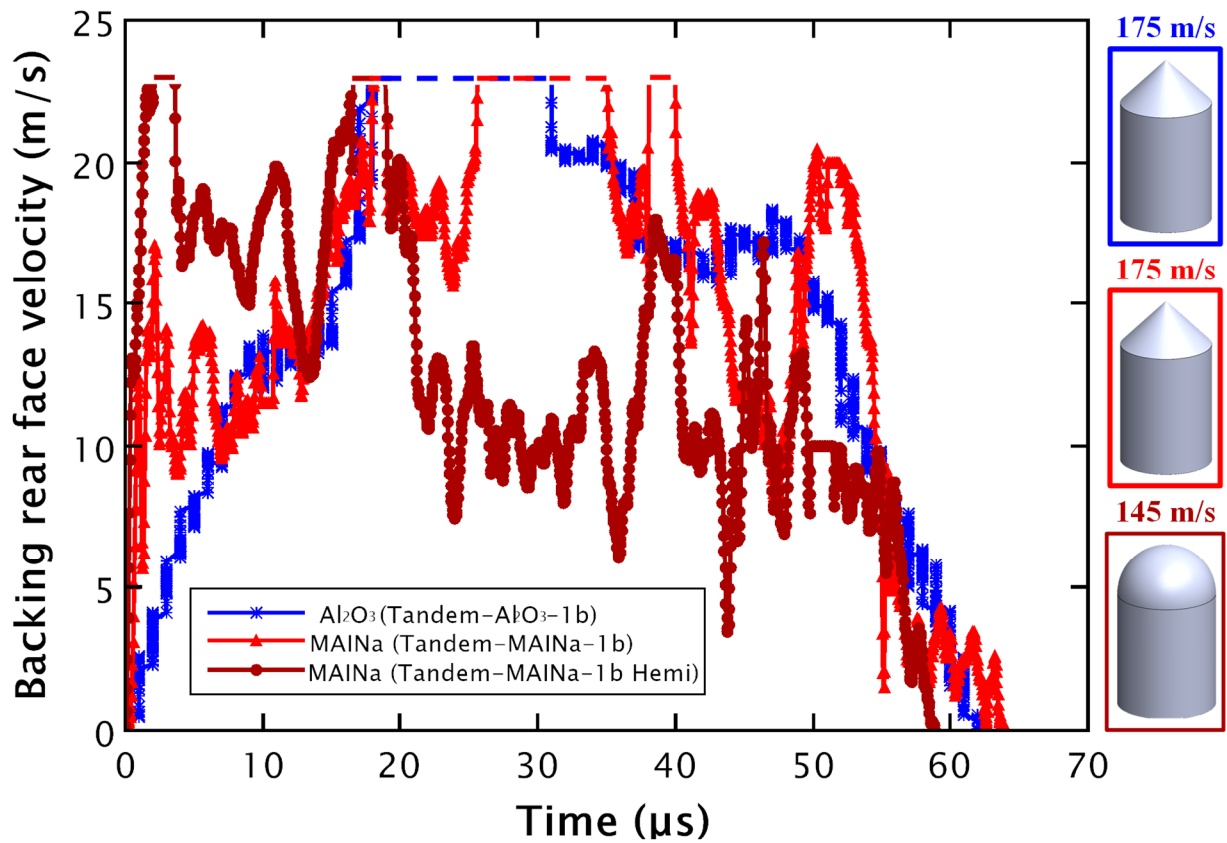
550

[2-column fitting]

551

The velocity profiles measured on the rear surface of the aluminium backing are  
 552 plotted for the two microstructures in Figure 17. The two tests performed with conical end  
 553 projectiles show that, during the first microseconds, the increase of velocity is much faster  
 554 with MAINa than in  $\text{Al}_2\text{O}_3$  ceramic. The higher acceleration observed with MAINa, which  
 555 translates into a higher initial tangent, results from a higher resistance to penetration with the  
 556 nacre-like ceramic. Besides, the test performed with a hemispherical end projectile on MAINa  
 557 shows a much higher amplitude of particle velocity during the first tens of microseconds of  
 558 the test, which demonstrates the influence of the projectile nose shape on the penetration  
 559 resisting force exerted by the projectile against the fragmented media.





560

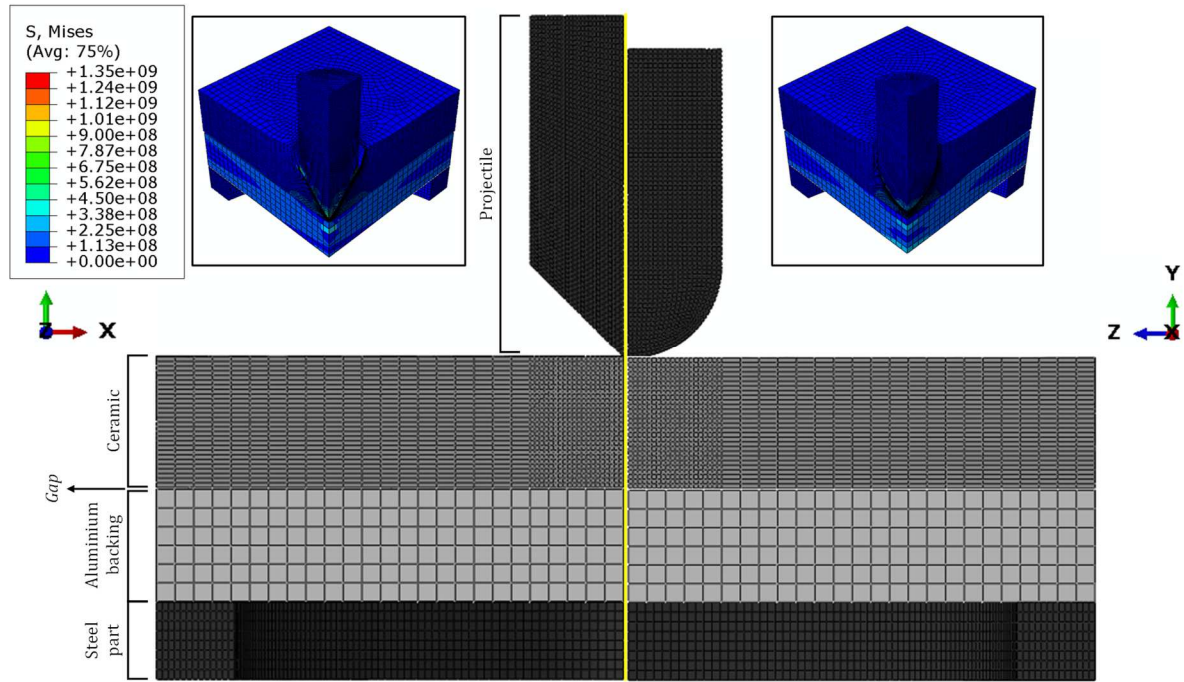
561 **Figure 17.** Free surface velocities at the rear face of the aluminium backing for the tests performed with the  
 562 different alumina ceramics and the two projectile shapes. [single-column fitting]

563 5.2 Numerical simulation: identification of Drucker-Prager parameters

564 The penetrating impact, that is the second stage of tandem test, is simulated with  
 565 ABAQUS. The impact velocity is set to 175 m/s (conical end impactor) and 175 and 145 m/s  
 566 (hemispherical end impactor). Two symmetry conditions are considered (quarter model).  
 567 These symmetry planes force the projectile on a straight penetration path, contrary to what  
 568 happened in the full experiment, but this assumption can be considered as reasonably true in  
 569 the considered interval of time (roughly 60  $\mu$ s as projectile rotation is clearly observed only  
 570 after 80  $\mu$ s, *cf.* Figure 16). The mesh is made of C3D8R elements (8-node linear brick,  
 571 reduced integration, hourglass control), with a mesh size of 0.25 mm for the projectile, 0.25  
 572 mm to 1.00 mm for the ceramic, 1.00 mm for the aluminium backing and 0.50 mm for the  
 573 steel part. As presented in Figure 18, the meshing technique is structured for all constituents,

574 while for the ceramic it is structured under the projectile radius. Material parameters for the  
575 steel projectiles, the steel backing and the aluminium plate are reported in Table 4. A friction  
576 coefficient of 0.1 is applied to all surfaces in contact, using the general contact algorithm. The  
577 Figure 18 displays the geometries of the numerical model using both types of projectile. The  
578 displacement is set to zero to all nodes belonging to the bottom surface of the steel part to  
579 prevent displacement along the Y-direction ( $u_y = 0$ ). An element control option called  
580 “distortion control” is set on the ceramic to prevent solid elements from inverting or distorting  
581 excessively. This technique is used in many analysis with large compressive and shear  
582 deformations [58]. The value of the distortion length ratio parameter, between 0 and 1, should  
583 not be high enough to not change the physical response of the material. The parameter is  
584 finally set at 0.3 *i.e.* a constraint activates as soon as an element under uniaxial compression  
585 undergoes 70% of nominal strain, thus reducing or even removing convergence issue. Upon  
586 examination of the energies provided by the explicit finite element simulations it is concluded  
587 that no major instabilities arise as the simulations progress. For instance, in the case of  
588 alumina target (Drucker-Prager model,  $d_0 = 80$  MPa,  $\beta = 16^\circ$ ,  $\psi = 0$ ) impacted by a conical  
589 projectile at  $V = 175$  m/s (no tilt) it is noted that, whereas the Total Energy is keep perfectly  
590 constant during the calculation, at time  $T = 75$   $\mu$ s the Kinetic Energy tends to 0 (projectile  
591 arrest) and the Internal Energy that represents (90.4%) of the total energy is mainly composed  
592 of Inelastic Dissipated Energy (78.2%), Elastic Strain Energy (6.5%) and Artificial Strain  
593 energy (energy stored in hourglass resistances) (4.7%). Finally, at that time, Frictional  
594 Dissipated Energy represents (8.9%) and Distorsion Control Dissipation Energy is only  
595 (1.1%).

596



597

598 **Figure 18.** Numerical model of penetrating impact in tandem with both projectile shapes, with equivalent (von  
 599 Mises) stress fields within Al<sub>2</sub>O<sub>3</sub> impacted at 175 m/s at the end of the simulation (100 μs). [1.5-column fitting]

600 The constitutive model used to represent the fragmented ceramic is the Drucker-Prager  
 601 (DP) model, as already employed by Zinszner [10] for simulating the response of different  
 602 SiC ceramics subjected to tandem test, with the aim to identify the DP parameters ( $d_0, \beta$ ) by  
 603 inverse approach. The constitutive equation (4) describes the equivalent stress  $\sigma_{eq}$  as function  
 604 of the pressure  $P$  in the DP model, in which the confined strength is related to the applied  
 605 pressure. The parameters  $d_0$  and  $\beta$  are respectively the cohesion and the (internal) friction  
 606 angle of the material.  $d_0$  is equal to the yield strength when  $P = 0$ , and the expression is  
 607 limited to a maximum value denoted  $S_{max}^f$  for the highest pressures.

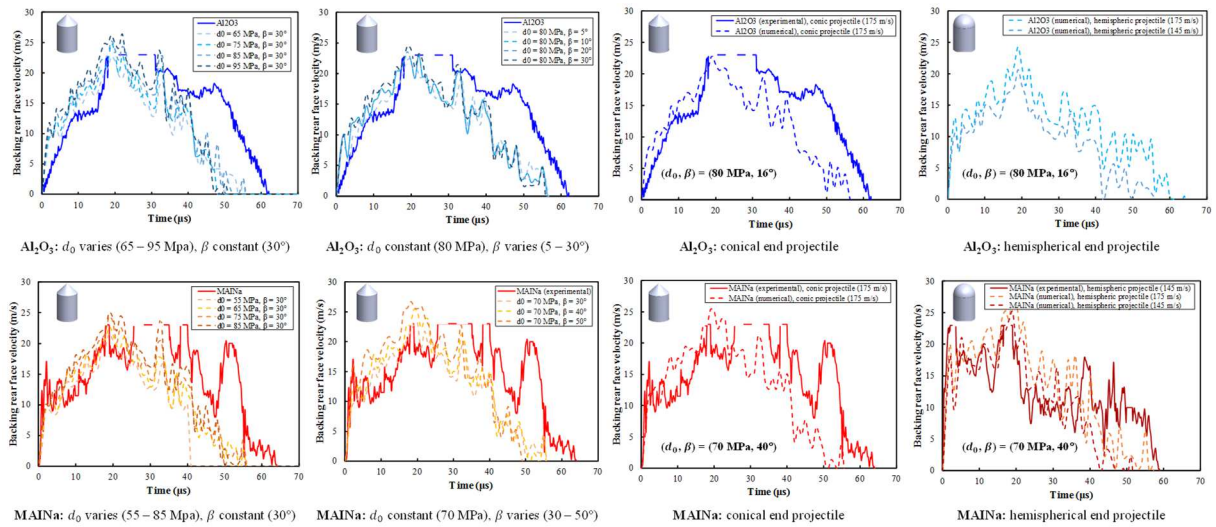
$$\sigma_{eq} = \min(d_0 + P \cdot \tan(\beta), S_{max}^f) \quad (4)$$

608 A dilation angle  $\psi$  is also defined to represent the volume increase during shear  
 609 deformation. The DP model with no dilation ( $\psi = 0$ ) is employed in the present study. The  
 610 DP constant parameters for the studied SiC (Hexoloy SA®) are  $d_0 = 35 \text{ MPa}$  and  $\beta = 30^\circ$ .  
 611 The identification technique is the following:  $\beta$  is first set to  $30^\circ$  and  $d_0$  is changed to obtain

612 an appropriate velocity profile that best matches to the experimental data. Then, the  $d_0$  is fix  
613 and different  $\beta$  are tested until the best fit is obtained. Nonetheless, as mentioned in the  
614 introduction, the parameter  $\beta$  may not be unique and its determination might be addressed by  
615 a heuristic approach. For an impact velocity established at 175 m/s, the hydrostatic pressures  
616 obtained numerically is about 475 MPa in fragmented  $Al_2O_3$  and 745 MPa in fragmented  
617 MAINa with conical end projectile and below 225 MPa ( $Al_2O_3$ ) and 250 MPa (MAINa) with  
618 hemispherical end projectile. The numerical input parameters considered in the DP model are  
619 indicated in Table 5. The Figure 19 illustrates the identification strategy with different  
620 numerical curves considering various parameters of DP model, along with a comparison  
621 between experimental and numerical results for both ceramics. The numerical simulations  
622 support the experimental findings. Nonetheless, since the identification relies on an inverse  
623 approach, this methodology with tandem is restricted to the considered Drucker-Prager model  
624 which proposes a linear increase of strength with pressure.

Material	Plasticity		Hardening behaviour type	Flow stress ratio $\sigma_{flow}^*$ (-)	$\beta$ (°)	$\psi$ (°)
	$d_0$ (MPa)	$\varepsilon_p$ (%)				
<b>Ceramic</b> ( $Al_2O_3$ and MAINa)	$T_0$ <i>identify</i>	0	Shear	1	$T_0$ <i>identify</i>	0

625 **Table 5.** Parameters of Drucker-Prager model used in ABAQUS (the density and elastic parameters of each  
626 material are provided in Table 1 and 2, respectively).



627

628

629

**Figure 19.** Identification of Drucker-Prager model parameters and validation by comparison with experimental results. [2-column fitting]

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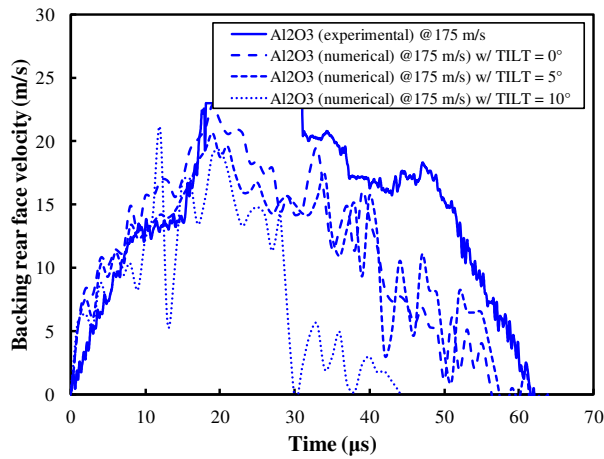
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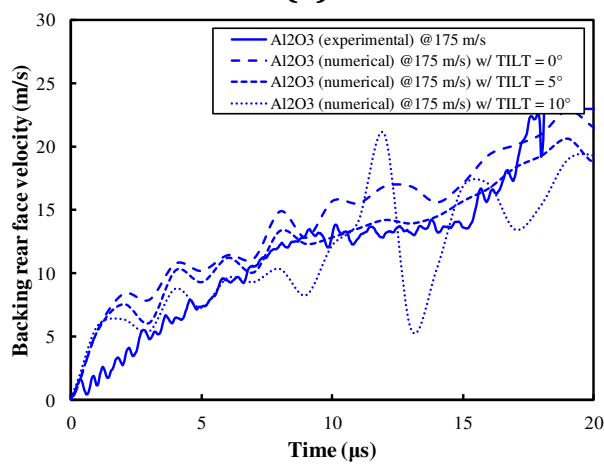
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639

The identification of a single set of  $d_0$  and  $\beta$  parameters remains delicate. However, the following parameters seem to be consistent:  $d_0 \sim 65 - 95$  MPa for  $\text{Al}_2\text{O}_3$  and  $\sim 55 - 85$  MPa for MAINa;  $\beta \sim 5 - 30^\circ$  for  $\text{Al}_2\text{O}_3$  and  $30 - 50^\circ$  for MAINa. Further numerical simulations were led to assess the influence of the tilt angle of the projectile, up to  $10^\circ$  with conical projectile, assuming that tilt angle did not exceed  $10^\circ$  in the tests. This, to verify if the previous statement, according to which the projectile rotation observed in the Figure 16 has no influence on the identification of DP model parameters, is valid. The numerical results, depicted in Figure 20, strengthen the fact that the tilt angle has a minor effect, at least as long as it does not exceed  $10^\circ$ . In conclusion, the different initial slopes on the rear face velocity profiles cannot be only due to a substantial tilt of the projectile.



(a)



(b)

640

641 **Figure 20.** Numerical particle velocity at the rear face of the aluminium backing, compared to  
 642 experimental data, considering the impact of a conical projectile against a fragmented alumina  
 643 without tilt or with tilt angles of 5° and 10°. Results are shown (a) from 0 to 70 μs and (b)

644

from 0 to 20 μs. [single-column fitting]

645

In comparison with the identification proposed by Zinszner's [10], it can be remarked

646

that:

647

- $d_0$  for both alumina ( $\text{Al}_2\text{O}_3$  and MAINa) materials is seen to be higher than in [10] so

648

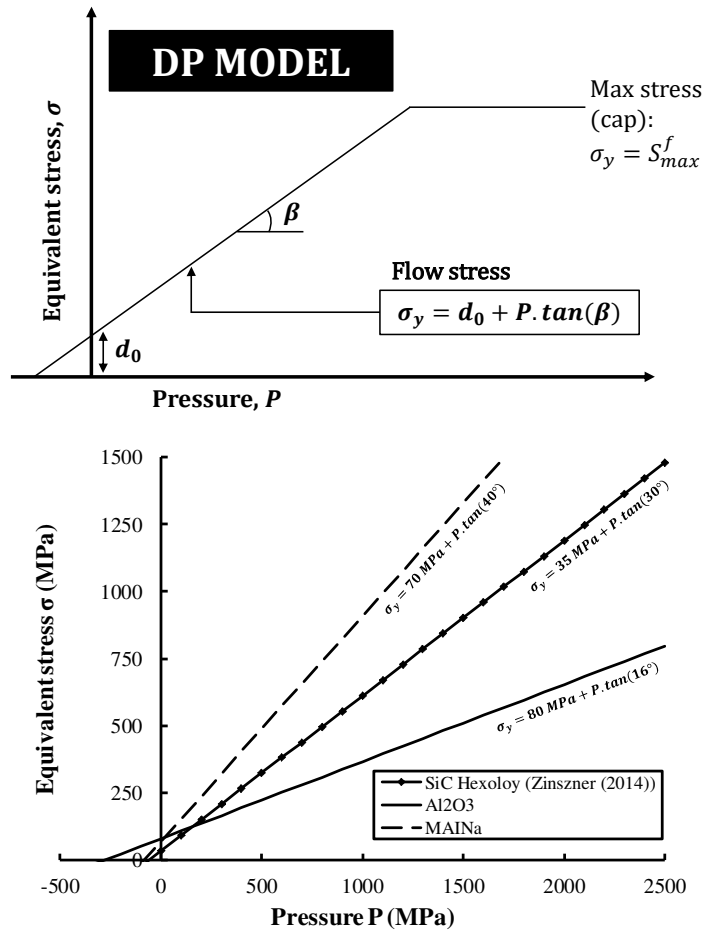
both fragmented alumina materials seem to be more resistant to penetration compared

649

to a fragmented SiC.

650 - Secondly: the identified friction angle  $\beta$  of both alumina ceramics is higher with  
 651 MAINa than in [10], and lower with  $\text{Al}_2\text{O}_3$ , as illustrated in Figure 17 compared to  
 652 Figure 1 (c).

653 The Drucker-Prager plasticity yield strength for Hexoloy (SA® grade),  $\text{Al}_2\text{O}_3$  and MAINa  
 654 can be compared in Figure 21.



655  
 656 **Figure 21.** Description of DP model in terms of strength, with plot using the DP parameters ( $d_0$  and  $\beta$ ) for three  
 657 materials. [single-column fitting]

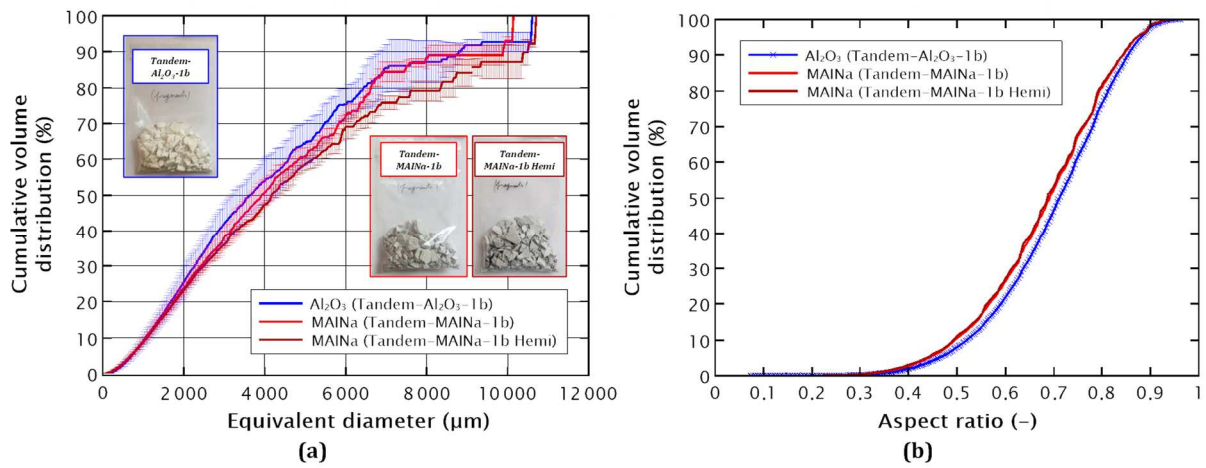
658 The explanation of why MAINa provides better performances in tandem test than  
 659  $\text{Al}_2\text{O}_3$  and Hexoloy SA® SiC may be attributed to the fragments size shape and orientation  
 660 just beneath the impact area of normal impact. As illustrated in Figure 15,  $\text{Al}_2\text{O}_3$  fragments  
 661 are smaller beneath the projectile contact surface and more vertical so they can be more easily  
 662 displaced during the penetrating impact.

### 663 5.3 Fragments size analysis by sieving

664 Finally, next to the second impact, the fragments of each ceramic are recovered for  
665 further analysis using a dynamic microscopic analyser (equipment: ANALYSETTE 28®  
666 ImageSizer from FRITSCH) for sieving. The fragments are poured on a metallic rail that  
667 vibrates at a certain frequency. Fragments fall within a measurement chamber, between a  
668 camera and a LED (Light-Emitting Diode) stroboscope, towards a collecting vessel.  
669 Additionally, thanks to the camera, interesting information can be extracted such as: aspect  
670 ratio, circularity or convexity. The distribution of fragments is reported in Figure 22 (a). First,  
671 it is observed that fragments impacted at 145 m/s with hemispherical end projectile are  
672 slightly larger than those impacted at 175 m/s with conic-end projectile (comparison between  
673 samples “Tandem-MAINa-1b” and “Tandem-MAINa-1b Hemi”). It can be supposed that the  
674 conic-end projectile, impacting at higher impact speed than the hemispherical-end projectile,  
675 might have slightly changed the fragments size distribution near the impact point. In addition,  
676 whereas the distribution of fragments in “Tandem-Al<sub>2</sub>O<sub>3</sub>-1b” after penetrating impact is  
677 similar to the one obtained in “Normal-Al<sub>2</sub>O<sub>3</sub>-2” from tomography (Figure 14 (b)) measured  
678 next to normal impact, the fragments size distribution seems to be changed with MAINa.  
679 Indeed, smaller fragments are noted in “Tandem-MAINa-1b” than in “Normal-MAINa-2”.  
680 Indeed, while smaller fragments were observed from tomography with MAINa compared to  
681 Al<sub>2</sub>O<sub>3</sub>, it is the opposite next to penetrating impact. It would suggest that a part of MAINa  
682 fragments would be crushed during the penetrating impact, due to the higher stresses  
683 supported by MAINa fragments during the second impact. The standard deviation is also  
684 reported in the curves of Figure 22 (a), the fragments of MAINa remain larger than the  
685 fragments of Al<sub>2</sub>O<sub>3</sub>, even if the gap is within the margin of error and the trend should be  
686 confirmed. Furthermore, the Figure 22 (b) reveals the aspect ratio  $AV_F$  of the fragments,  
687 which is the ratio between the maximum (Feret) diameter to the minimum (Feret) diameter



688 (the standard deviation is not reported as it tends to zero), from which two conclusions can be  
 689 drawn. First, the  $AV_F$  of MAINa is nearly the same whatever the impact velocity (175 or 145  
 690 m/s) and projectile shapes used in penetrating impact, which tends to demonstrate that the size  
 691 and shape of fragments remained unchanged before and after the second impact. Second, the  
 692  $AV_F$  of MAINa is overall smaller than that of  $Al_2O_3$ , which means that the fragments of  
 693 MAINa are more elongated than those of  $Al_2O_3$  (because when  $AV_F$  tends to 1, the fragment  
 694 tends to be circular). This difference in fragments shape is supposed to explain the higher  
 695 strength of MAINa under penetrating impact test.



696  
 697 **Figure 22.** (a) Fragments distribution in terms of cumulative volume as function of the equivalent diameter. (b)  
 698 Aspect ratio of the fragments after the second impact. [2-column fitting]

## 699 6 Conclusion

700 Two ceramic microstructures, a conventional alumina ( $Al_2O_3$ ) and a nacre-like alumina  
 701 (MAINa), are compared in terms of dynamic fragmentation under impact. The nacre-alumina  
 702 ceramic is a BM material conferring improved toughening mechanisms. The cracking  
 703 behaviour of both materials is investigated by means of tandem impact experiments and  
 704 micro-computed tomographic analysis. The tandem impact configuration consists in a first  
 705 impact of a flat-end projectile against the ceramic plate sandwiched between a steel sheet and  
 706 an aluminium backing. This test produces an intense fragmentation of the ceramic plate while

707 keeping the fragments in place. The second impact is a penetrating impact against the  
708 fragmented ceramic backed with the aluminium plate. A numerical simulation using the  
709 Drucker-Prager model is employed to identify the plasticity parameters of the model based on  
710 the experimental backing rear face velocity captured after the second impact of tandem test.

711 The fragmentation pattern of the ceramic plates was analysed after the normal impact through  
712 a tomography scan. The cracking density of MAINa is estimated to be lower than the cracking  
713 density of  $\text{Al}_2\text{O}_3$  by a factor that can go around 2 and 3. The fragments size analysis indicates  
714 that the fragments of MAINa are slightly larger than those of  $\text{Al}_2\text{O}_3$  around the point of  
715 impact and predominantly larger far from the impact point. The farther from the impact point,  
716 the greater the difference of fragments size between the two microstructures. Thus, MAINa  
717 microstructure could be more interesting in the case of a second impact. According to the  
718 penetrating impacts, the pre-fragmented MAINa clearly appears more resistant than  $\text{Al}_2\text{O}_3$ ,  
719 which suggests that the fragmentation pattern of MAINa could play an important role on its  
720 penetration strength during impact when the ceramic is already fragmented. Based on a  
721 numerical approach, in average, the following Drucker-Prager parameters identified are: ( $d_0 \sim$   
722  $80 \text{ MPa}$ ,  $\beta \sim 16^\circ$ ) for  $\text{Al}_2\text{O}_3$  and ( $d_0 \sim 70 \text{ MPa}$ ,  $\beta \sim 40^\circ$ ) for MAINa.

723 According to tomographic analysis and additional observations (SEM, dynamic microscopic  
724 analyser), the difference of fragmented behaviour between both materials results from a  
725 combined effect of a difference in fragments size, fragments orientation and fragments  
726 anisotropy. Indeed, compared to  $\text{Al}_2\text{O}_3$ , MAINa fragments are larger, horizontally oriented  
727 (they follow the platelets orientation) and more anisotropic (aspect ratio of fragments shape  
728 diverges from 1).

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753 **Declaration of interest**

754 The authors declare no conflict of interest and they have no known competing financial  
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