Influence of plate material on the contact strength of Li$_4$SiO$_4$ pebbles in crush tests and evaluation of the contact strength in pebble–pebble contact

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Abstract

Lithium orthosilicate (Li$_4$SiO$_4$) pebbles are considered to be a candidate as tritium breeder in the helium cooled pebble bed (HCPB) blanket. Their contact strength is of concern since they might be crushed in the blanket under thermomechanical load. Crush tests for single pebbles are carried out to evaluate their strength. In these tests, single pebbles are crushed by a pair of parallel plates. In this way, the crush load, i.e., the maximum contact force between pebble and plates before failure of the pebble occurs, can be obtained. In this study, the influence of the plate material used in the crush tests on the crush load is investigated. Single Li$_4$SiO$_4$ pebbles are crushed by plates made of aluminum alloy (AL) and tungsten carbide (WC), respectively. Two corresponding crush load distributions are obtained. A probabilistic strength model is proposed to explain the influence of the plate material. Moreover, this model will be used to predict the contact strength of pebbles in pebble–pebble contact. The pebble–pebble contact strength can be used to investigate the influence of pebble failure on the thermomechanical response of the pebble beds.

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1. Introduction

Pebble beds are integral parts of the fusion reactors as solid breeder and neutron multipliers in the HCPB blanket [1,2]. The blanket contains two types of pebbles, ceramic breeder (lithium compound) and neutron multiplier (beryllium). During the operation of the fusion reactor, pebbles will expand because of high temperatures in addition to thermal stresses due to thermal mismatch between the pebble material and the container material. Both effects may lead to the failure of pebbles. In other words, pebbles might be crushed into several fragments. It is foreseen that the failure of pebbles will have, at least, three consequences. At first, the fragments can constrain or even block the flow of purge gas in pebble beds. As a result, the tritium bred in a pebble bed cannot be brought away by the purge gas. Secondly, the local failure of pebbles can give rise to production of local hot spots [3]. Finally, pebble failure will affect the overall thermomechanical response of the pebble bed. Therefore, the knowledge of the strength of ceramic pebbles is necessary for the safe and reliable design of the HCPB blanket.

There have been some attempts to study the strength of ceramic pebbles by crush tests in the past [3–7]. In these crush tests, single pebbles were crushed by two parallel plates. The crush load, i.e., the maximum contact force between pebble and plates before failure of the pebble, was measured. The plate influence on the crush load was not taken into account in the
studies mentioned above. Different compression plates resulted in different values reported in literature even for similar pebble materials [6,7]. Consequently, the crush load cannot represent the pebble strength since it also depends on the plate material. This work reports the crush load for lithium orthosilicate (Li4SiO4) pebbles in air using a pair of aluminum (AL) plates and tungsten carbide (WC) plates, respectively. The crush load will be given in the form of probabilistic distribution for a number of samples. Comparison between the two distributions clearly shows the influence of plate material on the crush load. Finally, the crush load distribution for pebbles in dry inert gas environment crushed by BK7 glass will be also reported.

The objective of the crush tests performed in this work is not only to show the influence of plate material on crush loads but also to provide experimental data for the analysis of the strength of pebbles under pebble–pebble contact, which can be imported into Discrete Element Method (DEM) to analyze the overall crush probability of pebble beds [8,9]. The reliability of such an analysis, which is important to the HCPB blanket design, will depend on the accuracy of pebble–pebble contact strength. It is, however, difficult to carry out crush tests for pebble–pebble contact. This investigation provides a systematic method to study the pebble–pebble contact strength. At first, two or more crush tests for plate–pebble contact have to be conducted for different plate materials with various types of mechanical properties, like AL and WC plates in this study. Secondly, a contact strength model for pebbles which can explain the plate influence on crush loads has to be found or established. Such a probabilistic strength model is proposed in this work and validated by two crush load distributions. Finally, pebble–pebble contact strength can be derived using the strength model and experimental data from crush tests. The crush load distribution obtained for the BK7 plates will be used to predict the pebble–pebble contact strength where the influence of the relative humidity of the atmosphere is excluded.

This paper is organized as follows. The experimental setup for crush tests and material parameters are presented in Section 2. Crush load distributions for different plates are shown in Section 3. Subsequently, a probabilistic strength model is proposed in Section 4 to explain the plate influence on the strength of pebbles. Pebble–pebble contact strength is derived in Section 5 based on the model and on crush load distributions from crush tests. A discussion of the strength model is presented in Section 6. Finally, conclusions are drawn in Section 7.

2. Experimental testing conditions

Crush tests were carried out with a pair of plates where both were made either of aluminum (AL) or tungsten carbide (WC), respectively. The two different materials were selected in view of the different resultant stiffnesses of the corresponding plates so that there would be a significant difference between crush load distributions of pebbles obtained from the two different types of plates.Conditioned Li4SiO4 pebbles from a certain batch denoted as OSI 07/1 (see also [7]) were crushed by two plates made of the same material, AL or WC, in air. Before testing, the pebbles were exposed to air for some days so that there was enough time for the pebble material to react with moisture and CO2 in air [10]. Irrespective of the material used for the compression plates, the strength is expected to be an intrinsic property of the pebbles. Consequently, the difference between the crush load distributions solely stems from the plate material since the state of pebbles in both the tests was statistically the same. Let us mention that pebble–pebble contact which is difficult to handle in experiments is equivalent to the contact between a sphere and a rigid plate if contact friction can be ignored (the influence of friction will be discussed later). In experiments plates with finite stiffness are available, only. For the same contact force, plates with different stiffness will
result in different contact areas between pebble and plate, thus, leading to different crush loads in general. In this paper we aim at a strength model which can explain the influence of the plate material on the crush loads. This model will be able to predict the crush load for any plate material, thus characterizing the pebbles alone. In particular, the contact between rigid plates and a pebble will be described, which represents pebble–pebble contact.

The material parameters for OSi pebbles and plate materials are listed in Table 1, including the BK7 glass plates used in the crush tests at the Fusion Materials Laboratory (FML), Karlsruhe Institute of Technology (KIT). The crush tests at FML were performed in a dry inert gas environment, similar to conditions in a fusion reactor, with the exception that they were conducted at room temperature.

The experimental apparatus for different plates is shown in Fig. 1. The pebbles were placed on the plate on the left side under a microscope. The microscope (Nikon, Japan) combined with a 6.6 Megapixel CMOS camera (Pixelink, Canada) was used to examine the size and shape of pebbles and the failure form. The microscope was not intended to examine the microstructure of pebble surface. The microstructural characterization of the pebbles is reported in detail in [7]. However, such information cannot directly and quantitatively be connected to the pebble strength.

The plate on the left side was fixed and the right plate was moved horizontally towards the pebble until it failed. For this purpose a piezo actuator (Physikinstrumente, Germany) with 60 $\mu$m travel range with an integrated position sensor (resolution 1.2 nm) was used. The displacement was measured by means of this sensor and the actuator velocity was set to 0.015 mm/min. The load was measured with a ±50 N load cell (Disynet, Germany, resolution down to 25 mN). Being a ceramic material, the failure of pebbles is spontaneous and hence the crush load can be easily identified.

The setup at FML mentioned before is enclosed in a glove box in an inert gas environment, where pebbles are crushed along vertical direction by compression plates made of BK7 glass. This apparatus is not shown in this paper.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Young's modulus (GPa)</th>
<th>Poisson's ratio</th>
<th>Yield stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OSi</td>
<td>90$^a$</td>
<td>0.25$^b$</td>
<td>–</td>
</tr>
<tr>
<td>WC</td>
<td>732</td>
<td>0.22</td>
<td>2000</td>
</tr>
<tr>
<td>AL</td>
<td>70</td>
<td>0.33</td>
<td>500</td>
</tr>
<tr>
<td>BK7</td>
<td>82</td>
<td>0.206</td>
<td>–</td>
</tr>
</tbody>
</table>

$^a$ From Ref. [8].
$^b$ From Ref. [11].
The crush load probability for $F_c \leq F_{c,i}$ is $P_s(F_{c,i}) = i/(N + 1)$ where $F_c$ denotes the crush load. All crush loads $F_{c,i}$ ($i = 1,2, \ldots , N$) obtained in experiment are ranked in an increasing order, namely $F_{c,1} \leq F_{c,2} \leq \cdots \leq F_{c,N}$ where $N$ is the total number of crushed pebbles. Two crush load distributions for each type of plates are thus derived. The Weibull distribution is used to fit the experimental data, that is

$$P_s = \begin{cases} 
1 - \exp \left[- \left(\frac{F - F_u}{F_0}\right)^m\right] & F \geq F_u \\
0 & F < F_u,
\end{cases}$$

where $F_u, F_0$ and the Weibull modulus $m$ are fitting parameters.

### 3. Experimental results

The appearance of pebbles was examined by means of the microscope combined with the camera, mentioned above. The probability density functions (PDF) of the size and shape for 178 pebbles are shown in Fig. 2. The size is characterized by an average diameter, namely $D_m = (H + W)/2$, while the shape is characterized by $ho = H/W$, where $H$ and $W$ are the height (along the loading direction) and width (along the orthogonal direction), respectively, measured from the pebble picture taken before crush tests. For a pebble, the measurements of $H$ and $W$ will depend on its projection on the image of the microscopy. Since we do not take care of some specific orientations in the experiments, the resulting height and width of samples can be considered to be random.

Most pebbles have a size in the range of $0.44 \text{ mm} < D_m < 0.56 \text{ mm}$. The size distribution is quite different from the one obtained by L{"o}bbecke and Knitter [7] although both pebbles are from the same batch. The reason is that sieved pebbles were used in this work while as-received pebbles were examined by L{"o}bbecke and Knitter [7]. The sieved pebbles should have a size of $0.5 \text{ mm} < D_m < 0.56 \text{ mm}$ according to the hole size of the stacked sieves. Nearly half of the pebbles which were randomly chosen in this work had a size smaller than 0.5 mm. This indicates a certain inefficiency of the sieving method. On the other hand, most pebbles have a good sphericity since most data are around $\rho = 1$ in Fig. 2. The good sphericity of most pebbles conforms to the examination by L{"o}bbecke and Knitter [7]. The sphericity of Li$_4$SiO$_4$ pebbles is much better than that of lithium metatitanate (Li$_2$TiO$_3$) pebbles [12]. Note that pebbles with a nominal size of 0.5 mm crushed at FML were prepared in the same way. The size and shape of pebbles should be statistically the same as used here.

Fig. 3 serves the discussion of a possible size effect of the contact strength. Pebbles with a size of $0.44 \text{ mm} < D_m < 0.56 \text{ mm}$ are divided into five groups according to their sizes. The number of pebbles in each group is between 10 and 20. The contact strength here is defined as $F_c/D_m^2$, which is usually taken as the characteristic stress for the failure of brittle spheres [13]. The size effect describes the volume influence on the crush load. For instance, an increasing pebble volume can cause a decrease of the contact strength, if the flaw size distribution is independent of the volume and if failure is induced by a critical flaw. The contact strength is calculated for each pebble. The mean value and standard deviation are derived with these strengths for each group. The mean size of pebbles for each group is taken as the corresponding $D_m$ in Fig. 3. For both plates, it can be seen that the presence of a size effect, at least within this size range, is not clear. The pebble strength for WC plates is about 30 MPa while the pebble strength for AL plates is about 45 MPa. The difference between the strengths implies that pebble strength cannot be characterized by a stress constant that is equal to $F_c/D_m^2$. The strength can also be defined as the crush load divided by the contact area [14]. It is found that the strength using such a definition is approximately a constant for a plate material within the range of elastic deformation. However, the strength depends on the plate material, which is similar to the strengths shown in Fig. 3.

Fig. 4 shows the crush load distributions for all tested samples where the size difference is ignored. Clearly seen is the significant influence of the plates. Table 2 lists additional information on these tests. The crush load distribution obtained at FML is shown in Fig. 5. The crush load difference between AL and BK7 plates is mainly due to the experimental environment since AL and BK7 have similar stiffness. It should be noted that the environment modifies the surface state of pebbles,

![Fig. 2. Characterization of Li$_4$SiO$_4$ pebble geometry. Left: pebble size distribution; right: pebble shape distribution.](image)
e.g., presence of water at preexisting micro-cracks provides additional suction to prevent cracks from propagating, which results in a difference in crush load. On the other hand, it is assumed that the environment has no or little influence on the bulk elasticity properties like Young’s modulus and Poisson’s ratio. These two crush load distributions show that the strength of pebbles becomes much higher for pebbles crushed in air than in dry inert gas, which is in accordance with the findings by Knitter [15].

Finally, some typical forms of failure are shown in Fig. 6. It is found that most pebbles failed spontaneously. It is hard to identify where failure started. Besides, there are several distinct failure forms, which indicates the underlying complicated failure mechanism. For most pebbles, as soon as failure occurred, all or part of the fragments of the pebbles were ejected.

**Table 2**

<table>
<thead>
<tr>
<th>Plates</th>
<th>N</th>
<th>( F_c ) (N) (^a)</th>
<th>( (F_u,F_0,m) ) (^b)</th>
<th>Environment</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC</td>
<td>92</td>
<td>7.55 ± 1.55</td>
<td>(2.23, 88.88, 3.97)</td>
<td>air</td>
</tr>
<tr>
<td>AL</td>
<td>86</td>
<td>10.6 ± 3.02</td>
<td>(3.67, 7.87, 2.39)</td>
<td>air</td>
</tr>
<tr>
<td>BK7</td>
<td>200</td>
<td>5.88 ± 1.14</td>
<td>(3.03, 3.18, 2.50)</td>
<td>dry inert gas</td>
</tr>
</tbody>
</table>

\(^a\) Mean value plus standard deviation.

\(^b\) Fitting parameters according to Eq. (1).
from the apparatus making any post failure investigation impossible. Certain fragments sometimes stayed on the plates, see, for example, the first image in Fig. 6. On the other hand, some pebbles, after the first time failure when there was a jump of the contact force, could still sustain some load. In such cases, the pebbles broke into two or more parts, see the upper right and lower left images in Fig. 6. The failure possibly originates from the contact points. However, the lower right images seems to indicate that the failure of this pebble did not start from the lower contact point.

4. Probabilistic strength model

Various stress based strength models to explain the influence of plate material on the crush load have been explored [16]. For instance, it has been assumed in one model that multi-sized pebbles have a size-dependent strength in terms of a characteristic stress. In other models, a stress based strength distribution has been assumed for mono-sized pebbles. In the present study an energy based probabilistic strength model

\[ P_s = 1 - \exp \left( - \left( \frac{W_c}{W_{\text{Mat}}} \right)^m \right), \]

was found to be appropriate to characterize the influence of the plate material on crush load distributions. Here, \( W_c \) is the energy absorbed by a pebble, which is influenced by the plate material. \( W_{\text{Mat}} \) and \( m \) are two constants characterizing the pebble material. This strength model deals with a classical phenomenological approach, which has the particular advantage
of converting the information from the industrial-scale characterization processes for quality assurance, e.g., the crush tests in FML [12,7], to a quantitative statement on the pebble strength. The model (2) is validated in three steps:

1. Calculate \( W_c \) by Finite Element Method (FEM) for every crush load found in the experiments to obtain the corresponding distribution \( P_s(W_c) \), see the data points in Fig. 7. In contrast to the representation in Fig. 4, the probability data sets obtained for the two different types of plates more or less coincide when plotted over \( W_c \) as shown in Fig. 7. This indicates that the proposed energy based model according to Eq. (2) is appropriate to extract the pure strength properties of the pebbles from the crush tests.

2. Fit Eq. (2) to the obtained \( P_s(W_c) \) by adjusting the parameters \( W_{\text{Mat}} \) and \( m \) by the least square method. This fit is not represented in Fig. 7 in order not to overload it. Steps one and two are done two times: once for the data from experiments conducted with AL plates and once with the data from experiments conducted with WC plates.

3. For a validation, first introduce in Eq. (2) \( W_{\text{Mat}} \) and \( m \) from experiments with AL plates and compare it to \( P_s(W_c) \) from the experiments with the WC plates, see Fig. 7 left. Second, introduce in Eq. (2) \( W_{\text{Mat}} \) and \( m \) from experiments with WC plates and compare it to \( P_s(W_c) \) from the experiments with AL plates, see Fig. 7 right.

Details will be explained in the following. \( W_c \) is obtained from FEM simulations, where an elastic sphere representing the Li$_4$SiO$_4$ pebbles is compressed by two parallel plates uniaxially. The sphere has a diameter of 0.5 mm, which is the average size of the pebbles measured in our crush tests. Friction between the sphere and the plates is not considered. FEM calculations have shown that the contact friction coefficient ranging from 0 to 1 has little influence on \( W_c \) [16]. This means that friction has little influence on pebble failure. Such independence of friction/surface roughness has also been reported in crush tests with sapphire spheres in Table 5 of [17]. A perfect plasticity model with the von Mises yield criterion for the plates is used in the FEM simulations. For every crush load obtained in experiments, the \( W_c \) is calculated by FEM using the corresponding material parameters listed in Table 1. Note that information on the dependence of Young’s modulus and Poisson’s ratio of pebbles on environment can hardly be found in literature. However, we expect that the testing environment has little influence on the bulk elasticity properties of the pebbles listed in Table 1, although it indeed will have an influence on the crush load distribution which should be due to the surface state modified by the environment, as mentioned before. The failure distribution in terms of the crush load (Fig. 4) can be transformed into a distribution in terms of \( W_c \). Both distributions for the experimental results are shown in Fig. 7. They will be used to validate the proposed strength model accounting for the influence of the plate material.

Predictions and corresponding experimental results are shown in Fig. 7. Both predictions agree well with the corresponding experimental results at the same time which validates the proposed strength model. Therefore, the plate influence can be explained by the model and the model is then validated. Thus, the model can be used to predict the pebble–pebble contact strength from experimental results for plates of any material. The crush load from our tests in air cannot represent the pebble strength in fusion relevant conditions because of the difference in the prevailing environment. In the next section we will use the present model to derive the pebble–pebble contact strength under fusion relevant conditions.

5. Pebble–pebble contact strength in a fusion like environment

The pebble–pebble contact strength will be derived from the crush load data from FML (Fig. 5), in which the fusion relevant environment with dry inert gas is taken into account. Fig. 8 shows the critical failure energy distribution, i.e., the distribution of the energy absorbed by the pebble before failure. Since plastic deformation is negligible in experiments with plates of BK7 glass, the energy \( W_{\text{BK7}} \) is calculated from Hertz theory where spherical pebbles having an identical size of
0.5 mm are used. The fitting function of the energy distribution with the parameters $W_{\text{Mat}} = 8.2 \times 10^{-6} \text{J}$ and $m = 3.2$ is then taken as parameters describing the pebble–pebble contact strength in a fusion like dry inert gas environment.

For an assessment of the onset and the propagation of pebble failure in a pebble bed, each pebble in the pebble bed will be assigned to a critical energy randomly according to this distribution. When a pebble absorbs more energy than its critical energy it will be assumed to have failed. Note that the critical energy distribution in Fig. 8 corresponds to the critical strength distribution of pebbles from the batch of OSi 07/1. It might not be valid for pebbles from other batches for which a new fitting of the parameters $W_{\text{Mat}}$ and $m$ will be necessary.

6. Discussion

This work presents a quantitative energy based prediction for pebble failure in a fusion like environment for the first time. There are several points about the proposed strength model:

1. The number and magnitude of contact forces applied on a pebble can be considered to contribute to the failure of pebbles. For elastic contact between spherical pebbles (same material), $W_c$ of any pebble can be calculated from the Hertz contact theory given by

$$W_c = \sum_{i=1}^{N_c} \frac{1}{5} \left( \frac{9}{16R_i^3} \right)^{1/3} \frac{1}{E^{1/3}F_i^{1/3}},$$

where $N_c$ is the number of contacts, $F_i$ is the $i$th contact force, $E^* = E/(2(1 - v^2))$ is the equivalent Young’s modulus and $R_i$ is the relative radius of curvature of the $i$th contact. For mono-sized pebbles $R^* = R/2$. This is an important feature since most pebbles in pebble beds are in contact with more than two neighboring pebbles and the contact forces on one pebble are usually not equal. It is essential to consider the contribution of all contact forces rather than the maximum contact force [18]. Care should be taken that taking $W_c$ in the strength model as the sum of the strain energies of all contacts of a pebble is a hypothesis until justification by experiments is possible.

2. The critical energy failure model, i.e., Eq. (2), is similar to the one in [19–22] where the Weibull modulus is limited to 1 and the minimum critical energy required for failure is a fitting parameter. In the paper [22], the failure model based on critical energy has been validated for some comparable crush test data, i.e., impact, falling weight and slow compression comminution (crush tests) for particles which are made of different materials. Thus, it is also expected that Eq. (2) used in this work could be applicable to comparable systems like other kinds of pebbles having a similar size range. However, one has to be careful to extend the application of Eq. (2) because the failure of brittle spheres made of other materials might be described by other criteria, such as the maximum tensile stress inside of the sphere [23] or on its surface, or by the maximum shear stress, or by other kinds of critical stresses. In some cases, failure is only decided by the maximum contact force [24].

3. In FEM simulations [16] it was found that the absorbed energy $W_c$ of a spherical pebble is insensitive to the friction coefficient between pebble and plates, leading to the conclusion that the present strength model is insensitive to the friction coefficient, as well. In other words, pebble strength is independent of the friction coefficient according to the prediction using the present energy-based model. If for other kinds of pebbles, such as ellipsoidal Li$_2$TiO$_3$, it is found in crush tests that the pebble strength depends on the friction coefficient, the applicability of the present strength model has to be reconsidered.
4. The current crush experiments have been performed using a selected narrow size range of pebbles. To import the pebble–pebble contact strength into DEM of pebbles with various sizes, the current strength model can be modified by using the critical energy density \( \phi_c(r) \sim W_c(r)/r^3 \) and \( \phi_{\text{MAT}}(r) \sim W_{\text{MAT}}(r)/r^3 \), rather than the absolute energy \( W_c \) and \( W_{\text{MAT}} \), for certain size group of pebbles with an approximate radius of \( r \). However, in general the type of failure criterion may depend on pebble size. Therefore, for the complete range of considered pebble sizes the applicability of the energy criterion has to be verified. In the positive case a proper extrapolation method for \( \phi_c(r) \) and \( \phi_{\text{MAT}}(r) \) needs to be developed at different size groups. If the verification is negative for certain pebble size ranges, identifying an appropriate failure criterion for such a size range can follow the procedure shown in [16].

5. The influence of temperature on the pebble strength has not been considered yet in this model. In view of the engineering application of the strength model in the HCPB blanket, the following consideration appears to be essential: Higher temperature will usually lead to a lower Young’s modulus of the bulk material. For the same configuration of pebbles in a pebble bed, namely the distribution of pebble centers and their radii, the contact force between pebbles will decrease along with decreasing Young’s modulus according to the Hertz contact theory. Meanwhile, the strain energy in each pebble will decrease as a result. On the other hand, the critical strain energy is assumed to be a constant independent of Young’s modulus. Thus, at the same overall strain level, there will be less chance for pebbles to be crushed at a higher temperature. In other words, the effect of temperature on Young’s modulus will decrease the failure probability of pebbles compared to the one predicted in this work under the same load level. For long term operation of the fusion reactor, creeping may contribute to further failure of pebbles slowly and steadily. However, the critical energy model adopted in this work does not contain any time-dependence. Thus, it might not be relevant for such a failure mode by creep deformation. Note that the critical strain energy being independent of temperature should be validated by further experiments if the influence of temperature has to be considered.

6. The strength model adopted in this work is a phenomenological model. The failure mechanism behind it could be related to some kind of stresses. The analytical stress solution [25] shows that stresses at any point inside the sphere are proportional to the contact pressure. Thus, there may be an implicit relation among contact force, contact area and \( W_c \).

The pebble–pebble contact strength provides accurate input for DEM modeling to generate information about pebble failure initiation and propagation inside pebble beds. Through DEM simulations, we can link the relatively simple crush tests to gain insight of randomly packed beds under thermomechanical loads.

7. Conclusion

Crush tests for single \( \text{Li}_4\text{SiO}_4 \) pebbles are performed using plates of two different materials, and the corresponding crush load distributions are obtained. The average crush load for soft plates (AL) is much higher than that for hard plates (WC). A strength model is proposed to explain the influence of the plate material. The model is validated by experimental results. The pebble–pebble contact strength for conditioned pebbles from a certain batch of \( \text{Li}_4\text{SiO}_4 \) pebbles has been derived based on the crush load distribution at fusion relevant environment. The obtained strength distribution can be used to investigate the influence of pebble failure on the macroscopic thermomechanical response of pebble beds by means of DEM.

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