Research proposal to the International Fine Particle Research Institute

Measuring Fracture Stress in Irregular Agglomerates

Can one stress criterion predict failure across loading orientations?

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Research question and industrial need

An irregular agglomerate can fail at different forces when it is turned between the loading platens. Dividing the force by a projected area only partly addresses the problem: it does not account for where and over what area the particle contacts the platens or how the load generates tensile and shear stresses inside it. Nor does it separate the loading geometry from the flaws that make identical particles break at different stresses. IFPRI members need a stress criterion that accounts for these effects, with a critical stress that can be collected rapidly enough to characterise a population, not just a few particles.

We will test whether a coarse-grained stress criterion gives a reproducible threshold at fracture across loading orientations for particles of the same material and architecture. The decisive experiment is to crush nominally identical 3D-printed replicas in different orientations. If breaking force changes but the inferred critical stress does not, within a practical tolerance, the criterion has passed a test that peak load cannot. We will then test how far it transfers across sizes, shapes and to real agglomerates. The target size range is 250 μm to 5 mm.

The proposed industrial test combines optical imaging with a calibrated compression stage. A spring-lattice model, updated from the measurements as loading proceeds, provides estimates of internal stresses. In the project we will also use our DynaTom system, in which the X-ray source and detector turn around a particle that stays still between the platens, to record three-dimensional images of its interior while it is loaded. These images show where deformation concentrates and where fracture starts, which gives more data for improving the model and an independent test of what it predicts. The use of DynaTom will be limited, since routine testing that may eventuate for industry will not require X-ray tomography. The critical unknown is whether the force, the external shape and the visible contacts of an irregular particle, which are what a routine test can measure, are enough for the model to fix its internal stress.

Experimental and modelling programme

Controlled crushing tests

We will synchronise force and platen displacement with Hirox images of the particle boundary, visible contact locations and fracture. We will calibrate machine compliance, image scale, timing and platen alignment. A second simultaneous view will be tested where a single view leaves important out-of-plane motion or geometry unresolved. We will distinguish first detectable fracture from peak force and record whether it starts at a contact. If onset falls between observations, we will report the corresponding load interval.

The first tests will use printed spheres, followed by irregular 3D-printed elastobrittle replicas with controlled external geometry and, where fabrication permits, designed pores or weak regions. From each print batch we will also break simple bars in bending, printed along and across the layers, which measures the stiffness and the tensile strength of the print material in both directions and gives a known value against which the critical stress found from crushing can be checked. Each replica will be crushed once. We will randomise the assignment of replicas to loading orientations within print batches and check dimensional scatter and the bias introduced by the printed layers, since stiffness and strength can differ along and across them. Pilot results will determine the replication needed to separate orientation effects from manufacturing and test variability. If the intended internal features cannot be reproduced at the smallest sizes, controlled tests will use larger replicas and the applicable size range will be established separately on real particles.

Indicative test matrix. We will begin with the sphere and two irregular designs made from the same print material. An initial series of three loading orientations and six replicas per design–orientation combination would give 54 destructive tests, balanced across two print batches. We will record print direction separately from loading direction so that print anisotropy is not mistaken for a shape effect. A second series will introduce a controlled pore or weak region, provided the initial tests show that fabrication scatter is small enough to distinguish its effect. As a step between the prints and real agglomerates, we will also crush model agglomerates made of beads bonded together, in which the position of every bead and the strength of a single bond are known. Each bond is then one spring of the lattice, and we can check that the first bonds to break are those where the model gives the highest stress. The counts are planning assumptions: pilot variance and the agreed tolerance will determine whether to add replicas, revise the designs or narrow the comparison.

From lattice forces to fracture stress

The digital shadow is a spring lattice that fills the measured external shape of the particle and follows it through the test. For real particles the external shape is reconstructed optically from several views before loading. The lattice is fitted as the loading proceeds, through a feedback loop that corrects the model from the measurements. At each step the lattice is compressed by the measured platen displacement and predicts the force on the platens and the motion of the particle boundary. The difference between these predictions and the measurements is fed back to correct

the state of the lattice and a small set of effective parameters, such as the stiffness of the springs and the friction at the contacts, so that the model keeps reproducing the force–displacement curve and the boundary motions seen by the microscope. Where a second microscope or DynaTom is used, its measurements enter the same loop. Only data before fracture are used, and we will test how much the result changes for plausible alternatives of contact friction, stiffness and internal structure. A close fit to the load curve alone will not be taken as evidence that the internal field is correct. The fit starts from the external shape, the contacts and the elastic properties of the particle, and its internal structure is added only as a second step.

Once the lattice reproduces the measurements, the forces in its springs give the stress inside the particle. We will coarse-grain lattice forces and branch vectors over a specified physical neighbourhood, whose size depends on the internal structure of the particle, to estimate Cauchy stress, and use displacement gradients to estimate strain at a compatible scale. Force balance and boundary tractions will provide checks on the stress calculation, and on spheres the result must reproduce the known elastic solution. The stress measure of the criterion is evaluated on this field at the first detectable fracture, which gives the critical stress of that particle. We will report particle-scale stress–strain curves separately from the local stress measure used to assess fracture. For local estimates we will propagate measurement and model uncertainty and test sensitivity to lattice spacing, averaging length and proximity to the platens.

What DynaTom will test

For selected replicas and, where contrast and resolution permit, representative real agglomerates, DynaTom will image the interior of the particle repeatedly during compression, and each image will be matched to the measured load. We will compare predictions made without the validation scans against observable internal displacements, changes in resolvable pores and contacts, and the location of the first detectable crack. Where texture supports it, volume correlation will quantify internal displacement and strain. DynaTom does not measure stress directly: it tests the deformation and failure mechanism on which the stress inference depends.

If continuous scans cannot resolve a fast fracture event, we will use interrupted loading or before-and-after volumes to bracket damage, and check whether load holds alter the response. The comparison will establish when one optical view is sufficient, when additional views improve the inference, and when boundary measurements cannot support a defensible local stress estimate.

Independent validation on selected specimens. We will choose cases to distinguish competing mechanisms, for example orientations predicted to produce interior tensile localisation versus contact-adjacent damage. Before inspecting the internal images, we will record the predicted localisation region and onset load or interval using only the measurements available to the optical workflow. Scans used to refine the model will be kept separate from those used to check it. Where contact geometry or pores cannot be resolved, we will report which comparisons remain possible rather than treating an unobserved feature as absent.

How we will judge the criterion

The first candidate is maximum principal tensile stress at a specified averaging scale. A larger region under high tension is more likely to contain a weak spot, so we will also test whether the amount of material under high tension matters in addition to the peak value. We will compare it with stress-invariant combinations for mixed loading and use tensile elastic energy density as a competing descriptor. We will select the spatial statistic and averaging scale on development data, then fix them before evaluating held-out orientations, shapes and sizes. In a held-out test the criterion must predict the onset load without using the observed crack location to choose where to evaluate the field. When fracture starts at a contact, the test does not give the critical stress of the body of the particle. It shows only that the body withstood the stress it carried at that load, so it is used as a lower bound and not discarded.

We will compare performance with peak force, force divided by projected area and force divided by the squared platen separation, the nominal strength in common use. We will estimate the orientation effect and its uncertainty within replica families, account for print-batch variability, and assess whether the residual effect lies within a practical tolerance agreed with IFPRI members. Reduced scatter alone will not establish orientation independence. The critical stress must also agree with the strength measured on bars of the same batch, allowing for the different volumes under tension, which tests accuracy as well as consistency. If a common scalar threshold fails, we will test whether material-specific, directional or probabilistic criteria improve prediction and report the conditions under which a simple strength number is not justified.

Decision rule. Before the held-out tests, we will agree with IFPRI members on a practically meaningful difference in critical stress between orientations, taken at a fixed probability of fracture. The uncertainty interval for the estimated orientation contrast must lie within that tolerance to support an orientation-independent threshold. If the interval straddles the tolerance, the result is inconclusive; if it lies beyond the tolerance, the common threshold is not adequate for that family. We will report onset-load prediction error alongside this test so that a criterion cannot pass merely by giving similar inferred stresses after fracture is already known.

Three-year programme

Year 1: Establish the measurement and benchmarks. Calibrate the synchronised compression and microscopy platform, characterise the repeatability of printed specimens and the stiffness and strength of the print material on bars, and verify the lattice-to-stress calculation against the elastic solution for spheres. Complete an initial orientation series and use it to set replication and measurement requirements.

Year 2: Test the criterion and validate the inference. Extend the replica experiments across orientations, shapes and sizes and to bonded beads, rank candidate criteria on held-out tests, and use targeted DynaTom measurements to test predicted localisation and fracture onset. Determine the minimum optical measurements needed and which stress estimates remain reliable when internal structure is uncertain.

Year 3: Demonstrate industrial transfer. Apply the protocol to selected real elastobrittle

agglomerates. Each real particle has its own shape and breaks once, so the criterion is tested there on the population, where the critical stress must not vary with shape or with the number of contacts. Estimate material-specific fracture-stress distributions and measure repeatability, usable-test rate and operator time per particle. Compare a continuum formulation with the lattice only if it improves predictive performance or transfer across particle sizes and shapes. Deliver the test protocol, analysis workflow, uncertainty and quality-control rules, benchmark data and reference specimen designs.

Existing capabilities and IFPRI participation

The programme builds on the Einav group's work in grain crushing, breakage mechanics, coarse graining and imaging of granular materials. Existing Hirox and DynaTom capabilities support the optical development and the limited internal validation programme. The proposed industrial workflow uses a calibrated load frame, force and displacement sensing, and the simplest optical configuration supported by the validation results.

IFPRI members can help select materials and conditioning procedures, provide current breakage tests or process-survival data for comparison, and define useful tolerances for accuracy, throughput and equipment cost. A member laboratory could trial the reference particles and protocol to assess reproducibility outside the originating lab. We will provide annual reports, present progress at IFPRI's annual general meeting and deliver a capstone report setting out the criterion, where it works, and where it should not be used.