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# New artificial granular materials for analogue laboratory experiments: aluminium and siliceous microspheres

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**Abstract**

Laboratory tests have been carried out to determine the physical properties of high sphericity and low density, fine grained dry granular materials. In particular, we tested hollow aluminium microspheres and hollow siliceous microspheres. Shear tests have been carried out with a Casagrande apparatus for different values of normal stress (from 12.27 to 491.8 kPa) in order to determine the frictional properties of the microspheres. Diagrams of the shear stress against displacement have been used to describe the strength of the investigated materials at different confining pressures. The shear tests show that hollow aluminium and siliceous microspheres successfully replicate the mechanical behaviour of weaker layers within natural sedimentary successions undergoing deformation. The use of these artificial granular materials in specifically designed experiments indicated that they can be successfully used for constructing more "natural" multilayers in the laboratory.


*Keywords:* Analogue modelling; Artificial granular material; Coefficient of internal friction; Cohesion; Mechanical stratigraphy

## 1. Introduction

The capability of dry granular material, basically sand, has long been recognised in analogue modelling to simulate the mechanical behaviour of the upper crust (e.g. Hubbert, 1951; Ramberg, 1981; Davy and Cobbold, 1988, 1991; Krantz, 1991; Weijermars et al., 1993; Schellart, 2000; among others). The use of pure sand in normal gravity experimental conditions approximates the upper crust as homogeneous brittle material obeying the Mohr–Coulomb failure criterion (Hubbert, 1937; Ramberg, 1981). This approximation is valid for the first-order analysis of tectonic processes (e.g. Davis et al., 1983; Malavieille, 1984; McClay and Ellis, 1987; Marshak and Wilkerson, 1992; Wang and Davis, 1996) but fails to account for mechanical layering within sedimentary successions. Very thin inter-layers of vermiculite (e.g. McClay, 1990; Storti and McClay, 1995) or layers of silicone putty (e.g. Letouzey et al., 1995; Bonini et al., 2000) have been used to impart a mechanical anisotropy to the experimental sandpacks. Despite the demonstrated effectiveness of these solutions, the precise rheological scaling of the mica interlayers and of the sand–silicone interfaces is somewhat problematic when great detail is required in the mechanical stratigraphy specifically when analysing individual structures. More complex laboratory techniques using the centrifuge have been designed to simulate deformation of natural rock multilayers (e.g. Ramberg, 1981; Liu and Dixon, 1995). The use of microlaminates for introducing mechanical anisotropies in centrifuge models has been quantified by Koyi (1996). However, the great complexity of the centrifuge technique and its strong sensitivity to the experimental procedures and analogue materials still favour the use of sandbox modelling at natural gravity conditions (e.g. Koyi and Mancktelow, 2001).

The close experimental simulation in natural gravity conditions of sedimentary successions characterised by alternating weaker and stronger layers needs the use of granular materials other than loose sand, like glass microspheres, whose roundness strongly contributes to reduced cohesion and internal friction (e.g. Schellart, 2000). To further broaden the availability of artificial granular materials suitable for analogue modelling at natural gravity conditions, we laboratory tested the mechanical behaviour of hollow aluminium and siliceous microspheres. Our

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results encourage the use of these new analogue materials to construct strongly anisotropic, scaled multilayers in the laboratory.

## 2. Material and methods

Hollow aluminium hydroxid microspheres (hereafter MICROBALLS) and hollow siliceous microspheres (hereafter SI-CEL) are produced by Prochima (Italy) with different particle sizes and colours. Tested materials have grain size of, respectively, 25 μm (SI-CEL) and 40 μm for (MICROBALLS) (Fig. 1). Physical properties are listed in Table 1. Roundness has been determined according to Powers (1953). Sphericity has been assessed by the ratio between the minor and the major axes of the particles. Particle size has been determined using a series of sieves with different mesh apertures. Density has been determined by measuring the mass of known volumes of material with analitical balance at the nearest 0.001 g. Measure errors are ±0.65% for MICROBALLS and ±1.41% for SI-CEL, respectively (see Table 1).

The use of hollow materials implies the possibility of the load-driven crushing of microspheres, which significantly alters their frictional and physical properties. Such microsphere crushing is expected to significantly increase their density. To test this possibility, we determined the mass of each experimental material for 10 different sediment column heights. Plotting the masses against the corresponding sediment column heights gave almost perfectly linear results (Fig. 2), ruling out the possibility of microsphere crushing in this load range, which is typical of analogue experiments.

Shear tests were carried out in a Casagrande direct shear box (Fig. 3), which is a reference apparatus for measuring shear properties in soil mechanics (e.g. Lambe and Whitman, 1979; A.S.T.M. D 3080-90 direct shear test). Granular materials were held in the Casagrande box, which is split across its middle. Normal stresses, ranging from 12.27 to 491.8 kPa, were applied by dead weights, and then a shear stress was applied to cause relative displacement between the two parts of the box. The magnitude of the shear stress was recorded as a function of the shear displacement. Shear stress measurements were triplicated for each normal stress and standard errors were calculated (Table 2).

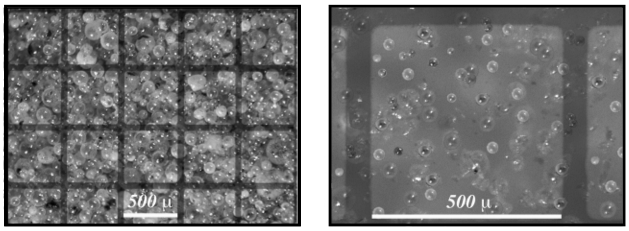


Fig. 1. Microphotograph of the artificial granular materials tested in this work.

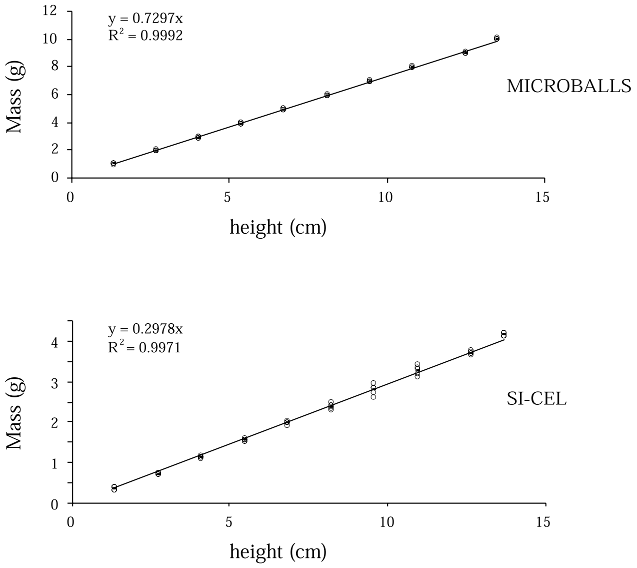


Fig. 2. Plot of data displaying the relationship between mass increase and height increase of the sediment column (h) for the investigated materials. Straight lines are best-fit lines for the data.

## 3. Shear test data

Plotting the shear stresses against displacement for different values of normal stresses highlighted the lower shear strength of SI-CEL microspheres compared with MICROBALLS (Fig. 4a). Shear stresses plotted as a function of normal stresses are shown in Fig. 4b. Data points in Fig. 4b align well along best-fit lines ($R^2 > 0.995$), whose direction coefficients give the coefficients of internal friction ($\mu$) of the tested materials. In particular, $\mu$ is 0.46 ($\phi = 24.7° \pm 0.009$) for MICROBALLS and it is 0.44 ($\phi = 23.9° \pm 0.008$) for SI-CEL. The interception of the best-fit lines with the shear stress axis gives the extrapolated cohesion ($C'$) for both types of the granular materials. Such intersections are very close to the origin, indicating negligible values of extrapolated cohesion. In particular, the estimated cohesion is 6 Pa for MICROBALLS and 1.5 Pa for SI-CEL.

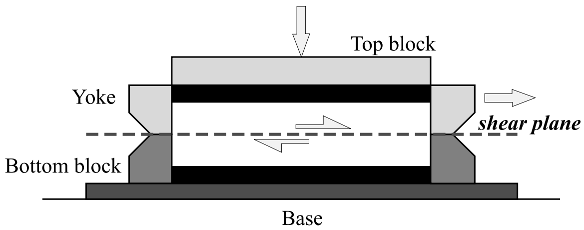


Fig. 3. Cross-sectional sketch of the Casagrande direct shear box (after Lambe and Whitman, 1979). Top block and yoke are free to move up or down to allow for volume changes, while top and bottom blocks are fitted with teeth for gripping the sample.

Table 1
Physical properties of hollow microspheres used in the experiments. $D$ is density; g.s. is the grain size, $\mu$ is the internal friction coefficient, $\phi$ is the internal friction angle, and $C'$ is the extrapolated cohesion

| Material | $D$ (g/cm$^3$) | g.s. (μm) | $\mu$ | $\phi$ | $C'$ (Pa) | Roundness | Sphericity |
|---|---|---|---|---|---|---|---|
| MICROBALLS | 0.39 ± 0.65% | 40 | 0.46 | 24.7 ± 0.009 | 6 | Very well rounded | Very high |
| SI-CEL | 0.15 ± 1.41% | 25 | 0.44 | 23.9 ± 0.008 | 1.5 | Very well rounded | Very high |

The validity of the shear test data was checked according to a number of statistical tests described in Sokal and James Rohlf (1987). In particular data were tested for conformity to assumptions of variance homogeneity (Cochran's $C$-test) and log-transformed (for count) or aresine-trasformed (for percent data). Post-hoc comparisons of means were performed by SNK tests. Analyses of regression have been performed and error of intercept has been estimated. The variability range of the extrapolated cohesion values has been calculated by estimating the interception probability ($\alpha$) at 99% along the shear stress axis as (Sokal and James Rohlf, 1987)

$$C' - T\cdot\mathrm{Es} \le \alpha \le C' + T\cdot\mathrm{Es} \quad (1)$$

with $T$ being the corresponding $T$-student test value and Es the standard deviation error (Table 2). The obtained $\alpha$ values indicate that MICROBALLS have $-1.6$ Pa $\le C' \le 9.8$ Pa, while the variability for SI-CEL microspheres is virtually zero.

## 4. Discussion

It has been recently demonstrated by Schellart (2000) that failure envelopes of granular materials have a non-linear behaviour for very small normal stresses (<400 Pa) and a linear behaviour for higher normal stresses. This implies that linear best fitting (e.g. Krantz, 1991; Cobbold and Castro, 1999) overestimates the values of cohesion at very low normal stresses. Such a limitation applies to our laboratory results since we used normal stresses higher than Krantz (1991) or Cobbold and Castro (1999) and, consequently, our linear extrapolations of the Mohr–Coulomb behaviour of the studied microspheres are less constrained at very low normal stresses. Despite this, the statistical validation of our result supports the estimations of negligible cohesion for the tested artificial microspheres. The very low coefficient of internal friction and cohesion of dry granular materials are mainly dependent on sphericity and rounding of the individual particles and less on particle size (e.g. Schellart, 2000). The SI-CEL and MICROBALLS microspheres have a very high to perfect sphericity and are very well rounded, and this supports the estimated negligible values of cohesion.

Table 2
Statistical analysis of shear test data. Coefficients $q$ and $m$ are, respectively the intercept and the direction coefficient of the best-fit line $Y = mX + q$. In particular, $q$ corresponds to $C'$

| Test | SI-CEL | MICROBALLS |
|---|---|---|
| $T$-student | 0.000 | 0.002 |
| Coefficient ($q$) | 0.0015 | 0.006 |
| STD error ($q$) | 2.103 | 2.420 |
| Coefficient ($m$) | 0.4448 | 0.461 |
| STD error ($m$) | 0.008 | 0.009 |
| $F$-ratio (1–39) | 3215.205 | 2608.043 |
| $P$ | >0.0001 | >0.0001 |

The efficacy of the tested microspheres as décollement material in sandbox analogue modelling has been tested in 53 experiments with different configurations of the undeformed sand-microsphere multilayers. Experiment D01-08 is described as an example of highly variable mechanical stratigraphy. The undeformed multilayer model D01-08 was constructed by sieving loose granular materials above a basal sheet of drafting film ($\mu = 0.47$; c.f. McClay, 1990). The sand–microsphere multilayer (Fig. 5) consisted of a 'thick' basal layer of SI-CEL overlain by a thick layer of MICROBALLS, in turn overlain by two thick layers of loose sand ($\phi = 37.5°$; Acocella et al., 2000) separated by a thin layer of MICROBALLS. A thick layer of MICROBALLS was sieved on top of the upper thick loose sand layer. The upper half of the multilayer consisted of alternating thin layers of loose sand and thicker layers of MICROBALLS. The top of the multilayer was made up by a thick layer of loose sand. The total thickness of the undeformed sand–microsphere multilayer was 15.5 mm. The scale factor for lengths is $5 \times 10^{-6}$ and therefore 1 cm in the box represents about 2 km in nature. Deformation occurred in a typical sandbox (e.g. Storti and McClay, 1995) by pulling the basal drafting film below the rigid end wall of the rig, which acted as the undeformable backstop against which the multilayer collided. It is important to note that different deformation styles characterise different mechanical units (Fig. 5). In the early and intermediate stages of contraction ($S < 10\%$) faulting occurred only in the thick sand layers of the lower half multilayer. The overlying thinly layered mechanical unit underwent disharmonic folding, whereas the basal SI-CEL layer homogeneously thickened. In the late stages of the model growth ($S > 10\%$), increased contraction caused the upward propagation of thrusting and the occurrence of discrete faulting in the basal SI-CEL layer. The presence of the thick MICROBALLS basal layer prevented faults in the SI-CEL layer from linking with those in the overlying multilayer. Despite the evidence that discrete thrust sheets formed above the basal MICROBALLS thick layer, deformation within the thrust sheets was strongly disharmonic

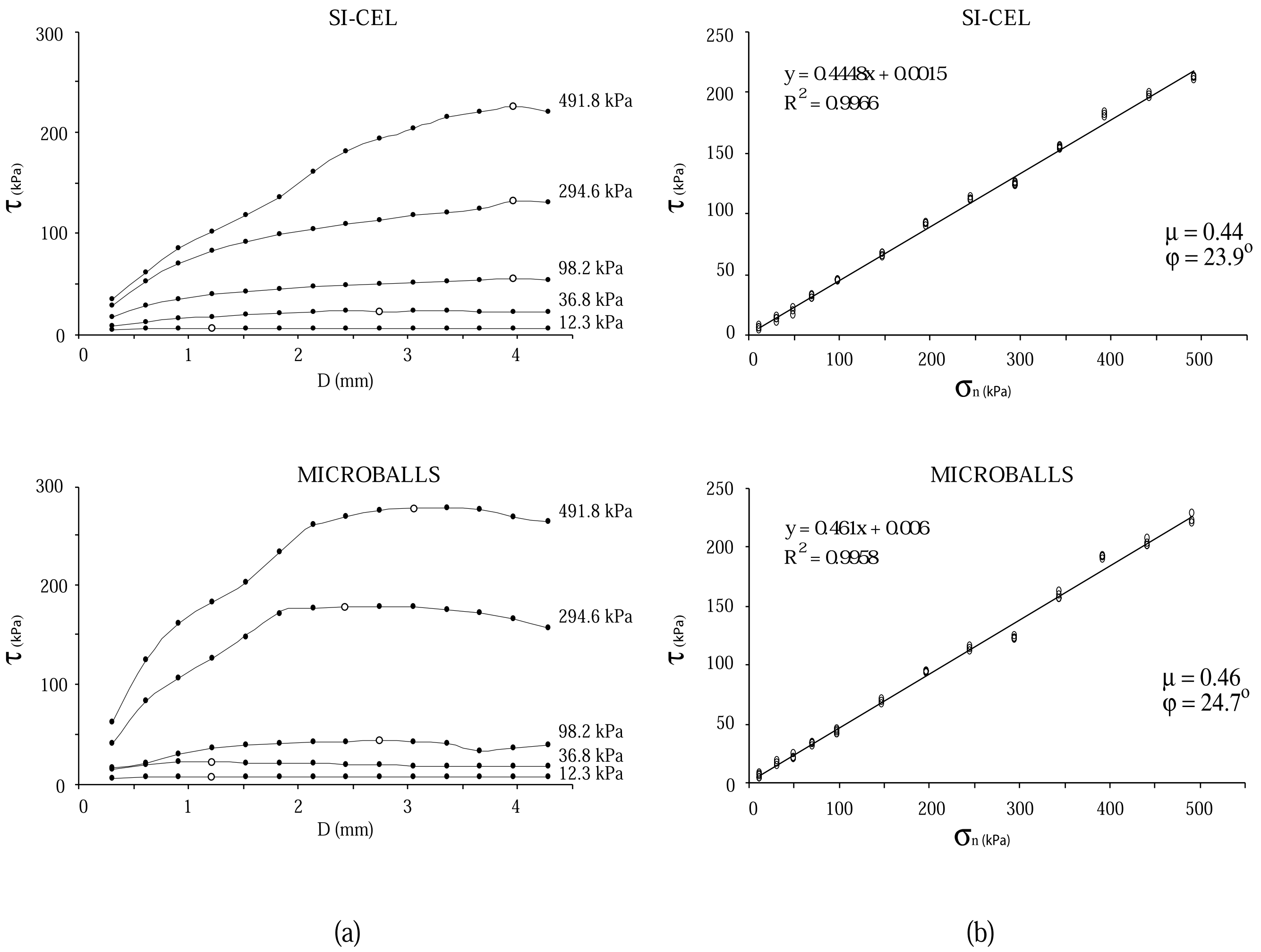


Fig. 4. (a) Shear stress vs. displacement; (b) regression of shear test by Casagrande apparatus for MICROBALLS and SI-CEL (normal stress $\sigma_n$: from 12.275 to 491.8 kPa).

due to the decoupling effect exerted by the hollow microsphere layers interbedded within sand.

Comparison between model D01-08 and a silica sand model produced with the same experimental apparatus (D01-12) highlights first-order differences related to the presence of hollow microsphere layers (Fig. 6). In particular, the internal architecture in the silica sand model consists of few thrust sheets involving the whole sand-pack, and this contrasts with the highly disharmonic geometry of model D01-08.

Experimental results demonstrate that the lower shear strength of MICROBALLS and SI-CEL microspheres makes these materials suitable for simulating weaker layers within the brittle crust, obeying the Mohr–Coulomb failure criteria. These hollow microspheres have the lowest frictional parameters among other granular materials used in sandbox analogue experiments (Table 3) and can be used for simulating low-strength horizons within sedimentary successions and décollement materials other than evaporites within contractional or extensional tectonic systems. A first-order advantage of using hollow microspheres instead of silicone putty as décollement material for simulating frictional décollement layers within natural sedimentary successions is that the former obey the Mohr–Coulomb failure criterion and this prevents significant scaling problems characterising the sand–silicone interface. In fact, the tendency of sand particles (or glass microspheres) to sink into the viscous and less dense silicone putty unavoidably creates a mixed layer at the sand–silicone interface with a complex, non-Newtonian rheology.

## 5. Conclusions

Laboratory tests on fine-grained aluminium and siliceous hollow microspheres indicate that they are suitable analogue materials for simulating the mechanical behaviour of décollement layers within experimental 'sandpacks'. These artificial granular materials have high sphericity, low density, and negligible cohesion, and their coefficient of internal friction and angle of internal friction are lower than those of glass microspheres and loose sand. The extremely

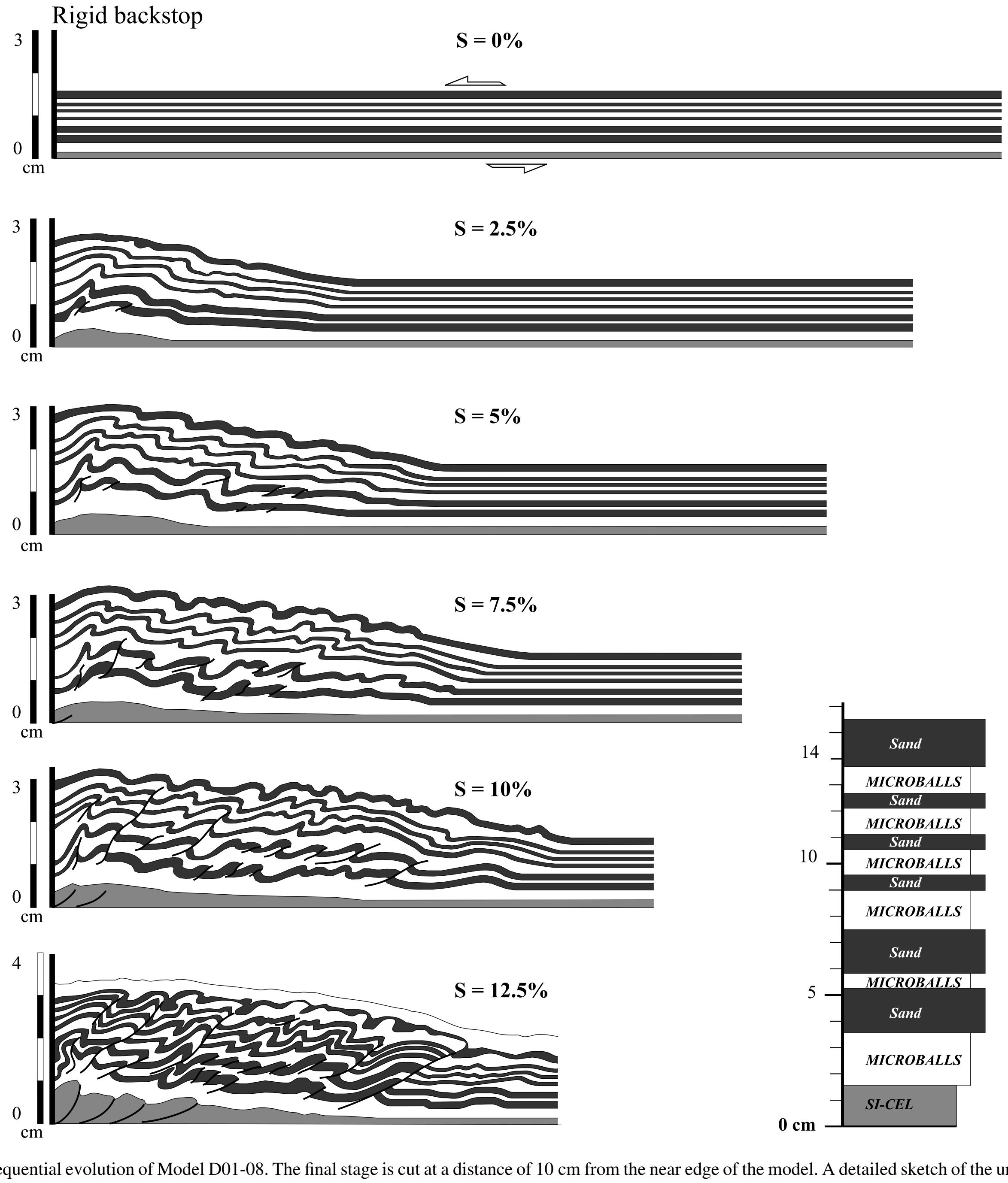


Fig. 5. Sequential evolution of Model D01-08. The final stage is cut at a distance of 10 cm from the near edge of the model. A detailed sketch of the undeformed mechanical stratigraphy is shown in the lower-right corner.

fine particle size of the tested materials allows the construction of very thin-layered multilayers and thus, coupled with the narrow width of fault zones, provides more structural detail and better scaling than coarse-grained materials. Sieving layers of aluminium and/or siliceous microspheres alternated with glass microspheres and/or sand layers allows the construction of undeformed experimental multilayers, which better simulate the variable mechanical stratigraphy of natural sedimentary successions. Results of sandbox experiments using highly anisotropic undeformed multilayers support the primary role of the mechanical stratigraphy for controlling the structural style of deformations within thrust wedges.

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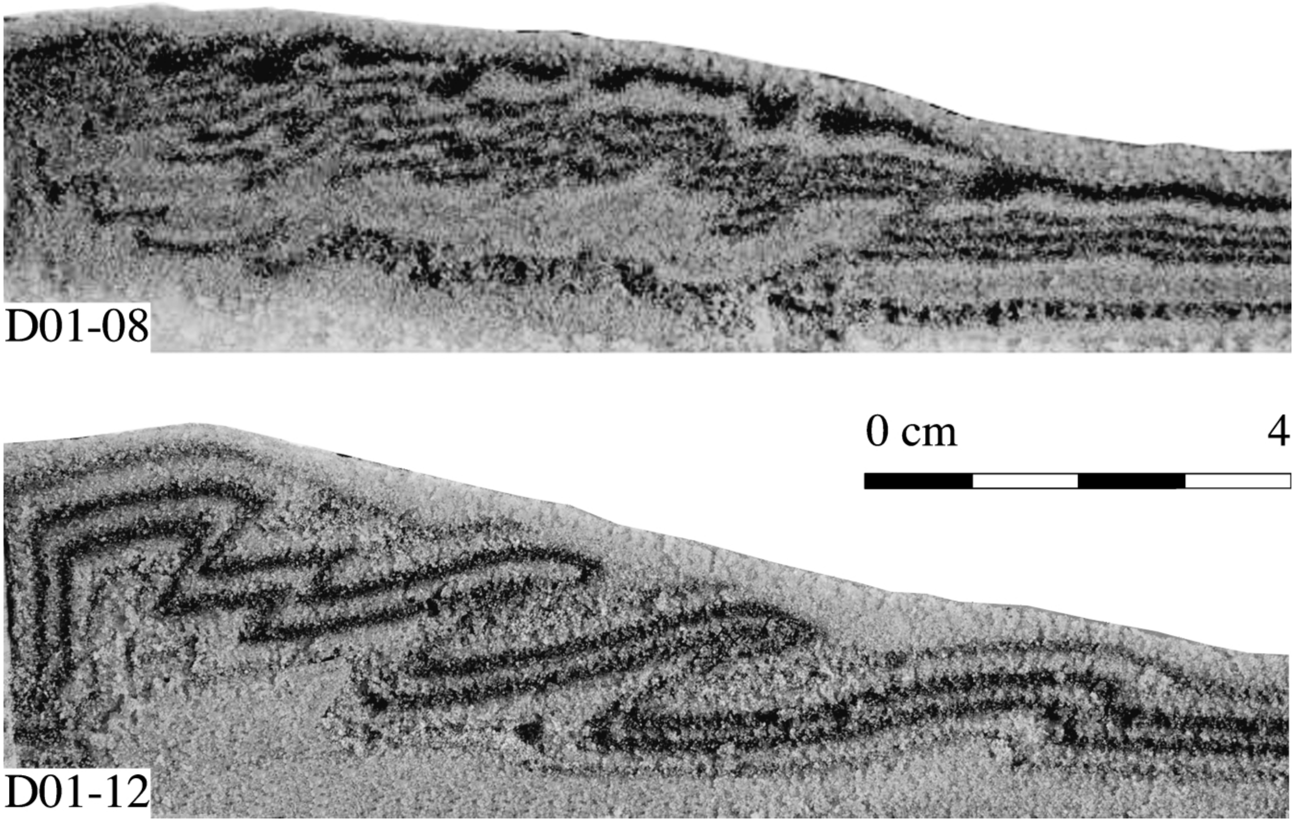


Fig. 6. Comparison between the internal architectures of model D01-08 (sand–microsphere multialyer), and model D01-12 (sand multilayer) at 12.5% of contraction.

Table 3
Physical properties ($C'$, $\mu$, $\phi$ and g.s.) of different granular materials tested by many authors. (1) Schellart (2000); (2) McClay (1990); (3) Grotenhuis et al. (2002); (4) Bonini et al. (2000); (5) Turrini et al. (2001); (6) Gutscher et al. (1998); (7) Acocella et al. (2000); (8) Cobbold et al. (2001); (9) Krantz (1991); (10) Faccenna et al. (1995); (11) Mart and Dautevil (2000)

| Material | g.s. (μm) | $\mu$ | $\phi$ (°) | C′ (Pa) |
|---|---|---|---|---|
| SI-CEL | 25 | 0.44 | 23.9 | 1.5 |
| MICROBALLS | 40 | 0.46 | 24.7 | 6 |
| Glass microspheres GMII (1) | 90–180 | 0.65 | 33.0 | 160 |
| Sand SII (1) | 90–180 | 0.88 | 41.3 | 230 |
| Quartz sand (5) | 100–130 | 0.65 | 33 | – |
| Aeolian sand (8) | 125–200 | 0.51 | 27.0 | – |
| Quartz sand (2) | 190 | 0.55 | 31 | – |
| Tapioca pearls (3) | 200 | 0.74 | 36.5 | 39 |
| Quartz sand (4) | <246 | 0.58 | 30 | 105 |
| Quartz sand (7) | 200–300 | 0.77 | 37.6 | 1252 |
| Aeolian sand (8) | 200–315 | 0.57 | 29.7 | 85 |
| Quartz sand (11) | 250–500 | 0.78 | 30 | – |
| Glass microsphere (5) | 300–400 | 0.44 | 23.9 | – |
| Aeolian sand (8) | 315–400 | 0.45 | 24.2 | 160 |
| Quartz sand (6) | 300–500 | 0.60 | 30.9 | 20 |
| Castor sugar (1) | <355 | 1.14 | 48.8 | 247 |
| Sand SI (1) | <400 | 0.89 | 41.7 | 245 |
| Glass microsphere GMI (1) | 400–600 | 0.87 | 41.0 | 137 |
| Sand (9) | 500 | 0.58 | 30.1 | 300 |
| Sand (10) | 500 | 0.90 | 42 | – |

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