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Experimental micro-mechanics of granular media studied by x-ray tomography: recent results and challenges

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Combining x-ray tomography and three-dimensional (3D) image analysis has finally opened the way for experimental micro-(geo)mechanics, allowing access to different scales of interest. When these correspond to a scale that has been imaged at high spatial resolution, high-quality measurements can be obtained (e.g. 3D displacements and rotations of individual grains of sand sample under load). However, there are issues when the scale of interest is smaller, for example the characterisation of grain-to-grain contacts (their orientations and evolution) or production of fines by grain breakage. This paper presents a short selection of new grain-scale measurements obtained using existing techniques. The challenges associated with smaller scale measurements on the same images are also discussed through a few examples from ongoing work.

KEYWORDS: laboratory tests; sands; strain localisation

INTRODUCTION

When modelling grain-scale phenomena such as shear banding, standard continuum approaches are well known to have difficulties. Discrete element models are a valid alternative and have allowed insights to be gained into grain-scale behaviour of particulate matter, through simulations with simple interaction laws between the particles. However, fundamentally, these remain simulations and consequently require experimental observations at the grain scale for validation. X-ray tomography has been successfully used to image small specimens of sand, subjected to triaxial compression, in various different states of loading (e.g. Alshibli & Alramahi, 2006; Matsushima *et al.*, 2010; Andò *et al.*, 2012a, 2012b). When imaging a given assembly of particles with a fixed-resolution detector, a trade-off must be made between the detail with which the particles are imaged and the number of particles imaged. Mechanical representivity demands a sufficiently large number of particles, whereas a precise three-dimensional (3D) description of each grain requires a small specimen.

The specimens in this work were composed of around 50 000 grains (with a D_{50} of around 400 μm), which allows individual grains to be imaged (with around 5000 voxels per grain) and therefore successfully tracked throughout a test. However, there are related issues where imaging is ideally desired at a smaller scale – such as the characterisation of grain-to-grain contacts (orientations and evolution) or the production of fines by grain breakage. In this paper, new observations of grain-scale kinematics are briefly presented and the considerable challenges associated with smaller scale measurements (mentioned above) on the same images are discussed using a few examples from ongoing work. The philosophy adopted in this paper is that of making mechanically representative measurements rather than a high-detail characterisation of single occurrences of small-scale mechanisms; the long-term objectives are not to characterise breakage or contact orientations on a few grains, but rather to make quantitative measurements of breakage on a meaningful number of grains in order to statistically relate this quantity to macro-behaviour. The objective of this letter is therefore not to present major findings, but to discuss a general point. In doing so, some challenges and possible solutions are discussed that might make a significant impact on current understanding of the behaviour of granular materials.

EXISTING TECHNIQUES AND RESULTS

Triaxial compression tests on dense specimens of dry sand performed inside the x-ray scanner in Laboratoire 3SR (Fig. 1) allow specimens to be scanned at key points during the test. Since the objective of this work is to image each individual grain, the specimen size is considerably less than standard (22 mm height, 11 mm diameter). Further details on the experimental apparatus and procedures are reported by Andò (2013). A 3D image of x-ray attenuation in the scanned domain is obtained each time the specimen is scanned.

A variety of image analysis techniques have been developed to identify and measure individual grains in these 3D images. Very simply, these methods require the reliable identification of the inside of each grain by a marker, and the markers are then expanded to occupy all the grain volume by the watershed algorithm implemented in Noesis' Visilog 6.910, based on the work of Beucher (1991). When these procedures are applied to several different images from a test, ID-Track (see Andò *et al.*, 2012a) is used to follow the grains. This paves the way for measurements of the 3D displacement (by following each grain's centre of mass). The measurement of each grain's 3D rotation presents more of a challenge, which has been met by developing a hybrid ID-Track and digital image correlation (Andò *et al.*, 2012b).

These grain-based measurement tools are extremely powerful in that they allow micro-scale explanations of macro-scale responses. As an example, Fig. 2 shows the micro-mechanisms at play in the macroscopic residual

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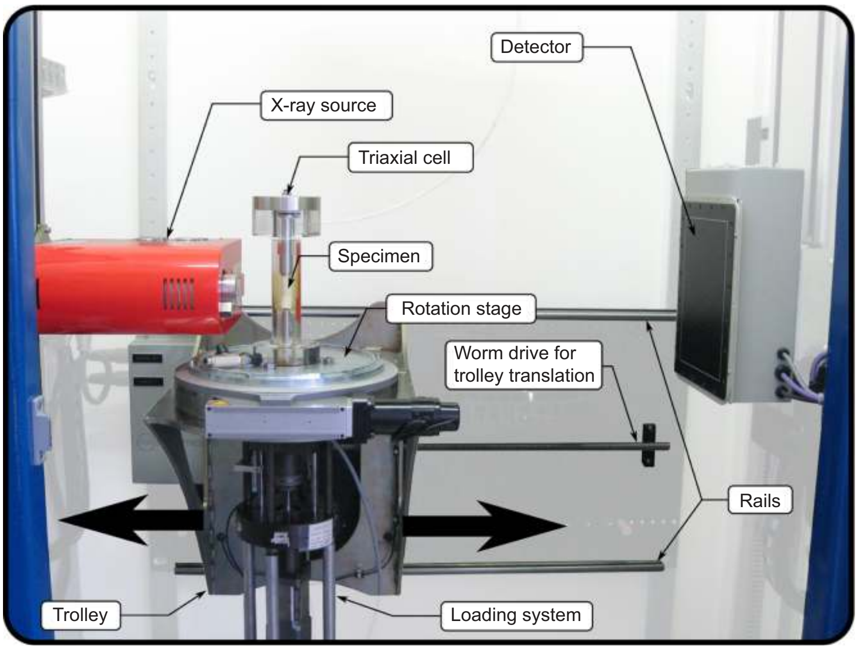

Fig. 1. Laboratoire 3SR x-ray scanner. The background is faded out for clarity; blue edges visible left and right are the cabin door frame

stress state for three different sands – angular Hostun HN31 sand, rounded Ottawa 50–70 and the very rounded Caicos ooid – all tested at 100 kPa confinement. The measured grain displacements described above were triangulated (in the style of Bagi (1996)) using YADE (see Šmilauer *et al.*, 2010), providing access to a grain kinematics-derived strain tensor. The second invariant of the strain tensor thus derived (maximum shear strain) is plotted (Fig. 2(a)) in the triangulated domain of the specimen. In all three cases, this deviatoric strain can be seen to be strongly localised into a thin band. For Hostun sand, discrete blocks of very intense shear in the direction of the band can be clearly seen. These blocks are situated inside a wider shear band than that in Caicos ooid and one which has a less abrupt change in shear strain in the direction normal to the band. The change in the shear strains in Ottawa sand in the direction normal to the band presents an intermediate case.

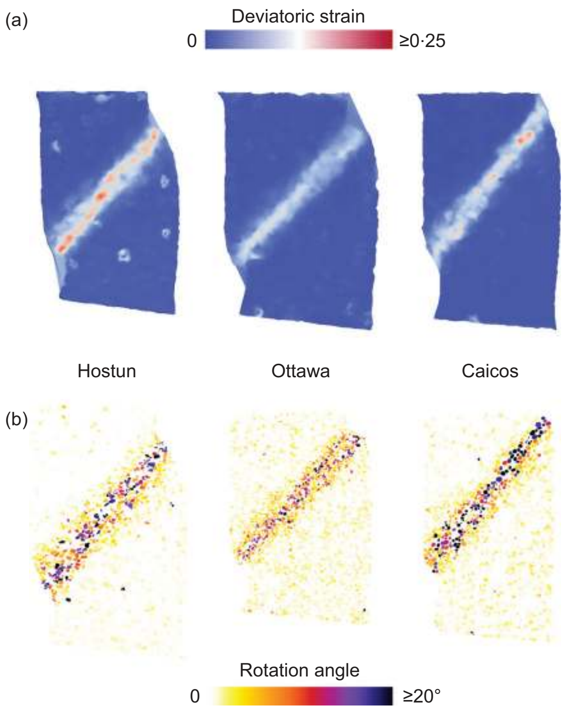

Fig. 2. Measurements made on increments in the residual stress state of Hostun sand, Ottawa sand and Caicos ooid tested in triaxial compression at 100 kPa confinement. The fields plotted are deviatoric shear strain (a) and the intensity of 3D grain rotation (b). Note that the strain increments are not all the same: the one for Hostun sand is particularly larger than the rest

The mechanism behind the increasing sharpness of shear strains with increasing grain roundness may find its origin in the grain-based rotation fields (Fig. 2(b)). These show a concentrated band of grains (six to eight grains across) with intense rotations coincident with the band of high shear strain in all three cases (the scalar shown is the angle in the 'axis and angle' representation of a 3D rotation). The values of grain rotations are very large for Caicos ooid, with many grains rotating more than 20°. The values of rotations for both Ottawa and Hostun sands in their residual stress increments appear to be the same, with grains rotating around 10–15°, but the axial strain increment obtained for Hostun is larger. Normalising grain rotations back to size of the increment studied in the other tests, rotations of 5–8° are obtained. The intensity of grain rotations therefore appears to be correlated to grain roundness in the residual stress state – in the rounded material, grains rotate in a thin band whereas, in angular material, grains rotate in a wider band with smaller rotations. In the Caicos and Ottawa specimens, when the shear band is fully developed, grain shapes offer little resistance to rotation (and therefore to shear), so the band is very concentrated. In the angular material however, grains are not able to rotate as freely due to interlocking; this causes 'secondary rotations' and therefore a wider band. Shearing this localised but interlocked system is likely to require more energy than shearing a localised material with less interlocking; this may explain why larger resistance to shear is obtained in the residual state.

It should be noted that the free-end platen at the bottom of all specimens in Fig. 2 tilts visibly with increasing deformation (this is even more striking in Fig. 5). Obviously, the specimen's overall response depends on these boundary conditions: not allowing rotation of the bottom platen (which is the alternative conventional boundary condition) would give a different specimen response; however, this is a completely different concept to that of material response and would only be a source of concern in the absence of the full-field information presented (i.e. in conventional testing).

CHALLENGES

Contact characterisation

A full micro-mechanical description of the kinematics occurring at the grain scale needs to go beyond grain kinematics (i.e. the rotations and displacements of grains shown in the previous section). In particular, it requires the characterisation of contact kinematics (the gain or loss of contacts and the orientation of those contacts) during deformation. In the work described thus far, the material was imaged at a resolution sufficient to resolve individual grains but not the details of the surface of each grain – these are visible in Fig. 3(b), which shows a voxelated image of a single Caicos ooid. When contacts between such voxelated objects are characterised using relatively standard image processing tools, the lack of detail at each contact surface will inevitably result in extremely biased measurements (see Andò *et al.* (2012b)). Figures 3(a) and 3(c) illustrate this challenge for 3D images of glass ballotini and Hostun sand, respectively.

When the solid phase of a 3D image is to be split into individual grains, a separation surface between each grain must be defined in some way in order to isolate individual grains – this is currently obtained using a classical watershed algorithm (using a Euclidean distance map). It is tempting to consider the voxels defining the separation surfaces directly as a volumetric representation (of unit thickness) of a contact. However, Fig. 3(a) and Fig. 3(c) underline how small the contact areas can be in these images (i.e. how few voxels are used in the definition of the separation surface). The orientations of these separation surfaces are highly dependent on the particular watershed implementation used (and, furthermore, are highly biased) and therefore cannot be considered accurate. This is clearly visible in Fig. 4, where the contacts between a series of

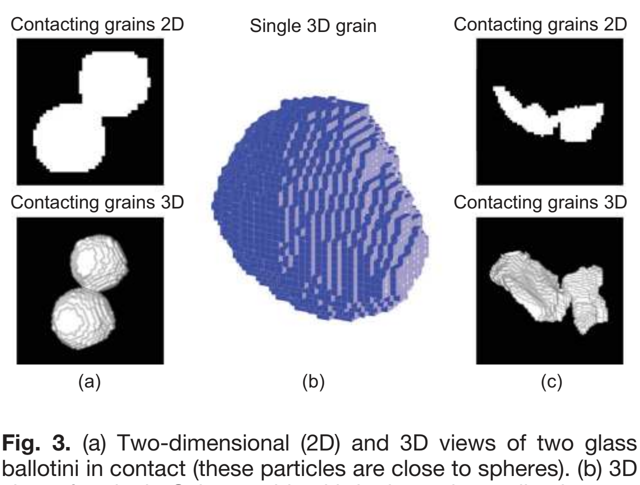

Fig. 3. (a) Two-dimensional (2D) and 3D views of two glass ballotini in contact (these particles are close to spheres). (b) 3D view of a single Caicos ooid, with its jagged, voxelised nature underlined (courtesy of J. Andrade). (c) 2D and 3D views of two Hostun sand grains in contact

automatically generated touching spheres are analysed. Figure 4(a) shows the orientation of the contacts as obtained from the branch vectors (i.e. the vectors connecting the centres of the two spheres in contact – this is the 'exact' distribution since, in the special case of spheres, contact orientation and branch vector coincide). It is clear that the 'measured' orientation distributions coming from classical watershed approaches (Fig. 4(b) and Fig. 4(c)) are both highly biased and substantially different from each other (reproducing an error already noted by Andò *et al.* (2012b)). More importantly, they are both a long way from the known distribution of orientations (Fig. 4(a)).

When a mechanically representative set of grains is imaged at a given resolution, this appears to cause serious problems with measurements of features at a smaller scale – in this case the local surface of the grains, which should yield the critically important information of contact orientation. Given the importance of this measurement, considerable efforts have been made in this direction. Recent work has involved the deployment of much more advanced techniques, whose results are shown in Fig. 4(d) for the case studied above. The technique is currently implemented on a 'per contact' basis (i.e. treating one pair of grains at a time). Starting from markers well inside the grains, it assigns – for each voxel of the solid phase – the probability of belonging to either marker using a power watershed. The contact plane is then estimated with sub-voxel precision by estimating the equal-probability isosurface between the two grains. Despite the considerable computational cost of this approach, it gives high-quality results that are extremely encouraging; Jaquet *et al.* (2013) present further details of the implementation.

Grain breakage

Grain breakage is another grain-scale phenomenon, and is responsible for the difference between the low- and high-pressure behaviour of sands. Several triaxial compression tests (up to the maximum cell pressure of 7000 kPa) were recently performed on Hostun HN31 sand with in situ x-ray scanning. The axial stress response of the specimen tested at 7000 kPa cell pressure (shown as a plot of q/p against axial shortening of the specimen in Fig. 5(b)) is considerably different from the response of the same material at 100 kPa cell pressure (the other test on Hostun sand discussed above, and also shown in Fig. 5(b)). The qualitative change in the axial stress response between 100 and 7000 kPa is confirmed by the high-pressure triaxial tests performed on the same material by Colliat-Dangus *et al.* (1988).

Figure 5 shows the micro-mechanisms occurring at high pressure, which go a long way to explaining the difference in the behaviour of this material at a higher mean stress: grain breakage can be seen to occur on a very large scale as the specimen is sheared. Figure 5(d)) follows a grain and its neighbours through five different states in the high-pressure test. These pictures show the grain in the centre of the image first cracking in two (with a vertical crack, images 2 and 3) and then being progressively broken into smaller pieces (images 4 and 5). These images suggest the progressive evolution of the specimen's grain size distribution (the production of fines during shearing): the clearly binary (i.e. grain and pore) image of state 1 develops into an image where pores are filled with an intermediate colour greyscale value representing a porous material – a new material whose grains are smaller than the voxel size. Tools such as ID-Track are clearly no longer relevant in this case since the grains are no longer persistent objects in the

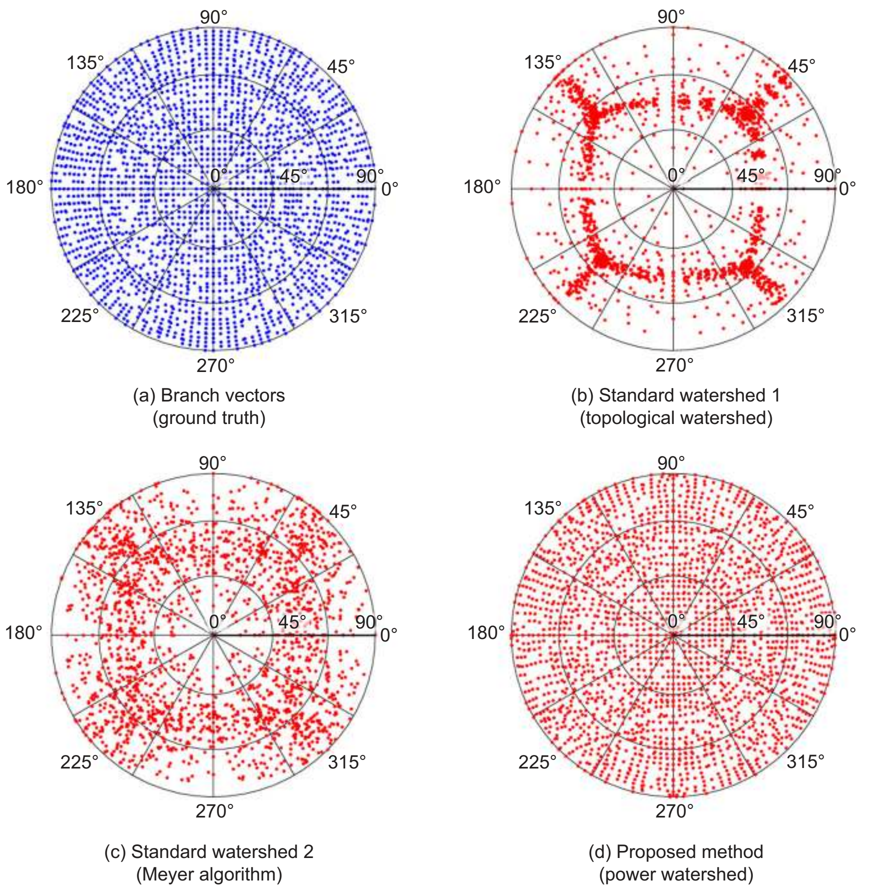

Fig. 4. Equal-area projection of contact orientations from simulated spheres (after Jaquet *et al.* (2013))

images and, furthermore, rapidly become so small that they cannot be individuated.

In order to quantify the amount of grain breakage occurring during a test, an ideal quantity to be able to evaluate locally would be grain size distribution. However, this presents an image processing challenge similar to the measurement of contact orientations: the scale of the object to be measured is now smaller than the grain scale and, therefore, less well-resolved. This paper does not present solutions to permit this measurement, but a useful point of entry might to measure the solid volume of the specimen before grain crushing begins, integrate the conservation of mass in the image processing in order to accurately measure the part of the grain size distribution where grains are larger than a voxel, and then estimate what is smaller than the voxel.

Bonded grains

Cemented granular materials are another example of the grain-based image processing challenges discussed in this paper: again, a useful number of grains must be included in a single image in order to have a mechanically significant specimen. This is done at the expense of the ability to resolve the cement that bonds the particles together, which is key to understanding its effect on the mechanical behaviour of the material. Furthermore, identification of the cement between particles can be further complicated if that cement has a similar x-ray attenuation coefficient to that of the grains: this means that in images resulting from x-ray tomography, there is no clear greyscale distinction between grains and cement (see Fonseca *et al.* (2013)).

CONCLUSIONS

As noted in previous work (in particular, see Andò *et al.* (2012a, 2012b)), high-quality kinematic measurements – although challenging – are now possible on the vast majority of the 50 000 grains (imaged to have around 30 pixels for a grain diameter) that make up the specimens tested in this work. The specimens tested measure approximately $30D_{50}$ in diameter and $60D_{50}$ in height; this is considered a small sample, but has been shown (Andò, 2013) to represent well the material behaviour of the materials tested.

This paper has attempted to highlight some of the challenges that arise when smaller-than-grain-scale measurements are required in the characterisation of the deformation of a granular material. This is quite a natural requirement: in situ x-ray tomography combined with advanced image processing now allows experimental micro-(geo)mechanics. The application of this tool can reveal different scales of interest.

When imaging the samples discussed in this paper, a trade-off had to be made between sample size and the precision of the description of each grain. Objects at a smaller scale are therefore described less precisely than would ideally be the case, but sample size cannot be safely reduced in order to benefit from a larger zoom. Consequently, the simplest approach is to accept the images as they are and try to meet the challenges of paucity of information with advanced techniques.

In the case of the natural progression from grain kinematics to the measurement of contact orientations and their evolution, the 'ordinary' approach to the orientation of a contact plane between two grains imaged

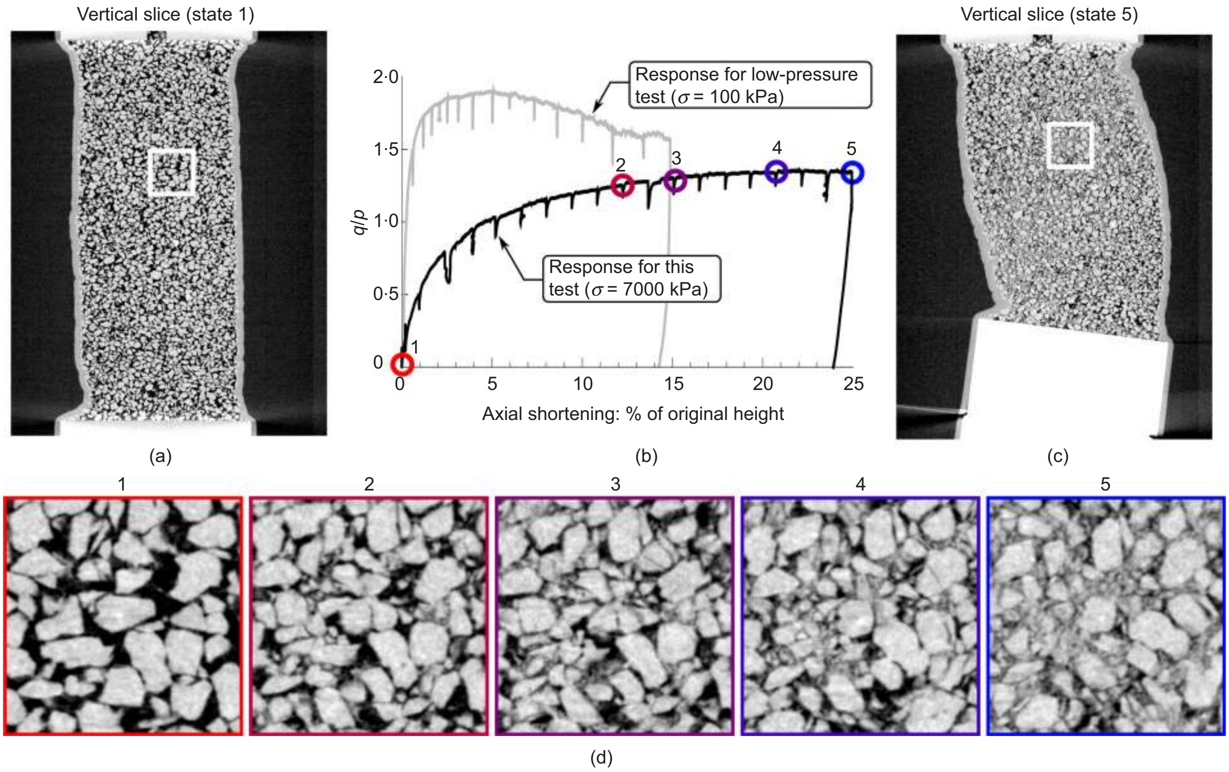

Fig. 5. Production of fines during a triaxial compression test on Hostun sand at a relatively elevated confining pressure of 7000 kPa: (a) and (c) vertical slices through the 3D image of the specimen after isotropic compression and at the end of deviatoric loading, respectively; (b) q/p versus axial shortening for a test on Hostun sand at cell pressure of 7000 kPa (black) and 100 kPa (grey); (d) zoomed images of the region shown in the full slices ((a) and (c)) for states 1–5 indicated in (b)

at this scale has been shown to introduce extreme bias into the measurements to the extent that they are no longer usable. Close collaboration with experts in image processing has allowed this measurement to be made successfully using very advanced techniques.

At higher mean stresses, imaging of the production of fines during shearing is exciting. Although local porosity can be calculated from these images, measurement of a grain size distribution from these images remains a challenge that requires the development of new tools.

Similarly, when a third phase – such as cement or a second fluid phase – is involved in the mechanics of deformation, a clear objective is to also identify this other phase in order to gauge its mechanical effect. This is another very challenging aspect, as noted by Fonseca *et al.* (2013).

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