8 COMPARISON OF MATERIALS' PROPERTIES AND ENDURANCE TESTING

8.1 Aim and considerations

Testing the stability of different graft materials designated for impaction grafting using the ovine or human model in block loading endurance tests is both time and material consuming and thus relatively costly. Simple, faster and less material consuming basic tests were conducted and it was hoped to find correlations between the characteristics measured by these fundamental tests and the stability properties found from the complex endurance tests.

Three simple, standardised and easily reproducible experimental designs and protocols were developed and used for testing different graft materials. These experiments were the die-plunger compression test described in chapter 4, the shear box test described in chapter 5 and the impactometer controlled cone impaction procedure which was described in chapter 7.4.3.

In the die-plunger test $10cm^3$ graft volumes were compressed quasi-statically in a constrained die of 10mm diameter to an initial peak force of 500N and after relaxation were re-compressed a second time to 1000N. Data collected were stiffness moduli, relaxation and elastic recoil from initial and secondary compression. Shear box tests applied a standardised experimental method of soil mechanics to bone grafts and derived the shear properties shear angle and cohesion. Controlled cone impaction monitored the set per impaction blow until full cone insertion was achieved.

8.2 Results and discussion

Results from the impactometer procedure and the correlation between impaction set and stability were analysed in chapter 7.4.3 and need not to be repeated here.

8.2.1 Shear properties

The shear box test was performed for three types of graft materials only because of the low sensitivity of the technique and the high graft volumes required. Figure 8.1 compares shear angles and cohesion coefficients of ovine bone, a 1:1 and a 1:9 b/c graft mix to the stability performance of pure bone, a 1:1 b/c mix and pure ceramic under block loading. It can be seen that the increase of the shear angle correlates well with the gain in stability against subsidence when the pure bone graft is mixed with a rising fraction of ceramic granules. This indicates that shear strength and in particular the shear resistance angle, plays a role in providing stability in impaction grafting. Cohesion as the second component in defining the overall shear strength dropped when ceramic granules were added to bone graft and their fraction was increased in the graft mix. As stability against subsidence nevertheless rose, the effect of cohesion on the total shear strength must be so minor that it seems irrelevant for the provision of stability in impaction grafting. Considering the Mohr-Coulomb failure envelope and the high normal stresses acting in all directions within a graft compacted and loaded as *in-vivo* and the experimental models it becomes clear that the total shear strength relevant for the loading conditions in impaction grafting is dominated by the shear angle.

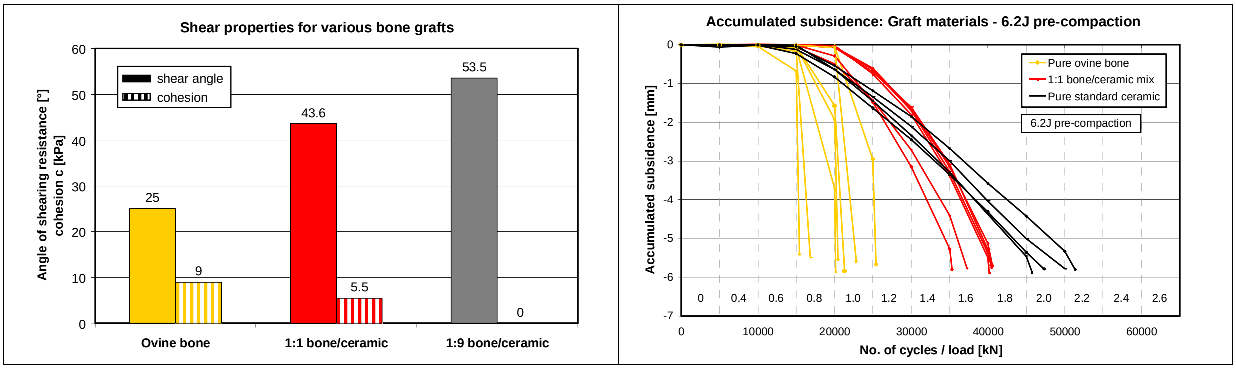

Figure 8.1: *Comparison between shear angles and cohesion (left) and stability against vertical subsidence (right) for ovine bone, a ceramic graft extender and bone/ceramic mixes.*

8.2.2 Compression properties

The correlation between compression moduli and stability against subsidence was very high and very sensitive for all graft configurations tested. Figure 8.2 shows the stress-strain curves for human and ovine bone graft during initial and secondary re-compression (left) and compares them to the subsidence curves accumulated under block loading for both grafts at the standard and high pre-compaction energy levels (right). In both cases the higher compression moduli measured are reflected in higher stability levels determined. It even seems that the increased relative modulus difference between initially compressed and re-compressed human and ovine bone samples is reproduced by the increased relative stability differences measured for the highest pre-compaction energy level.

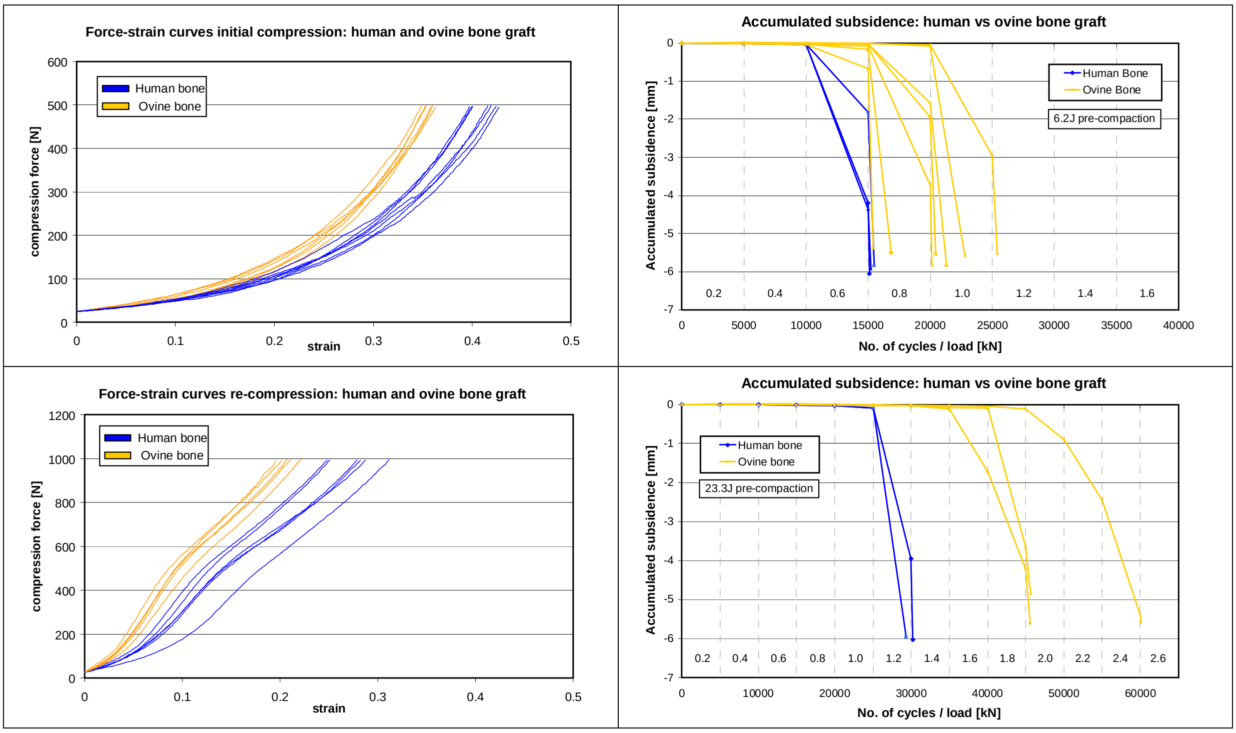

Figure 8.2: *Comparison between compression performance (left) and stability against vertical subsidence (right) for human and ovine bone .*

With the correlation between graft stiffness and stability against subsidence being sensitive enough to resolve a relatively small differences between ovine and human bone it was possible to correlate larger modulus and stability differences. Figure 8.3 compares, for various bone/ceramic mixing ratios, the stress-strain curves with the stability performance. Again a clear connection between stiffness and stability becomes obvious. The strong validity of the correlation and its potential to predict the stability performance of graft materials is confirmed by figure 8.4 where reversed stiffness ratios between bone and ceramic are also reflected in a reversed stability relationship between bone/ceramic mixing ratios. As seen in the left hand graph, the highly porous pure HA ceramic graft scored much lower modulus values than the standard HA/TCP granules and it was even less stiff than pure bone as the reference graft. Therefore, as seen in the right hand graph, the stability against subsidence of bone/ceramic mixes containing highly porous HA was lower than for pure bone and stability degraded further when the ceramic content was increased.

Such a strong and sensitive correlation between compression moduli and stability against subsidence indicates that graft stiffness and strength are critical properties for a bone graft material supposed to provide initial mechanical stability in impaction grafting. It also shows that graft compression in the tightly constrained die-plunger set-up activates similar load transfer mechanisms as present in the experimental tube-cone model or *in-vivo*.

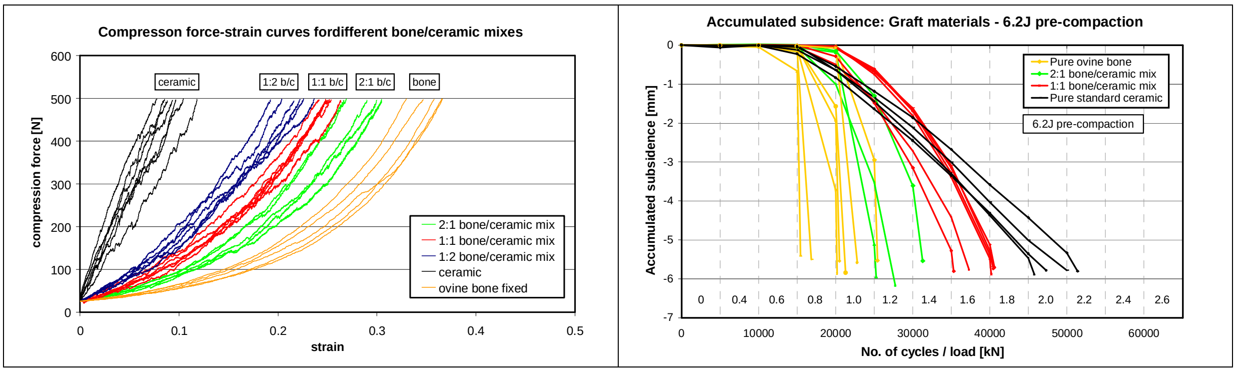

Figure 8.3: *Comparison between compression performance (left) and stability against vertical subsidence (right) for bone/ceramic graft mixes with standard ceramic.*

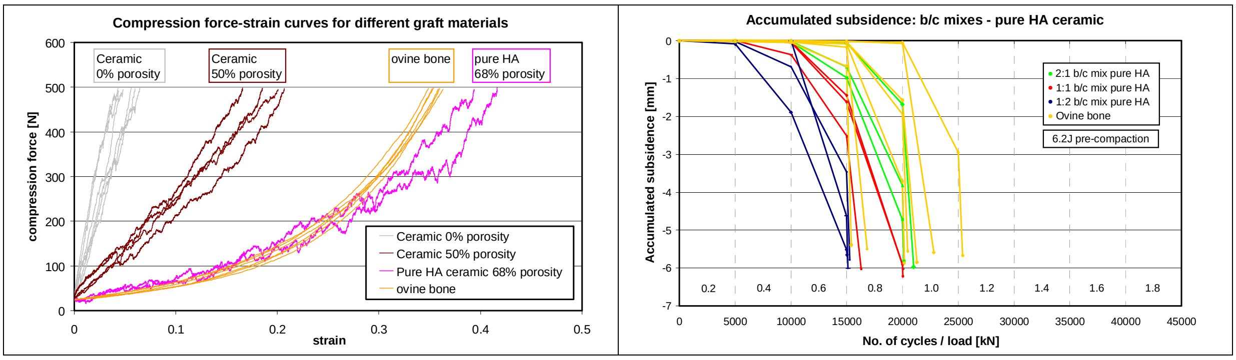

Figure 8.4: *Comparison between compression behaviour of bone and ceramic extenders (left) and stability against vertical subsidence (right) for b/c mixes with a highly 68% porous HA.*

When porosity, sintering temperature and chemical composition were varied for the ceramic phase the compression moduli of the pure granules allowed the prediction of the ranking of the stability performance of all 1:1 bone/ceramic mixes prepared thereof. Figure 8.5 shows for all ceramic configurations tested the compression secant moduli during initial compression (left) and secondary re-compression (right). In the same way as the stiffness decreases with increasing porosity or lower sintering temperature, the stability against subsidence drops for graft mixes comprising these ceramics as shown in figure 8.6 (right) and figure 8.7 (right) respectively. The stiffness difference between ceramic granules of reversed chemical composition was very small at least during initial compression and also this similarity was reflected in near equal stability levels during endurance testing as shown in figure 8.8 (right). The slightly superior endurance performance of the high HA composite ceramic came through in the higher modulus during secondary compression.

For particle size as a ceramic variable, the modulus-stability relationship could not be identified as clearly. During initial compression, small granules produced the highest while large granules possessed the lowest modulus but endurance test identified bone/ceramic mixes containing medium sized ceramic particles as the most stable combination (Fig. 7.33). Compression testing of ceramic granules with highly different sizes is strongly affected by the dimension of the die, especially when tested in pure form. Here, the particle size to die diameter ratio varies between less than 1.6 and more than 10, thus by almost one order of magnitude so that during initial compression the packing advantage of the pure, non-mixed smaller granules dominates. In a bone/ceramic mix with another phase present and after many impaction blows having fractured and re-organised larger granules, these differences are diluted and not as relevant for stability during endurance testing. However, during secondary compression the medium sized granules produced the highest compression modulus, an observation in agreement with the superior stability against subsidence recorded. It can be speculated that a re-compressed graft, like a densely impacted graft, has fractured and re-arranged in such a way that particle size distribution and load transfer are improved to lead to the higher compression modulus measured during re-compression. Medium sized particles which balance packing density, particle fracture and re-arrangement most effectively should benefit most from this mechanism. The factor of modulus increase for the re-compressed ceramics was 8.3 for the medium sized ceramic but only 4.6 for the small sized granules. Large granules also benefited from the stabilising mechanisms described and were seven times stiffer, confirming the theory.

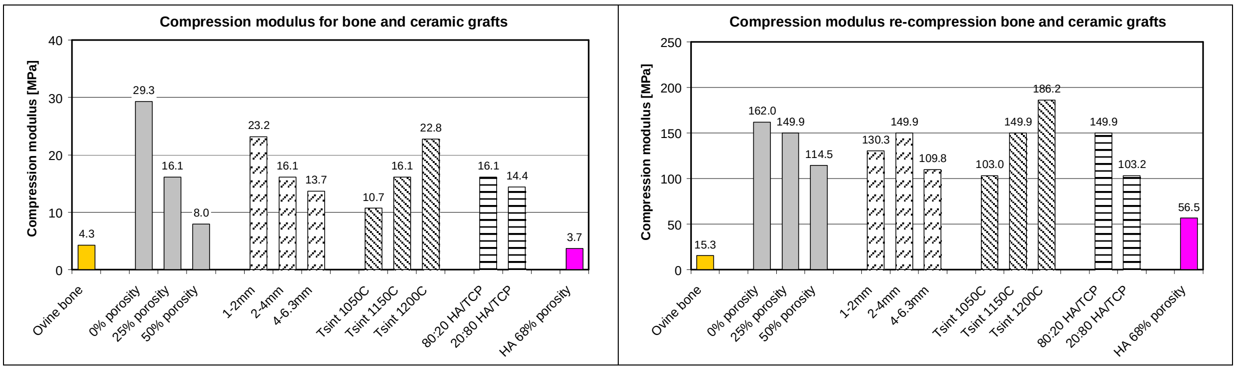

Figure 8.5: *Compression stiffness of pure bone and pure ceramic graft extenders during initial compression (left) and re-compression (right).*

Besides compression modulus, ceramic friability could be correlated to the initial mechanical stability of bone/ceramic mixes and thus can also serve to differentiate potential bone graft extenders with regards to their mechanical suitability for impaction grafting. Ceramic friability was quantified as the weight percentage of sub-millimetre particles produced after compression testing ceramic granules of originally 2-4mm size. The post-compression size distributions and in particular the dust weight fraction for pure ceramic grafts of different porosity, different sintering temperature and different chemical composition are given as bar charts (left) and compared to the corresponding subsidence curves (right) in figure 8.6, 8.7 and 8.8 respectively.

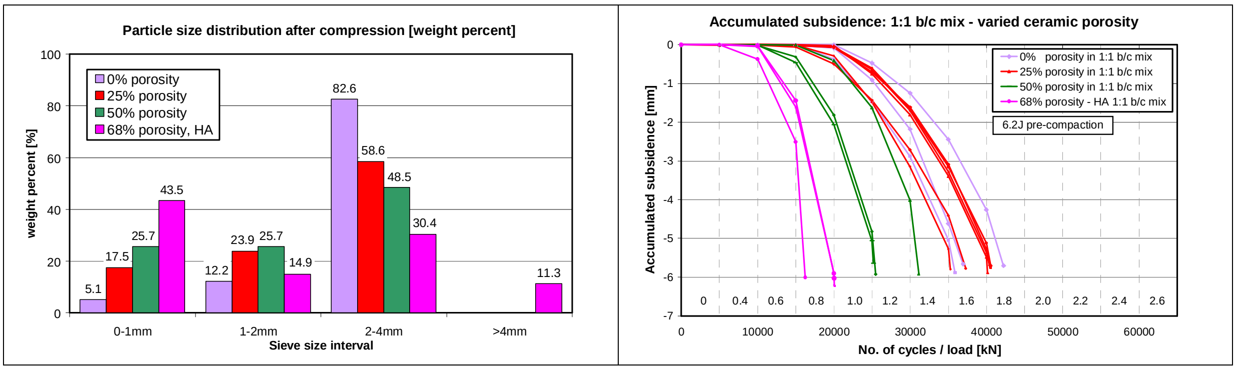

Figure 8.6: *Comparison between particle size distribution of ceramics after compression (left) and stability against subsidence for 1:1 b/c mixes. Varied parameter: Ceramic porosity.*

The lower the post-compression dust weight fraction of a ceramic graft extender the more stable a 1:1 bone/ceramic graft mix was against vertical subsidence. As figure 8.6 and 8.7 show for varied porosity and sintering temperature respectively, the correlation between dust-fraction and stability was not linear. Stability hardly fell when dust weight fractions were increased up to a level of ca. 20% but dropped massively once this value was exceeded. The dust weight fraction produced during compression testing the pure HA/TCP ceramic sintered at 1050°C was 28.7% and the stability recorded for a bone/ceramic mix containing such friable granules was only slightly higher than for pure bone. At 43.5% dust weight fraction measured for the highly porous HA, stability of a bone/ceramic mix was even lower than for the reference pure bone. The sensitivity of the dust fraction and stability relationship is underlined by the near equal fractions recorded for ceramic granules of the standard 80:20 reversed HA/TCP composition and the near equal stability against subsidence measured for both in a bone/ceramic graft mix (Figure 8.8). As ceramic particles of different original sizes produce different dust weight fraction under compression as a result of their initial size distribution, correlating their friability to stability does not make sense.

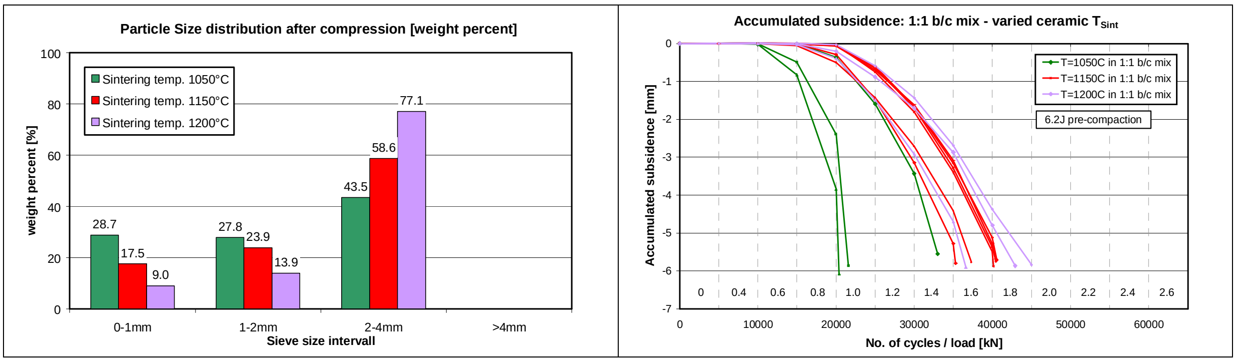

Figure 8.7: *Comparison between particle size distribution of ceramics after compression (left) and stability against subsidence for 1:1 b/c mixes. Varied parameter: Ceramic T_{Sint}.*

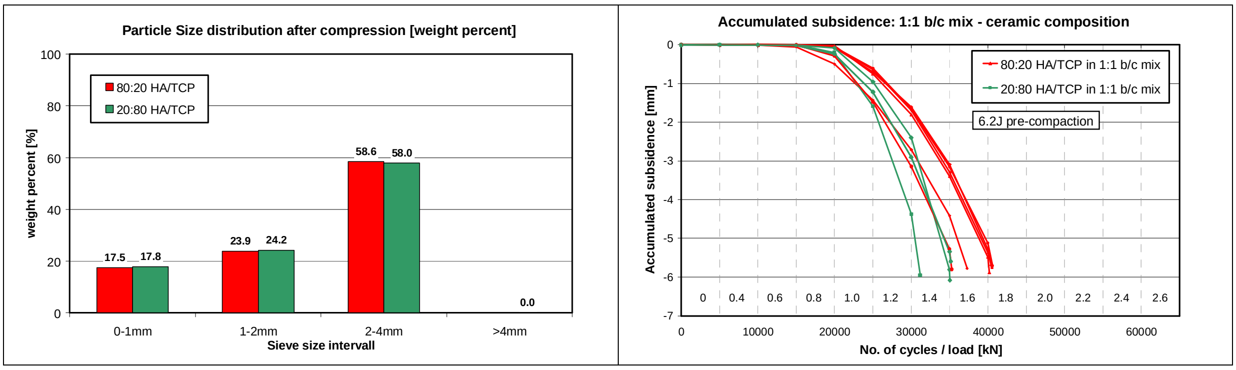

Figure 8.8: *Comparison between particle size distribution of ceramics after compression (left) and stability against subsidence for 1:1 b/c mixes. Varied parameter: Ceramic composition.*

The relationship between the post-compression dust weight fraction as a measure for ceramic friability and the stability of bone/ceramic mixes in impaction grafting shows that strength and hardness of the ceramic granules are a crucial contribution of the synthetic graft to the stabilisation of bone/ceramic mixes. Ceramics are brittle materials and cannot produce stability by elastic compression, a mechanism active in compacted bone grafts. The potential advantages of ceramic materials are high strength and hardness and as graft extenders these must be optimised to maximise the contribution to stability in a bone/ceramic mix. Ceramic granules in a graft mix support the strong trabecular fragments in load bearing as the ratio between elastic soft tissue phases and strong particles is shifted towards the latter.

Other characteristic graft properties identified during die-plunger compression testing like relaxation and recoil also helped to understand the different properties and load bearing mechanisms of different bone and ceramic grafts as discussed in chapter 4. However, a clear functional relationship between those characteristics and the stability against subsidence in impaction grafting could not be identified.

8.3 Conclusions

Three simple experimental methods were analysed to find out whether they are suitable to identify quantitative characteristics of bone graft materials which can be correlated to and thus predict the stability which those graft materials provide in impaction grafting experimentally and *in-vivo*. Shear-box tests and impactometer set were not sensitive enough to identify numerical properties of materials which could be used to differentiate the stability performance of the bone and synthetic grafts. Thus both tests are not well suited to select promising and identify unsuitable graft configurations prior to extensive endurance testing.

However, die-plunger compression tests of pure bone, pure ceramic and bone/ceramic mixes allowed compression moduli and dust production rates to be calculated which sensitively corresponded with the stability levels measured during endurance testing with the human impaction grafting model. The experimental method is particularly sensitive when the ceramic graft extenders are tested in pure form. Thus, compression moduli and post-compression dust weight fractions can be used to quickly preselect bone grafts and synthetic graft configurations with the potential for providing stability in impaction grafting. Using the die-plunger tests during the development of synthetic graft extenders could reduce the amount of time consuming and expensive endurance tests otherwise required for every graft configuration. The simple design, easy protocol, low cost, low graft consumption and fast procedure promote its use as a standardised tool for testing grafts.

The modulus or dust fraction to stability relationship was not valid when granule size was tested as a ceramic variable. Mechanisms leading to deformation under compression or to subsidence in impaction grafting are particle fracture and re-organisation within the confined granular aggregate and both mechanisms depend on the size ratio between the confining space and the granules. This is especially the case when size ratios below 10 mean that load transfer cannot be modelled as a bulk material as in soil mechanics but must also take into consideration shape, size, contact points of individual particles as well. In impaction grafting and during die-plunger compression testing of granules with the standard 2-4mm medium size, load transfer is a balance between soil like behaviour and the properties of individual granules. Thus changing the granule particle size in the compression tests without adapting the diameter of the die will produce distorted results. If die-plunger tests are meant to be comparable and meaningful, the ratio between die and particles must be fixed and should be around 2-4 to maintain the relevance for stability prediction established here.