Effect of hydrostatic pressure on flow and deformation in highly reinforced particulate composites

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Abstract: Infiltrated particle reinforced composites combine a dense matrix with particles that are in mutual contact and can therefore transfer compressive stress directly from one particle to the next. As a result, these composites may combine characteristics of the plasticity of their matrix with those of granular matter plasticity. We measure here the influence of a 200 MPa superimposed fluid hydrostatic pressure on the flow stress of high volume fraction (56 to 62 vol. pct) particulate Al₂O₃-Al composites produced by infiltration and show that the yield response of such composites is indeed pressure-sensitive. A simple analysis that transposes to metal matrix composites the theory of fluid-saturated granular media mechanics explains the phenomenon quantitatively.

Keywords: Particle Reinforced Composites, Yield behavior, Pressure Dependent Plasticity, Yield criterion, Granular Material.
1. Introduction

In metals, and also in some ceramics, plastic shear is produced by the multiplication and motion of crystal dislocations, or by the nucleation and growth of twins. Deformation then preserves volume and yield is insensitive to hydrostatic stress. The simplest laws governing metal plasticity are the von Mises yield criterion, which corresponds in principal stress space to a circular cylinder having the bissectrix (i.e. the hydrostatic axis) as its axis, and the associated flow rule.

Disordered or granular materials can also yield under stress to undergo permanent plastic deformation. In such materials, irreversible shear is caused by the relative sliding, along discrete surfaces, of elements making the solid in question: atoms in glasses, molecules in polymers, or particles in granular aggregates such as soil or packed powder. Here, relative sliding occurs along non-planar surfaces and hence does not preserve volume; yield is therefore pressure-sensitive in such materials. Granular media [1]-[2], polymers [3]-[5], metallic glasses [6]-[7] or fractured ceramics all have a yield stress that varies with the local hydrostatic stress. For isotropic granular media the simplest yield surface corresponds to the Drucker-Prager flow criterion, which traces, in principal stress space, a cone having again the bissectrix as its axis.

Consider now a composite combining both material types, namely a close-packed particle bed fully infiltrated with metal [8]-[11]. Plastic deformation in such a material requires the simultaneous operation of both mechanisms described above: crystal plasticity in the matrix, and also the relative motion of randomly packed ceramic particles. We present here an exploration of the plastic deformation of such a material, aiming to elucidate how both yield mechanisms operate and interact in such a material. In particular, we seek to know whether this class of composites carries the signature of granular material deformation, namely a flow stress that increases with the level of superimposed (compressive) hydrostatic stress.
In work to date, the influence of superimposed pressure on yield, deformation and ductility in metals reinforced with ceramic particles has been investigated in metals reinforced with non-touching ceramic particles, using high-pressure testing rigs that immerse samples within a fluid-filled pressure vessel through which uniaxial stress can additionally be applied [12]-[18]; data up to 1998 are reviewed in detail in Ref.[19]. The influence of tensile hydrostatic stress has also been assessed using tensile test specimens having different machined notches, which induce varying levels of hydrostatic stress in the narrowed section of material [20]-[27]. All of the above studies were conducted on composites containing up to roughly 25% ceramic particles by volume. In composites containing 40-60% (touching) particles, the influence of tensile triaxiality on deformation and fracture was investigated by Hauert et al. [28]. Results showed that, as the triaxiality ratio increases from 0.3 to 1.3, the yield stress does not vary much while fracture occurs at higher uniaxial stress but also lower deformation.

We explore here the deformation of such composite materials under high compressive triaxiality; as will be seen, hydrostatic pressure does influence the flow stress of this class of composites and the phenomenon can be rationalized and quantified using a simple approach.

2. Experimental methods

2.1 Materials - Composites of densely packed alumina particles embedded within a matrix of dense aluminium, Fig. 1, were produced by gas-pressure infiltration, where flow of the liquid metal is driven against adverse capillary forces into the open pores of a close-packed ceramic preform using pressurized argon gas (Refs. [9] and [29] describe the process). In order to vary the reinforcement volume fraction in the composite, the preform was packed either (i) to its maximum tapped density or (ii) by cold isostatic pressing (CIP) at 250 MPa.

Specifically, we employ a matrix of 99.99% pure Al and use two types of particulate reinforcement, namely (i) polygonal- and (ii) angular-shaped Al₂O₃ particulates, both of average particle size near 10 µm. The polygonal particles (designated by their maker as
AA10) are produced under the tradename “Sumicorundum” by Sumitomo Chemicals (Osaka, Japan) using a proprietary process. The angular particles, produced by comminution and supplied by Treibacher Schleifmittel (Laufenburg, Germany), are designated as F600 powder. The polygonal particles have faceted near-spherical shapes, whereas angular particles are more irregular and characterized by sharp asperities. Polygonal Sumicorundum particles are of high internal perfection and produce tough and ductile composites, while comminuted angular particles are of lower perfection, resulting in somewhat weaker composites [8]-[11][29]-[36].

The reinforcement volume fraction, \( V^{(r)} \), was determined by densitometry prior to testing (before gluing the strain gages) knowing that these composites are pore-free [29]. Since testing of the matrix-free particle beds under elevated hydrostatic pressure was not practical, composites with identical particulate reinforcements but with a highly compliant epoxy matrix were also made. The two-component epoxy that was used is named LME10435/LME10346 by its producer, Huntsman (Basel, Switzerland); it is mostly employed to produce aerospace composites. This resin was selected for its low mixed viscosity (which makes it easy to inject) and low tensile modulus (\( \approx 2 \) MPa) and also for its good bonding properties with alumina. Alumina-epoxy composite ingots were made by gas-driven pressure infiltration of ceramic preforms prepared similarly as were corresponding metal matrix composites. Before injection of the epoxy under vacuum, air was evacuated from the preforms placed in a crucible and vacuum was maintained for 3 hrs. The crucible assembly was then enclosed within an infiltration apparatus and argon pressurized to 5 MPa was injected, reaching peak pressure in approximately 5 min. Pressure was maintained overnight until complete polymerization of the epoxy matrix. The \( \text{Al}_2\text{O}_3 \)-polymer ingots were finally post-cured at 80 °C for 5 hrs. It was checked by densitometry (using the epoxy density
given by the datasheet, namely 1.1-1.2 g/cm³) that composites E-A61/E-P62 and E-P56 feature on average the same vol. pct reinforcement as the corresponding metal composites.

A summary of all composites produced for this study is presented in Table 1. The following designation is used: in “composite M-XY”, M designates the matrix material (Al for aluminium, E for epoxy), X denotes the particle shape (A for angular, P for polygonal) and Y the reinforcement volume fraction expressed in %. Test specimens with a nominal gauge length of 14 mm and a gauge section of 4.5 x 7 mm² were machined from cylindrical composite ingots that result from the infiltration process. Al-Al₂O₃ and epoxy-Al₂O₃ samples were machined by electro-discharge machining (EDM) and by milling, respectively.

<table>
<thead>
<tr>
<th>Composite designation</th>
<th>Particle specifications</th>
<th>Reinforcement volume fraction [%]</th>
<th>Average particle size [µm] and shape</th>
<th>Preform packing procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-A61</td>
<td>F600ᵇ</td>
<td>61.1 ± 0.8</td>
<td>9.3 µm, angular</td>
<td>CIP at 250 MPa</td>
</tr>
<tr>
<td>Al-P62</td>
<td>AA10ᵃ</td>
<td>62 ± 0.7</td>
<td>10 µm, polygonal</td>
<td>CIP at 250 MPa</td>
</tr>
<tr>
<td>Al-P56</td>
<td>AA10ᵃ</td>
<td>56.5 ± 0.9</td>
<td>10 µm, polygonal</td>
<td>Maximum tap density</td>
</tr>
<tr>
<td>E-A61</td>
<td>F600ᵇ</td>
<td>61.3 ± 0.3</td>
<td>9.3 µm, angular</td>
<td>CIP at 250 MPa</td>
</tr>
<tr>
<td>E-P62</td>
<td>AA10ᵃ</td>
<td>61.9 ± 1.3</td>
<td>10 µm, polygonal</td>
<td>CIP at 250 MPa</td>
</tr>
<tr>
<td>E-P56</td>
<td>AA10ᵃ</td>
<td>56.5 ± 0.4</td>
<td>10 µm, polygonal</td>
<td>Maximum tap density</td>
</tr>
</tbody>
</table>

Table 1. Al/Al₂O₃ and Epoxy/Al₂O₃ specifications (ᵃ Manufacturer classification; ᵇ FEPA (Federation of European Producers of Abrasives) standard 42-1984 R 1993).

2.2 - Fluid immersion triaxial testing. A dedicated apparatus was built to apply simultaneously a variable axial load and a controlled hydrostatic pressure to a test specimen enclosed and immersed in a fluid within a pressure chamber. During a test, the specimen gauge section is subjected to an axisymmetric triaxial stress state defined by (i) the axial stress (itself defined by both the fluid pressure and the uniaxial load applied outside the pressure vessel by a universal testing machine) and (ii) the lateral stress, uniformly equal to the negative of the fluid pressure (counting stress as positive when it is tensile).
The pressure chamber is designed to withstand a maximum fluid pressure $P = 200$ MPa. It is fixed within a screw-driven universal testing machine with a load capacity of $\pm 100$ kN. The fluid, Monoplex® DOS mineral oil, is pressurized externally and fed to the vessel through high-pressure fittings and pipes. Before filling the vessel with the fluid, vacuum is pulled so as to bleed possible air pockets. The fluid pressure is brought to the desired value by rotating a fine-thread spindle hand-pump while $P$ is read on a pressure transducer designed for 700 MPa operation with $\pm 0.3\%$ accuracy, fitted to a T-valve outlet in the pressurization unit.

A vertical load-train is assembled within the pressure vessel, which is fixed on the static lower platen of the uniaxial testing apparatus frame and connected on its lower surface to the pipe linking the outlet of the pressurization unit to the chamber. A schematic of the force-train in the apparatus is shown in Fig. 2a. It comprises (i) the uniaxial testing load train including a load cell, (ii) a piston traversing the pressure vessel and (iii) the test specimen enclosed and immersed within the fluid-filled chamber. The vessel has a feed-through for the piston, whose upper end is fixed to the moveable crosshead of the universal testing apparatus. At its lower end, the piston is engaged to the specimen upper shoulder, to which it transmits the quasi-static axial force generated by the universal test rig. The test specimen lower shoulder is connected by means of a similar dovetail to a sample holder directly screw-fitted into the (static) pressure vessel upper surface, thus completing the load-train.

Load and strain are both measured by means of electrical resistance strain gauges (SGs) situated inside the pressure chamber. A high-pressure feed-through for electrical wires is employed to duct signals through the vessel walls (see Fig. 2b). Its design is similar to that developed by Balzer and Sehitoglu [37]; this uses a steel cone fitting inside a mating bore of identical tapered angle drilled into a stainless steel plug, which itself seals against the pressure vessel wall with a conical fitting. Pressure acting onto the plug is thus resisted by the steel cone, while a thin layer of epoxy, filling the gap between the mating
surfaces of the bore and the steel cone, prevents fluid leakage. Enamelled 100 µm diameter electric wires are embedded into a layer of epoxy that was selected for its high ductility and good wetting properties with steel; the latter were enhanced using a silane coupling agent [38]. Signals from (i) the external load cell, (ii) the movable crosshead and (iii) the pressure transducer are collected using a National Instrument NI 6221 37-pin data acquisition card, whereas load and strain signals from bridge-based transducers inside the chamber are collected via a National Instrument NI USB 9237, the latter providing four connections for quarter, half, and full Wheatstone bridges. All transducer readings are recorded and then written into a file, for subsequent analysis and processing, by means of a Labview computer program. Tests are conducted in monotonic compression, with occasional elastic unload/reload sequences.

2.3 – Load measurement. The vessel has a feed-through for the piston, which passes through a custom-designed seal where it exits the vessel (see Fig. 2a): frictional forces then arise along the pressurized seal interface as a combination of static and dynamic friction. To measure load free of friction effects, we use a strain gauge load cell situated inside the vessel. Several studies have shown that accurate and reproducible load measurements can be achieved by means of fluid-immersion strain gauge load transducers [3]-[7], [14]-[19] and [39]-[47]; these compensate for variations in both pressure and temperature on strain gauges while having high sensitivity. The piston rod, constructed of Böhler (Kapfenberg, Austria) V155 tool steel, is designed to double as an internal load cell, in a design conceptually similar to that of Sakata et al. [39]. This uses two sets of 0-90° strain gauges arranged in a full Wheatstone bridge circuit which adds strain signals to give a measurement of the friction-free uniaxial force (net of pressure) applied onto the sample. To ease gauge installation, we use commercially available T-rosette strain gauges, which have two measuring grids made of Constantan alloy mounted on a single polyamide backing and offset by 90°. Each set of 0-90°
strain gauges is wired on an adjacent arm of the bridge circuit and is glued using hot curing epoxy resin on either side of the piston, Fig. 2b. The adopted strain gauge arrangement on the internal load cell and in the bridge circuit cancels the superimposed bending strains (caused by small eccentricity in the load axis) and suppresses both thermal and pressure effects. Curvature effects on the strain gauge response [48]-[49] are also nulled since SGs are installed on a flat surface, Fig. 2. Calibration of the load-measuring piston is conducted at atmospheric pressure using a steel specimen of identical geometry as a test specimen. In order to investigate the pressure sensitivity of the gauge factor, a tensile test was conducted at both $P = 0.1$ MPa and $P = 200$ MPa on a Al$_2$O$_3$-Al composite specimen of identical geometry as the test specimen, and Young’s modulus was measured during unloading from $\approx 0.2$ % strain and compared with results of the Impulse Excitation Technique (IET) conducted using a Grindosonic® apparatus (Lemmens Elektronics, Leuven, Belgium) [50]. Measurements at ambient and high pressure were found to differ by less than 1%, in agreement with previous studies [39]-[42] and [51]. The experimental Young’s modulus was found to be 5% lower than the modulus measured by IET; this was deemed to be a satisfactory level of precision.

2.4 – Strain measurement. Electrical resistance strain gauges (SGs) with small measuring grids were used to fit within the limited space available inside the pressure chamber; several studies have indicated that the pressure sensitivity of commercial metal foil SGs is less than $1 \mu \varepsilon$/MPa [48]-[49],[51]-[54]. We measured lateral and longitudinal strains individually and simultaneously by collecting the signals coming from two strain gauge-based transducers, with each strain sensor employing two sets of one “active” and one “dummy” strain gauge wired into a full-bridge arrangement inside the pressure chamber. For ease of installation, we used T-rosette strain gauges with two measuring grids (3-mm long and offset by 90°) mounted on a single polyamide backing; these can elongate up to 5 % in either direction. Pressure and temperature compensation are achieved by wiring active and dummy gauges on
adjacent arms of the bridge (as in Refs. [54] and [37]). Possible specimen misalignment effects are averaged out by wiring two active gauges, glued on both sides of the test specimen, on the opposite side of the bridge. The strain sensor is calibrated before each test in two steps: i) software offset nulling and ii) shunt calibration (i.e. gain adjustment) using a 100 kΩ shunt resistor internal to the NI9237 USB module.

3. Experimental results
The composites were either tested in ambient pressure or under a constant superimposed hydrostatic fluid pressure $P = 200$ MPa. All tests were conducted in displacement control at a nominal strain rate of $10^{-4}$ s$^{-1}$ and stopped before sample failure. For consistency, electrical resistance SGs of the same type were employed in all tests, namely Series Y- 350 Ω nominal resistance SGs purchased from HBM (Zurich, Switzerland). Note that when T-rosette SGs were used to collect individually and simultaneously multiaxial strain paths, signal saturation occurred in the data acquisition system when the axial strain reached 2.2%. Axial strains past this value could then be measured by connecting only one strain sensor (namely the one measuring longitudinal strain) to the data acquisition system.

In what follows, the conventional form of invariants for axisymmetric compression triaxial testing is used to represent engineering macroscopic stress and strain, namely: $\Sigma_{\text{eff}} = (\Sigma_1 - \Sigma_3)$, $\varepsilon_v = (\varepsilon_3 + 2\varepsilon_1)$, and $\varepsilon_{\text{eff}} = \frac{2}{3}(\varepsilon_1 - \varepsilon_3)$. The effective stress, $\Sigma_{\text{eff}}$, corresponds in magnitude to the uniaxial compressive stress (net of pressure) on the test specimen. The effective strain, $\varepsilon_{\text{eff}}$, is proportional to the difference between the lateral and axial strains, $\varepsilon_1$ and $\varepsilon_3$ respectively, while the volumetric strain, $\varepsilon_v$ corresponds to the first invariant of the strain tensor. We use a negative sign for compression or contraction, and a positive sign for tension or dilatation.

Stress-strain curves for Composites Al-A61, Al-P62 and Al-P56 tested under elevated fluid pressure $P = 200$ MPa are given in Fig. 3 together with corresponding curves obtained at
atmospheric pressure. Comparing the Al matrix composite yield response under high pressure with that measured at atmospheric pressure shows that an increase in flow stress is brought by the superimposed hydrostatic pressure. Furthermore, this effect increases (i) with the extent of prior composite deformation and (ii) with increasing vol.pct ceramic. Figure 3 shows that when a composite tested under \( P = 200 \) MPa is reloaded after depressurization (dashed black, magenta and blue curves in Figs. 3a, b and c, respectively) the composite flow stress falls onto the base line curve obtained at atmospheric pressure. Results are also overall nicely reproducible; the slight difference among flow stress curves of Composite Al-A61 at atmospheric pressure can be rationalized by consideration of its reinforcement, which being of lower perfection than polygonal particulates results in composites that are more susceptible to the accumulation of internal damage [8]-[11][30]-[36].

Volume changes during triaxial testing of the composites could be computed when T-rossette strain gauges were used. Figure 4 shows plastic volume changes during compressive deformation at \( P = 200 \) MPa and \( P = 0.1 \) MPa; for all three composites identical curve colours correspond across Figs. 3 and 4 to the same specimen. To subtract its elastic component from the total volumetric strain, we measured the composite linear compressibility under hydrostatic pressures up to \( P = 200 \) MPa by employing a test object of identical gauge geometry and using a similar longitudinal SG sensor as those adopted for triaxial testing, with dummy gauges installed on a tungsten specimen of known compressibility. The measured bulk moduli of Composites Al-A61, Al-P62 and Al-P56 are respectively 153±1 GPa, 154±3 GPa and 139±5 GPa. Data in Fig. 4 show that:

i) measured plastic volume changes are small for all Al-matrix composites tested in this study; values are of the same order as the scatter between similar measurements, Fig. 4a;

ii) this said, a tendency for the volume to increase slightly with plastic deformation can be discerned at \( P = 200 \) MPa for all Al-matrix composites;
iii) there is evidence of the onset of dilation (if any is present) being delayed at high pressure for Composite Al-A61 (compare dashed blue and solid red curves in Fig. 4b);

iv) within the range of strain explored (less than 2.5%) and within the scatter of data, compressive deformation of Composites Al-P62 and Al-P56 occurs without measurable plastic volume change at atmospheric pressure, while there is a slight tendency for the composite volume to increase when it is deformed at $P = 200$ MPa.

Results of tests conducted with epoxy matrix composites at $P = 200$ MPa or atmospheric pressure are shown in Fig. 5. A significant increase in flow stress is brought by the superimposed hydrostatic pressure and the effect is observed to increase (i) with the extent of prior composite deformation and with (ii) increasing vol. pct ceramic.

<table>
<thead>
<tr>
<th></th>
<th>Effective stress, [MPa]</th>
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<tbody>
<tr>
<td></td>
<td>$P = 0.1$ MPa</td>
</tr>
<tr>
<td>$\varepsilon_3 = 1.5%$</td>
<td>346 ± 4</td>
</tr>
<tr>
<td>$\varepsilon_3 = 2.22%$</td>
<td>396 ± 5</td>
</tr>
<tr>
<td>$\varepsilon_3 = 3%$</td>
<td>435 ± 6</td>
</tr>
<tr>
<td>$\varepsilon_3 = 1.5%$</td>
<td>55 ± 2</td>
</tr>
<tr>
<td>$\varepsilon_3 = 2.22%$</td>
<td>---</td>
</tr>
<tr>
<td>$\varepsilon_3 = 3%$</td>
<td>---</td>
</tr>
<tr>
<td>Al-A61</td>
<td>327 ± 4</td>
</tr>
<tr>
<td>E-A61</td>
<td>13</td>
</tr>
<tr>
<td>Al-P62</td>
<td>383 ± 2</td>
</tr>
<tr>
<td>E-P62</td>
<td>13</td>
</tr>
<tr>
<td>Al-P56</td>
<td>230 ± 1</td>
</tr>
<tr>
<td>E-P56</td>
<td>4</td>
</tr>
</tbody>
</table>

Table 2. Average uniaxial flow stress measured under axisymmetric compression at $P = 200$ MPa and $P = 0.1$ MPa across all samples and its corresponding standard deviation at different axial strain $\varepsilon_3$. Note that --- indicates that signal saturation had occurred before reaching the corresponding value of strain.

4. Discussion

Hydrostatic pressure increases the composite flow stress: for all three composites, Figs. 3 and 5, the flow curve at $P = 200$ MPa exceeds that measured under atmospheric pressure by an amount that depends on the type of particle, its volume fraction, the strain, and the matrix. To
compare the various systems, the average uniaxial flow stress measured across all samples and the corresponding standard deviation at discrete values of axial strain are indicated (where available) in Table 2. Triaxial strain measurements show first that, for all three metal matrix composites and for all applied strains of this work (extending up to \( \approx 2.5\% \) strain), the volumetric plastic strain remains small, around a few tens of percent. This agrees with what was reported for similar composites in Fig. 7 of Ref. [55]. As a consequence, curves of effective (von Mises) stress versus axial strain essentially superimpose on curves of effective stress versus effective strain for all composites of this work.

The observed tendency for volume to increase upon plastic deformation in the metal matrix composites at \( P = 200 \) MPa is consistent with what is documented for low porosity rocks (i.e. rocks whose initial porosity is less than 5\%), in which compaction is found to be small or absent, and dilation predominant, even at high confining pressures [2], [56]-[59]. Composites produced for this study contain indeed negligible levels of initial porosity [29] and therefore are unlikely to show the transition, from a strong dilatant behaviour (often accompanied by barrelling of the tested specimen) to compaction, that is observed with increasing confining pressure in high-porosity rocks and soils (see Refs. [56]-[58] and [60]), or in model cermet composites produced in a parallel study by Pickering et al., who show (see Fig. 4 of Ref. [61]) that the effect results from the collapse of voids within the material. The more brittle Composite Al-A61 of this work shows some dilation when it is deformed under atmospheric pressure: this is explained by vertical cracking of the ceramic particles during uniaxial compression; see Ref. [55]. For this composite, the observation that dilation is suppressed under \( P = 200 \) MPa suggests that hydrostatic pressure reduces or suppresses such cracking. The influence of applied hydrostatic pressure on the flow stress of the present composites can have two complementary origins, namely (i) a reduced level of damage accumulation, or (ii) an increased contribution of the packed particle bed to the composite flow stress at high \( P \).
That damage is not the cause for the observed flow stress increment is shown by the observation that, when a composite is deformed under $P = 200$ MPa and reloaded at atmospheric pressure, its flow stress superimposes quite precisely over that of the same composite deformed to the same strain entirely under atmospheric pressure, see Fig. 3. This shows that the composite deformed under $P = 200$ MPa has sustained damage, the effect of which on the flow stress is identical to damage produced after deformation under ambient conditions. The increase in flow stress cannot, therefore, be ascribed to a variation with $P$ in the rate of damage accumulation.

That damage is not the cause for the flow stress increment observed under elevated hydrostatic pressure is corroborated, for composites reinforced with the stronger polygonal particles, by the measured effect of hydrostatic pressure on deforming metal composite dilation, Figs. 4a&c. It is known that internal damage within composites of this class takes the form of particle cracking and/or matrix voiding at sites of high triaxiality [8]-[11],[30]-[36][55]. Since both damage mechanisms come with an increase in composite volume, the fact that these two composites do not expand less when compressed under $P = 200$ MPa (see Figs. 4a&4c) suggests that, within the strain range explored here, they do not accumulate less internal damage when they deform under elevated hydrostatic compressive stress.

The increase in composite flow stress that is brought by superimposed fluid pressure is, thus, caused by an increased contribution of the particles to the load borne by the composite. This is easily rationalized as follows: since in the present composites ceramic particles are in mutual contact, friction between touching particles at their contact points will cause the composite to dissipate additional energy when it deforms under pressure, exactly as a dense granular aggregate does. This will in turn cause the composite flow stress to be raised when it is deformed under elevated hydrostatic pressure. In this light, two of the trends observed are easily rationalized:
i) that the pressure-enhancement of the flow stress is greater when $V^r$ increases (62% vs 56.5% AA10 reinforced composites), with a matrix of either aluminium or epoxy (Figs. 3 & 4), is explained by the increase in volumetric density of interparticle contacts;

ii) that the flow stress increase caused by the superimposed pressure increases with the extent of composite prior deformation is to be expected, since this behaviour is generally observed in granular materials [1] and rocks [2].

The particle shape exerts, at equal $V^r$ ($\approx 61\%$), a relatively small influence, Table 2. On the other hand, the increment in flow stress is, in absolute value, clearly higher for the epoxy than the metal matrix composite. We propose the following rationalization of this effect.

Assume that, when the composite is deformed, the particles move in roughly similar manner, one with regard to the other, whatever the value of $P$. More precisely, assume that, at a given composite $\varepsilon_{eff}$ (essentially equal, in present experiments, to $\varepsilon_3$ since $\varepsilon_v \approx 0$) reached after the same proportional loading, relative particle motion remains the same whatever the matrix present between the particles. If we also assume that the law governing friction at particle-to-particle contact points is unaffected by the matrix, then the absolute increase in flow stress should be the same, regardless of the matrix embedding the particles. One should in other words observe the same increase $\Delta$ in flow stress $\Sigma_{eff}$ when either of the aluminium and epoxy matrix composite is deformed under $P = 200$ MPa:

$$\Delta \Sigma_{eff}^{MMC}(\varepsilon_{eff}) = \Delta \Sigma_{eff}^{EMC}(\varepsilon_{eff})$$

(1)

In Eq. (1) superscripts “EMC” and “MMC” stand for “epoxy” and “metal” matrix composite respectively. Now, the experimental data (Figs. 3 & 5 and Table 2) show that this is not observed. Indeed, if one uses interparticle friction data from the epoxy matrix composite, i.e. $\Delta \Sigma_{eff}^{EMC}(\varepsilon_{eff})$, to estimate the increase in flow stress caused by the superimposed fluid pressure in the Al-matrix composite, then a plot of:

$$\Sigma_{eff,P=0.1MPa}(\varepsilon_{eff}) + \Delta \Sigma_{eff}^{EMC}(\varepsilon_{eff})$$

(2)
should superimpose on the measured MMC flow curve under $P = 200$ MPa, 

$\Sigma_{\text{eff}, P=200 \text{MPa}}^{\text{MMC}}(E_{\text{eff}})$. Curves given by Eq. (2) are plotted in Fig. 6 by taking $\Sigma_{\text{eff}, P=0.1 \text{MPa}}^{\text{MMC}}(E_{\text{eff}})$ equal to the average of measured composite stress-strain curves for all samples of each composite material that were tested at atmospheric pressure. As seen, for the two higher volume fraction composites, curves predicted by Eq. (2) significantly overpredict the measured MMC flow curve.

Now, it is unlikely that the frictional contact law at the interparticle contact points is significantly altered in the metal matrix composite compared to the epoxy matrix composite, or compared to the dry particle bed. There is indeed no chemical interaction between $\text{Al}_2\text{O}_3$ and Al at the infiltration pressure used, nor is there any sintering or solution/reprecipitation of the alumina respectively before or after infiltration under conditions used in the present work. Moreover, the fact that aluminium has a contact angle with alumina that exceeds $90^\circ$ implies that there should not be metal atoms at particle-to-particle contact points, leaving the nanoscopic point of actual ceramic contact unaltered. It is therefore likely that the reason why there is less energy dissipation in the metal-matrix than in the polymer-matrix composites is purely mechanical: the matrix must shield part of the applied hydrostatic pressure from being transmitted to the chain of interparticle contact points, in turn reducing the amount of frictional energy that is dissipated within the particle bed upon composite deformation. To quantify the effect we draw an analogy between the composites and densely packed granular materials saturated with a pressurized fluid.

In “poromechanics of fluid-saturated media”, as the field is often called, the role of a pore-filling fluid pressure on the deformation and strength of granular media (often unconsolidated rock, soil or sand) is taken into account by introducing an “effective stress law” which reduces the constitutive response of the porous elastic solid, saturated with a pressurized fluid.
and subjected to a given applied stress, to that of the same granular material subjected to an “effective” stress. The most general formulation of the effective stress \( \langle \sigma_{ij} \rangle \) is given by:

\[
\langle \sigma_{ij} \rangle = \Sigma_{ij} - \alpha p_f \delta_{ij}
\]

(3)

where \( \Sigma_{ij} \) is the applied stress on the granular solid-fluid composite, \( p_f \) the fluid pressure (the latter stresses are taken positive in compression) and \( \alpha \) is a constant. In earlier literature, expressions for \( \alpha \) were empirically proposed, while only a few attempts were made to derive exact formulations. Initial seminal contributions, which include those of Terzaghi in 1923 [62] and a series of papers by Biot published starting in the early 1940s [63]-[65], have been completed by several other important contributions, both theoretical and experimental [66]-[68]. Terzaghi [62] first introduced the concept of effective stress to rationalize observations of time-dependent consolidation and failure of wet clay soils and suggested that \( \alpha = 1 \) under the assumption that the fluid and the solid skeleton can be idealized as perfectly incompressible. In order to account for the constituents’ compressibilities, Biot and Willis proposed in 1957 the following expression for \( \alpha \) [65]:

\[
\alpha = 1 - \frac{K_{\text{dry}}}{K^{(s)}}
\]

(4)

where \( K_{\text{dry}} \) and \( K^{(s)} \) are the bulk moduli of the dry aggregate, and of the material making the solid grains (in the form of a dense, continuous, phase), respectively. Equation (4) was also formulated independently by Geertsma [69] and Skempton [70], on empirical grounds. Nur and Byerlee [68] and more recent theoretical studies based on classical homogenization schemes [71]-[73] have proven Eq. (4) to be theoretically exact. If Eq. (4) is substituted in Eq. (3), then the following expression for the effective stress is obtained:

\[
\langle \sigma_{ij} \rangle = \Sigma_{ij} - \left(1 - \frac{K_{\text{dry}}}{K^{(s)}}\right) p_f \delta_{ij}
\]

(5)

which reduces to Terzaghi’s proposal that \( \alpha = 1 \) when the compressibility of the dry aggregate is much greater than the intrinsic compressibility of the solid grains (\( K_{\text{dry}} \ll K^{(s)} \)).
Eq. (5) has been proven valid in describing macroscopic properties such as strength [67],[57], frictional resistance [57] and electrical conductivity [74] of fluid-saturated porous media.

Let us now assume that the constitutive law provided by Eq. (5) also applies to particulate composites with a solid (vs. a liquid) matrix. We make this assumption despite the obvious differences that exist between solid and liquid matrices: with a solid matrix, relative particle sliding or rotation become more difficult and elevated tensile stress can be transferred across the interface, potentially breaking the particles in tension or shear. Also, the basic matrix rheological law differs (plastic versus Newtonian); however, under hydrostatic compression, with a ductile matrix and at the limited strain levels explored here, one may legitimately anticipate that particle trajectories and contact histories will be relatively similar, and that assumptions and conclusions derived from homogenization theory [73]-[74] will apply also if the matrix is a ductile solid instead of a newtonian fluid.

With this assumption, the amount of applied hydrostatic stress shielded by the matrix can be quantified and the effect of particle-to-particle contacts on the increment in composite flow stress, $\Delta \Sigma_{eff}^{MMC}$, can be predicted. Indeed, applied hydrostatic pressure should then, for a composite strain increment $\delta \varepsilon_{eff}$, cause an additional dissipation of energy that is the same as that displayed by the same powder bed after the same deformation history as the composite, reduced by a factor RF equal to:

$$RF = \frac{\Sigma_h - \langle \sigma_h \rangle_m}{\Sigma_h}$$

where $\Sigma_h$ is the hydrostatic pressure applied on the composite, and $\langle \sigma_h \rangle_m$ is the average hydrostatic stress in the (continuous and dense) composite matrix. There is little plastic volumetric strain in the present composites; we therefore estimate the hydrostatic stress concentration factor in the matrix $b_h^{(m)}$.
using expressions given in the literature for the compressibility of dense particulate composites (see Refs. [75]-[76]). The composite flow stress at \( P = 200 \) MPa is then given by:

\[
\Sigma_{\text{eff, } P=200\text{MPa}}(\varepsilon) = \Sigma_{\text{eff, } P=0.1\text{MPa}}(\varepsilon) + (1 - b_h^{(m)}) \Delta \Sigma_{\text{eff}}^{Al_2O_3\text{bed}}(\varepsilon) \quad (8)
\]

where \( \Delta \Sigma_{\text{eff}}^{Al_2O_3\text{bed}}(\varepsilon) \) is the flow stress increase in the (matrix-free) packed Al_2O_3 particle bed. To compute \( b_h^{(m)} \) we use two models: (i) the Hashin-Shtrikman upper bound (HS+) [77] and (ii) the self-consistent (SC) approximation [78]. The former coincides with a composite sphere model where the inner spherical inclusion is made of the more compliant constituent, (here the matrix), while the stiffer ceramic reinforcement is the outer spherical shell. The latter assumes that the same spherical inclusion is surrounded by a homogeneous medium whose properties are the unknown effective properties of the composite. The effective composite bulk moduli estimated by the two models, i.e. \( K_e^{(HS+)} \) and \( K_e^{(SC)} \), are given by Eqs. (9) and (10) respectively:

\[
K_e^{(HS+)} = K_e^{(r)} + \frac{1 - V_e^{(r)}}{1 + \left[ V_e^{(r)} \frac{(K_e^{(m)} - K_e^{(r)})}{(K_e^{(r)} + K_e^{(m)})}\right]} (K_e^{(m)} - K_e^{(r)}) \quad \text{with } K_e^{(m)} = \frac{4}{3} G_e^{(r)}
\]

\[
\left\{ \begin{aligned}
\frac{1 - V_e^{(r)}}{K_e^{(SC)} - K_e^{(r)}} + \frac{V_e^{(r)}}{G_e^{(SC)} - G_e^{(r)}} & = \zeta^{(SC)} \\
1 - V_e^{(r)} & = \frac{3K_e^{(SC)}}{3K_e^{(SC)} + 4G_e^{(SC)}}
\end{aligned} \right. \quad \text{with } \zeta^{(SC)} = \frac{3K_e^{(SC)}}{3K_e^{(SC)} + 4G_e^{(SC)}}
\]

\[
\left\{ \begin{aligned}
\frac{V_e^{(r)}}{K_e^{(SC)} - K_e^{(m)}} + \frac{1 - V_e^{(r)}}{G_e^{(SC)} - G_e^{(m)}} & = \beta^{(SC)} \\
1 - V_e^{(r)} & = \frac{3K_e^{(SC)}}{3K_e^{(SC)} + 4G_e^{(SC)}}
\end{aligned} \right. \quad \text{with } \beta^{(SC)} = \frac{3 - \zeta^{(SC)}}{5}
\]

In Eqs. (9) and (10) superscripts \((m)\) and \((r)\) stand for matrix and reinforcement respectively, while subscript \((c)\) is for composite; \( G \) is the shear modulus. Corresponding values for the stress concentration factors in the matrix and the reinforcement, i.e. \( b_h^{(m)} \) and \( b_h^{(r)} \) respectively, are obtained by writing stress and strain in the composite as the average of mean stress and strain in the composite constituent phases, namely:
\[ 1 = (1 - V^{(r)}) \eta_h^{(m)} + V^{(r)} \eta_h^{(r)} \quad \text{and} \quad \frac{1}{K_c} = (1 - V^{(r)}) \frac{1}{K^{(m)}} \eta_h^{(m)} + V^{(r)} \frac{1}{K^{(r)}} \eta_h^{(r)} \] (11)

Unlike the SC approximation, which yields an implicit expression of the effective bulk modulus \( K_c^{(SC)} \) that depends upon the effective shear modulus \( G_c^{(SC)} \), the Hashin-Shtrikman upper bound allows the derivation of an explicit expression for \( \eta_h^{(m)} \). The latter is given by Eq. (12) and is obtained by simple manipulation of Eq. (11) with substitution of Eq. (9):

\[ \eta_h^{(m,HS+)} = \frac{K^{(m)}}{(1 - V^{(r)})K^{(m)} + V^{(r)}K^{(r)}} \frac{(K^{(m)} + K^{(w)})}{(K^{(r)} + K^{(w)})} \] (12)

For the epoxy matrix composites, using both models, the values of \( \eta_h^{(m)} \) are very small, Table 3; hence, given the low flow stress of the epoxy (the epoxy tensile strength at room temperature is given by the manufacturer as 1-1.2 MPa) the polymer matrix composite flow stress increase under \( P = 200 \) MPa can be taken to be an approximation of the flow stress increase in the (matrix-free) packed Al\(_2\)O\(_3\) particle bed: \( \Delta \Sigma_{e_{\text{eff}}}^{\text{Al}_{2}\text{O}_3\text{bed}} (\varepsilon_{\text{eff}}) \approx \Delta \Sigma_{e_{\text{eff}}}^{\text{EMC}} (\varepsilon_{\text{eff}}) \).

<table>
<thead>
<tr>
<th>Hydrostatic stress concentration factor in the matrix, ( \eta_h^{(m)} )</th>
<th>Hashin - Shtrikman upper bound, Eq. (12)</th>
<th>Self-consistent approximation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-A61</td>
<td>0.6159</td>
<td>0.6544</td>
</tr>
<tr>
<td>E-A61</td>
<td>0.0095</td>
<td>0.0248</td>
</tr>
<tr>
<td>Al-P62</td>
<td>0.6124</td>
<td>0.6501</td>
</tr>
<tr>
<td>E-P62</td>
<td>0.0094</td>
<td>0.0232</td>
</tr>
<tr>
<td>Al-P56</td>
<td>0.6342</td>
<td>0.6766</td>
</tr>
<tr>
<td>E-P56</td>
<td>0.0103</td>
<td>0.0381</td>
</tr>
</tbody>
</table>

Table 3. Hydrostatic stress concentration factor in the matrix of Al-Al\(_2\)O\(_3\) and Epoxy-Al\(_2\)O\(_3\) composites estimated by means of the (i) Hashin-Shtrikman upper bound (Eq. 12) and (ii) the self-consistent approximation. Alumina and aluminium mechanical properties used for calculations are taken from Ref. [76], while the epoxy elastic constants are taken as \( E = 2 \) MPa (datum provided by the manufacturer) and \( \nu = 0.45 \).

Comparing now the experimental flow stress curves with theoretical predictions from the present simple analysis (using again the average of measured composite stress-strain curves
for all samples that were tested for each composite material at atmospheric pressure) shows that the agreement with data for the two composites containing \( \approx 61\% \) ceramic particles, Figs. 6a and 6b, is very satisfactory (in Fig. 6 curve colours at \( P = 200 \) MPa correspond to those in Fig. 3). For Composite Al-P56 containing 56.5 \% ceramic reinforcement, where the flow stress increment is far smaller, the agreement with data is clouded by the fact that experimental uncertainty is of the same order as the difference between the composite flow curves for the two models (Fig. 6c).

Our simple analysis thus explains the data. Note also how little the predicted composite flow stress increment varies with the mean-field approximation (Hashin-Shtrikman upper bound or self-consistent) used. This suggests that, in practice, the simpler Hashin-Shtrikman equation should suffice to predict the composite flow stress increment brought by applied pressure knowing the packed particle bed behaviour - even though it might not be a precise predictor of the actual composite compressive modulus.

5. Conclusion

We probe the mechanical response of high volume fraction (namely 56 - 62 vol. pct) ceramic particle reinforced metal matrix composites under triaxial axisymmetric compressive stress to investigate whether particle interlocking and mutual friction raise the composite flow stress stress when high superimposed fluid pressure squeezes particles against one another. To this end, we have constructed and calibrated a fluid-immersion triaxial testing apparatus capable of withstanding a maximum fluid pressure of 200 MPa and have used it to test composites produced by infiltrating closely-packed \( \text{Al}_2\text{O}_3 \) particle preforms with high-purity Al. Results show that hydrostatic pressure does enhance the flow stress of the composites after a few percent deformation and that the pressure-enhancement of composite flow stress increases (i) with the extent of prior composite deformation and (ii) with increasing vol.pct ceramic.
An additional finding is that when a composite of this class is deformed under $P = 200$ MPa and reloaded at atmospheric pressure, its flow stress superimposes precisely over that of the same composite deformed to the same strain entirely under atmospheric pressure. This, together with the measured effect of hydrostatic pressure on deforming composite dilation (for composites reinforced with the stronger polygonal particles), indicates that frictional energy dissipation between touching particles, and not internal damage accumulation, is the cause for the observed flow stress increment.

To link the flow properties of the composites with those of their packed particulate reinforcement, we have also tested composites that are similar to the infiltrated metal composites but have instead a matrix of epoxy. These show a greater flow stress increment, a fact that we explain using an analogy between the metal-infiltrated densely packed powder bed and a densely packed granular medium saturated with a pressurized fluid. The model shows good agreement with data, giving a simple predictive expression that quantifies the effect of superimposed pressure on the flow stress of composites reinforced with hard close-packed particles.

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7. References


under confining pressure: pore pressure tests.


Figure 1. Optical micrographs of the composites produced in this study and reinforced with 10 µm (a) polygonal and (b) angular Al₂O₃ reinforcements.

Figure 2. Schematic cross-sections of the testing apparatus: a) front view; b) side view. Index: 1) piston rod; 2) pressure vessel top case; 3) custom-designed seal; 4) specimen holder; 5) test specimen with strain gauges; 6) pressure vessel; 7) pressurized mineral oil; 8) pressure line inlet; 9) strain-gauge based load cell built on the piston; 10) high-pressure wire feed-through; 11) signals out to data logger.
Figure 3. Flow stress curves of Composites: a) Al-P62, b) Al-A61 and c) Al-P56 under axisymmetric compression at P= 200 MPa and P= 0.1 MPa.
Figure 4. Irreversible volume changes of Composites a) Al-P62, b) Al-A61 and c) Al-P56 under axisymmetric compression at $P = 200$ MPa and $P = 0.1$MPa. Note that positive volumetric strain in this graph denotes an increase in volume (dilatation).
Figure 5. Flow stress curves of Composites a) E-P62, b) E-A61 and c) E-P56 under axisymmetric compression at \( P = 200 \) MPa and \( P = 0.1 \) MPa.
Figure 6. Comparison between predicted and experimental flow stress curves of Composites a) Al-P62, b) Al-A61 and c) Al-P56 under axisymmetric compression at $P = 200$ MPa and $P = 0.1$ MPa.