



Full length article

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

M.G. Tarantino<sup>\*</sup>, L. Weber, A. Mortensen

Laboratory of Mechanical Metallurgy, Institute of Materials, École Polytechnique Fédérale de Lausanne, EPFL Station 12, CH-1015 Lausanne, Switzerland

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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 (56–62 vol pct) particulate  $\text{Al}_2\text{O}_3$ –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.

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## 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 bisectrix (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 bisectrix 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

<sup>\*</sup> Corresponding author. Laboratoire de Mécanique des Solides, C.N.R.S., École Polytechnique, University of Paris-Saclay, Route de Saclay, FR-91128 Palaiseau, France.

E-mail address: [tarantino@lms.polytechnique.fr](mailto:tarantino@lms.polytechnique.fr) (M.G. Tarantino).

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  $\text{Al}_2\text{O}_3$  particulates, both of average particle size near 10  $\mu\text{m}$ . 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 h. 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 h. It was checked by densitometry (using the epoxy density given by the datasheet, namely 1.1–1.2  $\text{g}/\text{cm}^3$ ) 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  $4.5 \times 7 \text{ mm}^2$  were machined from cylindrical composite ingots that result from the infiltration process. Al– $\text{Al}_2\text{O}_3$  and epoxy– $\text{Al}_2\text{O}_3$  samples were machined by electro-discharge machining (EDM) and by milling, respectively.

### 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<sup>®</sup> 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,

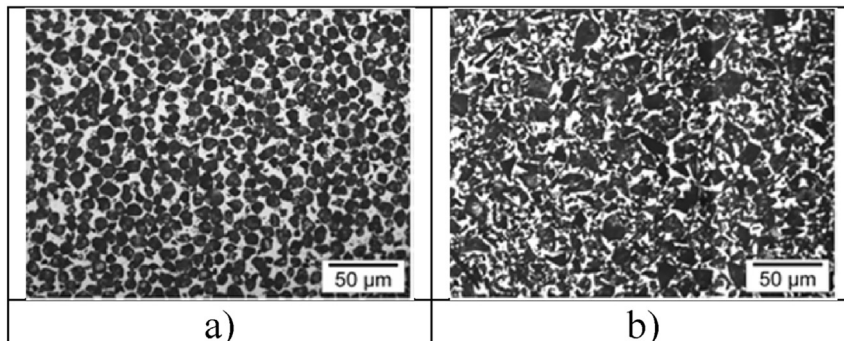


Fig. 1. Optical micrographs of the composites produced in this study and reinforced with 10  $\mu\text{m}$  (a) polygonal and (b) angular  $\text{Al}_2\text{O}_3$  reinforcements.

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