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In situ study of granular micromechanics in semi-solid carbon steels

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Abstract

The granular micromechanics of semi-solid steel at ~80% solid are studied by synchrotron radiography. A particulate soil mechanics approach to image analysis shows that deformation occurs by the translation and rotation of quasi-rigid grains under the action of contact forces, and that the changes in directional fabric and grain–grain contacts occur by mechanisms similar to those of highly compacted soils including "locked sands". Grain-scale phenomena are then linked to the macroscopic displacement and strain fields and it is shown that shear-induced dilation is a fundamental response at both the grain and macro scales. Based on this, recommendations are made on future rheology experiments.

Keywords: Casting; Solidification; Synchrotron radiation; Granular materials; Soil mechanics

1. Introduction

Casting defects including hot tearing, shrinkage porosity and macrosegregation result from flow and deformation of the solid–liquid mixture during solidification [1]. In all casting processes, loads and pressure gradients act on the semi-solid due to solidification shrinkage, thermal contraction and gravity, and in processes such as strip casting and semi-solid processing [2] deformation is applied externally by the process itself. For many decades, researchers have been building an understanding of semi-solid deformation with the aim of predicting and preventing casting defects and optimizing casting processes [1,3]. Research has shown that semi-solid deformation is complex, spanning from a Newtonian liquid through solid–liquid mixtures of increasing solid fraction to a polycrystalline viscoplastic solid.

It has been shown that semi-solid rheology is strongly dependent on the two-phase microstructure (solid fraction, morphology and degree of cohesion) and the loading conditions (mode, accumulated strain and shear rate) [1,4]. In the case of a globular morphology, semi-solid steels have been shown to be thixotropic [5], which has been attributed to the competition between agglomeration of globules with time and the break-down of agglomerates during shear [1,6]. A range of other deformation mechanisms are also known to occur including viscoplastic deformation of the solid [4,7,8], fragmentation of the solid [7,9] and "granular" phenomena associated with rearrangement of the solid as discrete bodies [9–13]. Furthermore, semi-solid deformation is not homogeneous at the micro-scale [12,14,15] and strain readily localizes [11]. Due to this complexity and multiple simultaneous mechanisms, in the last five years various groups have developed techniques to directly observe the deformation mechanisms in situ and link the crystal-scale mechanisms to the macroscopic semi-solid response [13–18].

In situ synchrotron radiography studies of semi-solid deformation in Al-alloys [13] and steels [18] at 30–60% solid have confirmed the importance of interpreting the material response within a granular mechanics framework. Granular materials are disordered assemblies of macroscopic

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particles in contact [19], and include soils, powders and cereal grain in silos. Under load, force is transmitted across the material via grain–grain contacts and a complex force chain network develops through the grain assembly. Consistent with this, radiography of equiaxed dendritic mush at ~30% solid and globular morphologies at 50–60% solid, deformed at $10^{-2}\ s^{-1}$, has shown that grains rearrange as quasi-rigid bodies within an assembly of grains in contact [13,18]. An important mechanical property in compacted granular materials is Reynolds' dilatancy [20], whereby the packing density of densely packed grains decreases during shear. This occurs when the grains are sufficiently compacted that they push and lever each other apart as they rearrange under load [20,21]. Recently, shear-induced dilation has been measured in macroscopic semi-solid deformation experiments on Al- and Mg-alloys [9,11,22] and crystals have been observed to push each other apart under load during synchrotron radiography of semi-solid Al- and Fe-alloys [13,18]. These combined observations suggest that semi-solid alloys deform with fundamental similarities to granular materials such as water-saturated soils under certain combinations of microstructure and loading mode.

This paper examines the micromechanics of semi-solid steel at 70–90% solid within a soil mechanics framework and seeks to build a fundamental understanding of how force is transmitted through partially solid alloys. Synchrotron radiography techniques from Ref. [18] were developed further to directly image the microstructural response to load of semi-solid carbon steels at 70–90% solid. The focus of this paper is on "fully-fed" high solid-fraction deformation in which liquid can flow into or out of the analysis region in response to any changes in solid packing density. After qualitatively analysing deformation in eight experiments, one sample that displayed behaviour representative of all "fully fed" samples at 70–90% solid was selected for a quantitative study of micromechanics in the sequence of radiographs. The approach quantifies the microstructural fabric, defined in soil mechanics as the arrangement of grains, grain groups and interstitial spaces [23]. This concept has proved useful in soil mechanics as two samples of the same granular material with similar solid fractions can exhibit a different mechanical response, depending on the grain orientations (e.g. [24]), and soil response is known to depend on the direction of loading relative to the particle and contact orientations (e.g. [25]). The analysis then focuses on (i) the evolution of grain–grain contacts during deformation, (ii) quantifying the displacement field, shear strain field and volumetric strain field, and (iii) comparing the overall response with soil behaviour.

2. Methods

2.1. Synchrotron radiography

Fe–2.08C–0.87Mn–0.45Si (mass%) high-carbon steel was produced by melting pure elements and TiN powder in a vacuum arc-melter. The Mn and Si contents were measured by inductively coupled plasma atomic emission spectroscopy and the C content was determined using high-frequency combustion infrared absorption. The as-cast equiaxed-dendritic microstructure was then coarsened and globularized by an extended semi-solid heat treatment of 100 h at 1250 °C (~70% solid) under Ar. This procedure produced an austenite grain size of ~350 μm and a non-dendritic morphology. Specimens with dimensions of 10 mm × 10 mm × ~180 μm thickness were cut and polished from this feedstock, to produce an approximate monolayer of austenite grains in liquid on reheating.

The time-resolved X-ray imaging of semi-solid carbon steel has only recently become possible and the challenges associated with high temperature (1150–1540 °C) imaging of a material consisting of γ-Fe and liquid phases with low absorption contrast are discussed in Ref. [26]. The experiments were performed on beamline BL20B2 at the SPring-8 synchrotron in Hyogo, Japan [27], building on techniques developed previously for steel [18,26]. A 23 keV X-ray beam was used and the transmitted beam was converted to visible light by a scintillator and recorded by a 1000 × 856 pixel CCD chip at 5 frames per second and with 4.9 μm pixel size.

In situ semi-solid deformation was conducted in the thin-sample shear cell shown in Fig. 1a, in which an Al_2O_3 push-plate penetrates into one half of the sample from below. Before an experiment, samples were partially remelted in the shear cell under a vacuum of 10^{-1} torr, and were held for ~10 min to thermally equilibrate the sample and cell. Isothermal deformation was then applied by a stepping motor with a displacement rate of 50 μm s^{-1}, corresponding to a global shear rate on the order of $10^{-3}\ s^{-1}$. These boundary conditions have similarities to the direct-shear-box test used in soil mechanics [28] and in studies on the rheology of semi-solid Al-alloys [29,30]. Direct-shear and penetration configurations have been widely used, due to their experimental simplicity, in the testing of soils (e.g. [31]), and both 2-D and 3-D models of the direct shear test have been studied using the discrete element method (DEM) [32,33]. The direct shear test is easier to carry out than a biaxial or triaxial test, but the stress and boundary conditions are more complex, complicating interpretation. However, it can capture dilatancy, stress-dependent stiffness and strength and other important characteristics of soil response, and therefore direct shear remains popular in both research and industry.

2.2. Image processing

Fig. 1c is an image of the selected semi-solid sample prior to deformation, produced by stitching six radiographs together. The dashed box marks the field of view during real-time imaging and the smaller dotted box marks the analysis region studied in this paper. The analysis region has a region of lower packing density above and above right of it, and there is a "reservoir" of liquid at the right-hand edge of the sample (arrow i in Fig. 1c) that