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The deformation of metal powder particles: hardness and microstructure

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Abstract

The modelling of powder densification requires knowledge about the material properties of the particles. Some models use the material properties of the material in bulk as inputs for modelling the densification of assemblies of powder particles. It is important to understand how these properties differ when the material is in powder form. Existing works tend to analyse bulk material or powder exclusively and rarely compare properties such as hardness between the two. This work compares the microstructures and hardness of metal powder with bulk material with the aim of guiding the assumptions made in the modelling of metal powder densification. It is expected that the rapid solidification typical in the atomisation of metals results in significant differences in behaviour when compared with material produced via casting and hot/cold working.

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Keywords: steel; powder; hardness; densification; deformation

1. Introduction

Powder metallurgy (PM) processes are used to make structural components when conventional processes are not able to meet the requirements. PM relies on the application of heat and/or pressure to transform loose powder into a product. This is called powder densification, as heat or pressure are applied, the space between the powder particles (pores) decreases and the component becomes more dense. It is necessary to understand how pressure, temperature and time relate to compact density in order to use these processes to the full potential.

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Part of densification occurs due to plastic yielding at the contacts between particles [1], which can be characterised by the material's stress-strain relation. The knowledge of these properties contributes to a more accurate prediction of how powder densifies under the effect of pressure and/or temperature. Over the past decades, a number of authors have modelled the densification of metal powders, either at room temperature [2,3], considering the effects of plastic deformation or at high temperatures, additionally considering creep deformation and particle boundary diffusion [4–7]. These authors obtain the physical properties of the powder by either performing experiments on assemblies of powder or on equivalent materials in bulk form. It is also possible to evaluate the mechanical properties of the powder particles directly. This work evaluates if this is advantageous when trying to predict powder densification.

The microstructure of metal powder often differs from the microstructure of the material in bulk. The rapid solidification typical of powder production processes originates materials with smaller grain sizes [8]. The deformation characteristics of powder differ from the material in bulk because the microstructure controls the mechanical behaviour of the material [9]. Ideally, densification models would use the mechanical properties of the actual powder. However these are difficult to measure, uni-axial tests cannot be applied to powder particles because these are typically smaller than 100 [μm]. The analysis of the microstructure and micro indentation tests provide ways to obtain deformation characteristics of powder particles.

In this work, the deformation properties of powders will be estimated from their microstructure and from micro indentation tests. These properties will be fed into the densification model proposed by [4]. The results of the model will be compared with the densification behaviour measured experimentally in hot pressing metal powder. This will help to verify if the knowledge of the deformation properties at the particle level is advantageous for the prediction of powder densification.

2. Methodology

The first step in the analysis was the selection of the material. The characteristics of the powder used in this work are described in table 1. 316L stainless steel was chosen because it is available both in powder and in bulk. There is also enough information on its material properties to be able to model its densification.

Table 1. Properties of the powder analysed in this work.

Alloy	Particle shape	Maximum particle size	Median particle size	Production method
Stainless steel (316L)	Spherical	106 [μm]	47 ± 2 [μm]	Nitrogen atomisation

The microstructures of the powder and the bulk material were analysed by optical micrography. Both samples were ground, polished and etched with Glicerigia. The powder particles were cold mounted in an acrylic mounting compound to provide support for microstructural analysis and the micro indentation.

The following equation was used to estimate the yield strength of both materials [11],

$$\sigma_y = 15 + \frac{33000}{T} + 65\frac{1690-T}{T}(\%N)^{0.5} + \big(7 + 78(\%N)\big)D^{-0.5} \quad (1)$$

where T is the temperature [K], (%N) is the mass percentage of nitrogen and D is the grain size [mm]. The grain size was measured using the line intercept method [10]. Only the cellular microstructures were considered.

Micro indentation tests were used to estimate the deformation properties of the powder. A pyramidal Vickers indenter was used to apply a maximum load of 98 [mN] which increased at a rate of 2.6 [mN/s] and was held constant for 10 [s]. The maximum load was chosen so the size of the indentation conforms to [12]. Both the load-depth relation and the indent diagonals were recorded. This information was used to estimate the material's yield strength according to [13],

$$H_v = 3Y \quad (2)$$

where H_v is the Vickers hardness and Y is the yield strength. This relation is only valid for materials that do not work harden during the hardness test. When this is not the case, Y has a value between the elastic limit of the

material and its tensile strength. The powder used in this paper was not mechanically worked after being atomised so it is likely to work harden as it is being indented. Despite not being an exact representation of the yield strength, the above equation still provides a useful indication of the material's deformation properties.

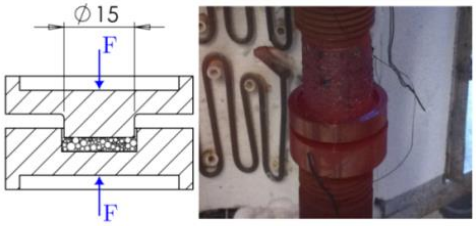

Fig. 1. Experimental setup used to compress the powder: (a) schematic representation (dimension in [mm]); (b) photograph of sample in the machine.

The model for powder densification proposed by [4] was implemented in Matlab in order to predict how the 316L powder densifies. Three hot pressing experiments were performed at 750 [℃] for one hour, the pressure was varied from 75 [MPa], to 100 [MPa] and 125 [MPa]. The experiments were performed using a universal testing machine with an environmental chamber. The experimental setup is represented in figure 1, the sample was heated at a rate of 15 – 25 [℃/min]. After the test, the sample was unloaded and cooled in air. As it was likely for powder to bond to the punch and die (figure 1a) these components were unique for each test.

After hot compression the samples were sectioned halfway through their diameter. This surface was then ground and polished for metallography. The density of the the samples was obtained by using the imageJ software to measure the area of the pores in the micrograph of the section of the sample.

The final step was the comparison of the experimental results with the results obtained with the densification model. The results are described and discussed in the following section.

3. Results and discussion

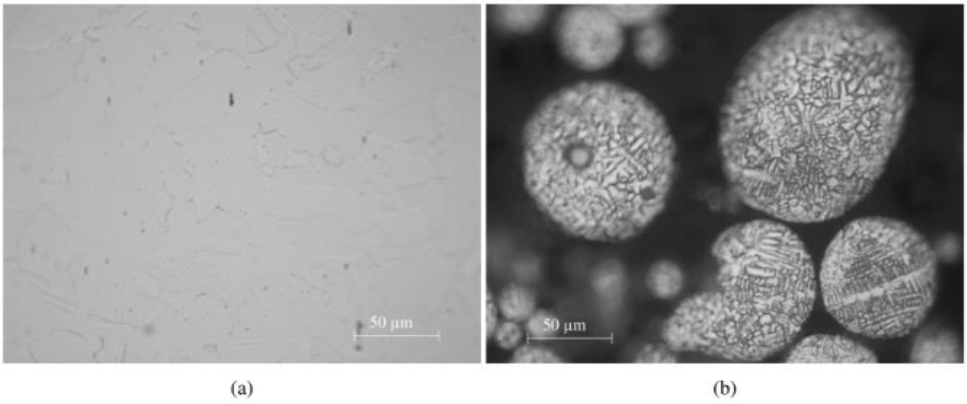

Fig. 2. Microstructure of 316L in the form of: (a) bulk; (b) powder.

The micrograph of figure 2a shows that the bulk material has a cellular microstructure. The microstructure of the powder is a mixture dendritic and cellular grains (figure 2b). The grain sizes of the powder and the bulk material are described in table 2. This table also shows the yield strength predicted by equation (1). This equation predicts that the yield strength of the powder is approximately 50% higher than the yield strength of the bulk material. Despite these values being obtained with an empirical relation they are a first indication of the differences in properties

between powder and bulk material. Micro indentation tests were performed in order to evaluate if this difference happens in practice.

Table 2. Relation between grain size and yield strength at room temperature for 316L stainless steel. The yield strength was obtained from equation (1).

	Grain size [μm]	Yield strength [MPa]
Bulk material	29	237
Powder	4	326

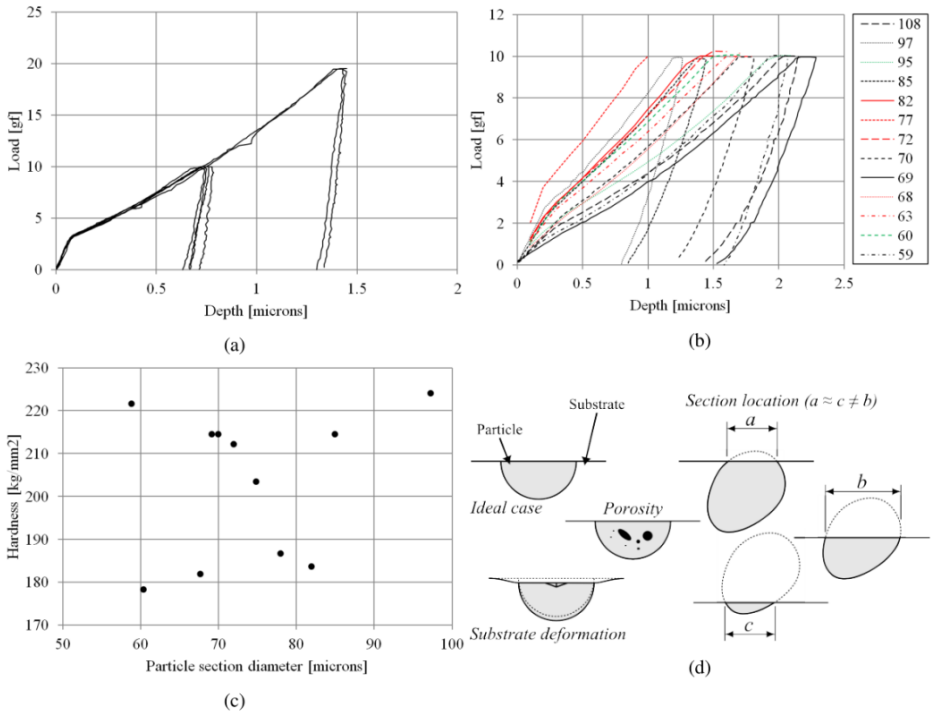

Fig. 3. Micro indentation of 316L stainless steel: (a) relation between load and indentation depth for the material in bulk; (b) relation between load and indentation depth for powder. The different colours represent the diameter of the section of the particle; (c) plot of Vickers hardness for powder; (d) schematic representation of sources of uncertainty in the measurement of the hardness of powder.

The results of the micro indentation tests are summarized in figure 3. The bulk material has a Vickers hardness of 208 ± 12 [kg/mm^2] and the powder particles have a hardness of 203 ± 16 [kg/mm^2]. Using those values in equation (2) gives a yield strength of 679 ± 38 [MPa] and 663 ± 54 [MPa], respectively. The fact that the materials have a similar hardness does not mean that they have the same deformation behaviour. As mentioned in section 2, the value of Y in equation (2) only represents the elastic limit of the material if it does not strain harden. [14] has studied the strain hardening of 316L and reported a strain hardening coefficient in the range of 0.3 to 0.4. The author also shows that the strain hardening coefficient decreases with the grain size. Since powder has smaller grains, its elastic limit could be higher than the bulk material. This assumes that Vickers indentation causes the same amount of plastic deformation for both materials. [13] mention that this value is in the region of 8%. The differences in mechanical behaviour between the powder can be further illustrated by their load depth curves during indentation, this is discussed in the following paragraph.

The comparison of figures 3a and 3b demonstrates that, for the same loads, the powder particles deformed at least 50% more than the bulk material. There is more variability in the load depth response of the powder. The unloading displacement of the powder is higher than the bulk material. This can be partially attributed to deformation of the mounting material which is less rigid than stainless steel. This deformation influences both the load depth relation and the value of the hardness but should be similar for particles of the same size.

There are a number of possible reasons for the high variability of the results from micro indentation of powder. It could be due to the fact that the particles may have varying mechanical properties. [8] have evaluated the relation between grain size and powder particle size of an austenitic stainless steel and concluded that the grain size decreases with particle size. Additional sources of uncertainty are schematically represented in figure 3d. The porosity of the particles can vary. This influences its deformation behaviour as there may be a varying amount of material to resist deformation during indentation. The hardness test is performed on a section cut of a particle, the visible diameter of the particle will depend on the position of the section cut in relation to the particle. As figure 3d shows: if $a \approx c$, the particle can appear to be the same size but the amount of material underneath could be different; if the visible diameter is equal to b, it is larger than c or a even though the particle could have the same geometry and be oriented in the same way.

Even though the micro indentation tests have not given an accurate representation of the deformation behaviour of metal particles these results are still useful to identify the main sources of uncertainty during these tests. The next paragraphs evaluate the impact of different deformation properties in the modelling of powder densification. A factor of two of difference in yield strength was used to assess its impact in the prediction of powder density.

Table 3. Relative density after powder hot pressing, experimental results and prediction from [4].

	Predicted density [%]		
Pressure [MPa]	Min.	Max.	Experimental density [%]
75	74	81	55 ± 4
100	76	84	89 ± 12
125	79	87	87 ± 15

The relative densities predicted by the densification model proposed by [4] are described in table 3. The minimum and maximum values correspond to assuming that the yield strength of the powder is 300 and 150 [MPa], respectively. These values were based on the microstructural examination and the indentation tests. This variation in yield strength results in a 8% difference in relative density when the highest pressure is applied. This difference reduces with the applied pressure.

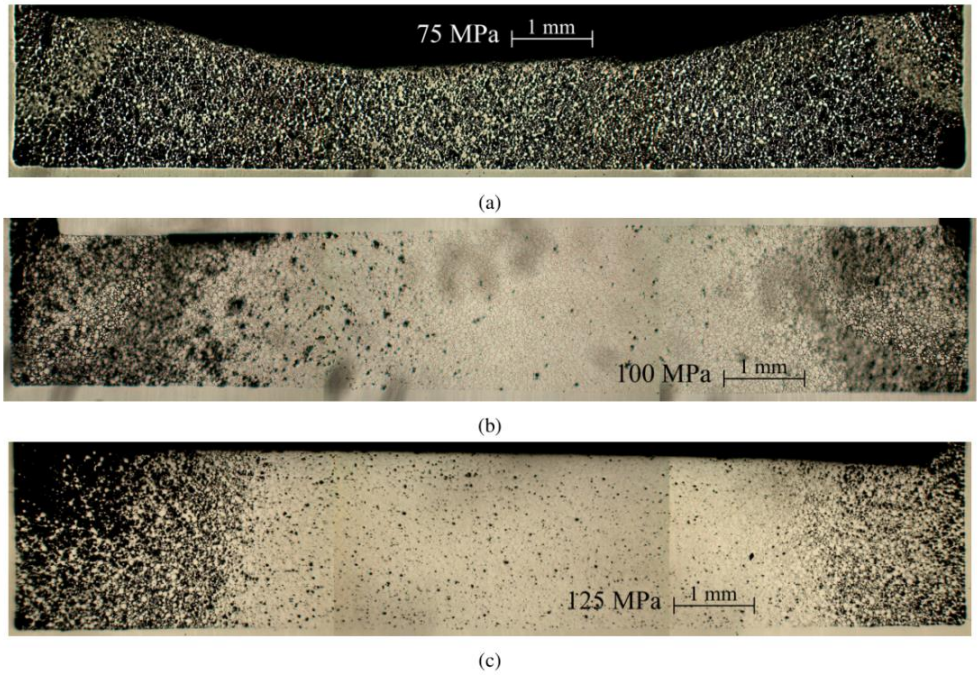

Fig. 4. Micrographs of the cross sections of the hot pressed samples at a pressure of: (a) 75 [MPa]; (b) 100 [MPa]; (c) 125 [MPa].

When powder is pressed at high temperatures it densifies due to plastic deformation, atomic diffusion and creep deformation. An increase in pressure causes more creep and plastic deformation so it is expected that a sample

compressed at a higher pressure has a higher relative density. Table 3 shows that this was not verified in practice and the relative density of the two samples compressed at the highest pressures is similar. One of the possible causes for this result is the fact that density is not uniform in the compact. Figure 4 shows the cross sections of the compacts after hot pressing. The black areas represent pores and the grey areas are the densified powder. The distribution of pores is not uniform across the section. This may have been caused by a non-uniform distribution of pressure under the punch. Furthermore the punch was not aligned with the die, this is visible in figure 4b, the solid material on the top of the image is not parallel to the solid material in the bottom (these are sections of the punch and the die, respectively). These factors mean that it is not possible to verify if there are advantages in evaluating the properties of the powder directly.

Conclusion and future work

This paper had two objectives: to verify if it is possible to obtain the deformation properties of the powder using microstructural analysis and micro indentation; to assess if this knowledge would help to predict how the powder densifies under high heat and pressure. The following paragraphs assess if the objectives have been reached.

The microstructures of the powder and bulk material differ significantly. The powder should have a higher yield strength, based on the grain size of the materials. This was not verified in the micro indentation tests which indicated that both materials have similar yield strengths. Even though the micro indentation tests have a limited accuracy some recommendations for future work can be made: the particles should be separated by small size ranges; the mounting material should be as rigid as possible and the powder particles could be indented unpolished.

According to the model proposed by [4] a difference in yield strength of a factor of two can cause a difference in predicted relative density of 8%. Hot powder compression tests were performed to verify this. The density of the compact varied significantly which prevented an accurate assessment of the validity of the model. However the tests were still useful to conclude that the pressure distribution under the punch is not constant and that alignment is crucial when studying density in hot powder compression.

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