Microscale Analysis of the Anisotropic Sintering of Metal Powder Compacts

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Abstract

The behaviour of steel powder compacts during sintering has been investigated by dilatometry and X-ray computed microtomography. Dilatometry measurements showed that the anisotropic deformation results from various phenomena arising at different moments of the cycle including the delubrication stage. Microtomography provided 3D images of the microstructure induced by prior die pressing and its changes throughout sintering. Finally a schematic description of the main phenomena responsible for the deformation of metal powder compacts during sintering is proposed.

Keywords : sintering, compacts, anisotropy, dilatometry, microtomography

1. Introduction

The sintering of metal powder compact obtained by die pressing is very different from the sintering of particle packing classically described in the literature. Since large interparticle contacts are formed during the pressing stage, the sintering process mainly consists in the formation of strong bonds at these contacts by diffusion mechanisms. The resulting macroscopic strains are very small, a few percents or even less, but it is crucial to control them, from industrial point of view in order to reduce or avoid expensive finishing operations.

To progress in the understanding and modelling of the sintering of steel powder compacts, a thorough analysis of anisotropic strains all along a sintering cycle has been coupled with 3D observation of microstructural changes in the course of sintering by *in situ* X-ray microtomography.

2. Materials and Equipment

The material used in this study is Distaloy AE Densmix^{TM}, a powder mixture designed for warm pressing. It is composed of iron particles with prediffused elements (1.5 wt.% Copper, 0.5 wt.% Molybdenum and 4 wt.% Nickel), mixed with 0.5 wt.% of graphite and 0.6 wt.% of lubricant. Particle size lies between 45 and 150 µm. This powder was heated to 130°C and pressed into a die previously heated to 150°C. Two green densities have been selected, 7.0 and 7.3 g/cm^3, corresponding to compaction pressures of 540 and 700 MPa, respectively. These densities will be referred to as D^- and D^+, respectively. The compacts are thus delubricated and sintered following the thermal cycle showed in Fig. 1. The shrinkage after sintering is anisotropic, around 0.2% in compaction direction and less than 0.1% in transverse directions for both D^+ and D^-, due to the anisotropic microstructure induced by prior die pressing. Also it has been found that the compacts exhibit swelling after dewaxing, which is more pronounced for D^+ compacts in axial direction (about 0.2 %).

The microstructure of Distaloy compacts has been observed by X-ray absorption microtomography at the European Synchrotron Research Facilities (ESRF). This non-destructive technique consists in recording digital radiographs of a specimen in several angular positions and next reconstructing with a mathematical algorithm the spatial distribution of the absorption coefficient within the specimen. For each data set 900 radiographies are recorded at different angles covering an interval of 180 degrees. From the radiographies, the reconstruction procedure provides an image of 1024x1024x255 voxels. Each voxel is a cube of 1.57 µm side with an effective absorption coefficient. Details about the experimental set-up and the data processing achieved to obtain 3D images can be found elsewhere [1, 2].

3. Results

Dilatometry curves recorded during delubrication and sintering cycle (Fig. 1) showed that the final deformation of Distaloy compacts results from various phenomena arising at different moments of the cycle: flowing out of the lubricant, iron phase transformation, copper melting, sintering mechanisms [2]. Each of these phenomena leads to a specific anisotropic effect that consists in either more swelling or more shrinkage, in either axial or transverse direction. The anisotropy in deformation measured after the complete cycle results from the accumulation of all these effects and is thus difficult to anticipate.

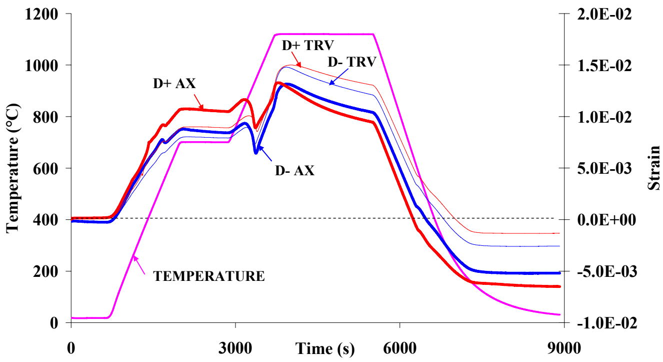

Fig. 1. Sintering cycle and dilatometry curves of steel Distaloy compacts with green densities 7 (D^+) and 7.3 g/cm^3 (D^-) in compaction (AX) and transverse (TRV) directions during sintering.

Fig. 2 shows three sections of the same D+ specimen, which have been extracted from 3D images taken at three different moments of the sintering cycle (green, completely dewaxed, completely sintered) during an in situ analysis. In the green compact it appears that most visible interfaces are perpendicular to the direction of compaction. These interfaces seem to slightly open during delubrication and disappear during sintering. This could be a reason for the swelling and shrinkage of compacts in the pressing direction of respectively after delubrication and sintering. These assumptions have been assed by quantitative image analysis [2].

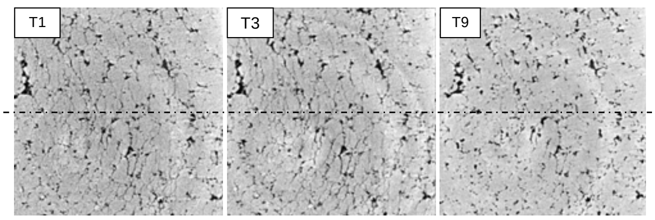

Fig. 2. Virtual sections of a Distaloy compact extracted from 3D images recorded during in situ experiment, respectively, in the green state (T1) after dewaxing (T3) and after sintering (T9). The direction of compaction is the dotted line. Dimensions are 780µm x 780µm.

4. Discussion

From this set of information a description of the main phenomena responsible for the deformation of steel powder compacts during sintering is proposed, which is schematized in Fig. 3. Two categories of pores are present in the green compacts:

- cusped pores that originate from the initial particle packing and have shrunk during compaction,
- elongated pores at contact interfaces that have been created during the unloading and the ejection of the compact out of the die and are mostly perpendicular to the axial (pressing) direction.

During delubrication, the effort of the lubricant to flow out of the material results in an opening of contact pores, which forms the easiest escape route, and thus in a swelling in axial direction, above all for a dense compact. At higher temperature, when the sintering starts, these contact pores get closed rapidly. This leads to a significant shrinkage in axial direction, and next classical sintering mechanisms shrink and round the packing pores. The shrinkage gets then more and more isotropic.

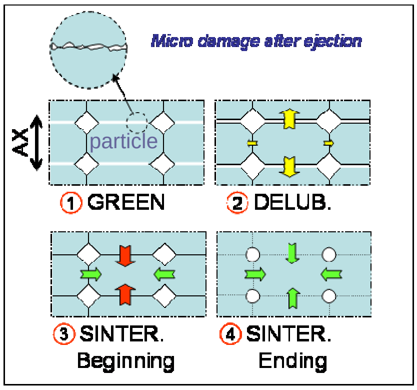

Fig. 3. Schematics of sintering of steel powder compacts

5. Acknowledgements

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6. References

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