Introduction

Synchrotron radiation X-ray microtomography has been used to investigate the sintering process of metallic powders. It has been shown that fast microtomography at high X-ray energy had spatial and temporal resolutions well adapted to investigate in-situ and in a non destructive way the sintering process of metallic powders at a pertinent scale [1, 2]. The principal aims of the experiments were:
1) to characterise microstructural changes and deformation of sintering powder at particle scale,
2) to investigate the origin of the anisotropic sintering of powder compacts,
3) to analyse the influence of defects (inert inclusions, macropores, etc.) on the densification.
For these purposes, two kinds of materials have been studied: a pure copper powder contained in a capillary which served as a modelled material, and compacts obtained by pressing an industrial steel powder (Distaloy AE) used for fabricating automotive components. Note that these experiments provided the first ever published 3D images of metal powders in the course of sintering.

Results obtained on Copper powder

Figure 1 exhibits 4 virtual slices perpendicular to the cylindrical axis at different times during a typical isothermal sintering at 1050°C. One can clearly observe the drastic effects of sintering: progressive neck formation and densification. The spatial resolution is high enough to exhibit relevant information.

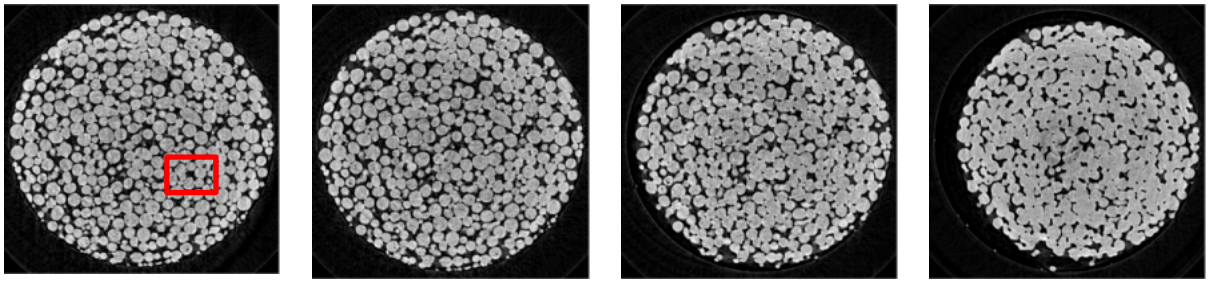

Figure1: Virtual slices of copper powder sample at different sintering stages (from left to right: image before sintering, and during the sintering process: t=0, 1h10' and 2h40').

Quantitative 3D image analysis allows following the changes of local neighbourhood configurations during sintering in the bulk of the material. Particle segmentation has been necessary to analyse particle and neck size distributions and particle co-ordination numbers and neighbourhood. Figure 2 shows a set of extracted particles at the beginning of sintering (see left) and after sintering (right), which clearly illustrates show interparticle neck growth. The whole analysis has been performed for each micro-tomographies taken during the sintering process.

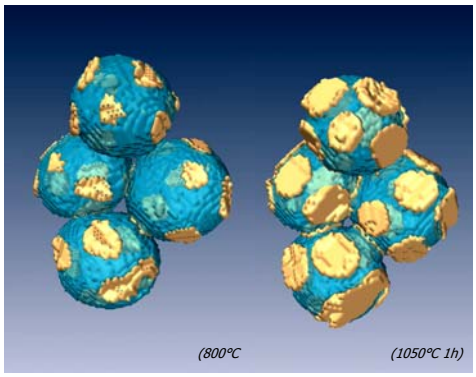

Figure 2: 3D rendering of copper particles with interparticle necks shown in yellow.

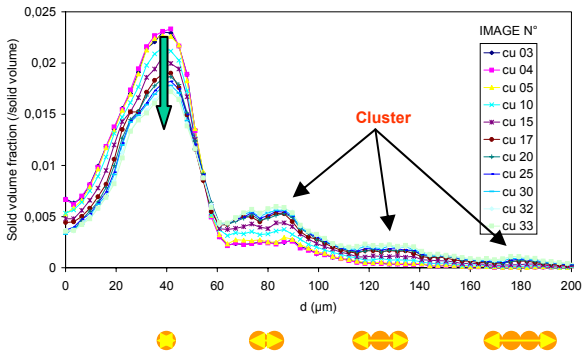

Figure 3: Intercept length distributions in the solid phase for the different micro-tomographies during the sintering process.

A quantitative analysis of 3D microstructural changes is reported in [3,4]. For instance, the variation of the intercept length distribution in the solid phase (Fig. 3) is characteristic of cluster formation: the distributions exhibit a major peak around 45 µm for all scans, corresponding to intercepts through a unique particle, while secondary peaks grow as sintering proceeds, corresponding to intercepts through two or more particles. The second peak position, which is exactly twice the position of the first peak at

the beginning of sintering (90 µm), is shifted about 4% to lower values at the end of the experiment, due to the sintering shrinkage. Other data showed that the sintering of copper powders was quite homogeneous and thus could be fairly well modelled with mean field assumption. Also it has been found that the interparticle co-ordination number evolved as predicted by classical Arzt's relation and neck growth was roughly in accordance with Kuczynski's model.

Results obtained on Distaloy compacts

Distaloy powder is a more complex material that contains an organic lubricant (wax) removed at the beginning of the sintering cycle. It is compacted to a relative density of 0.9 and then sintered. The deformation during sintering is very low, of the order of 1%, but very anisotropic. Microtomography tests allow understanding the origin of this anisotropy. As an example Figure 4 shows three sections of the same 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.

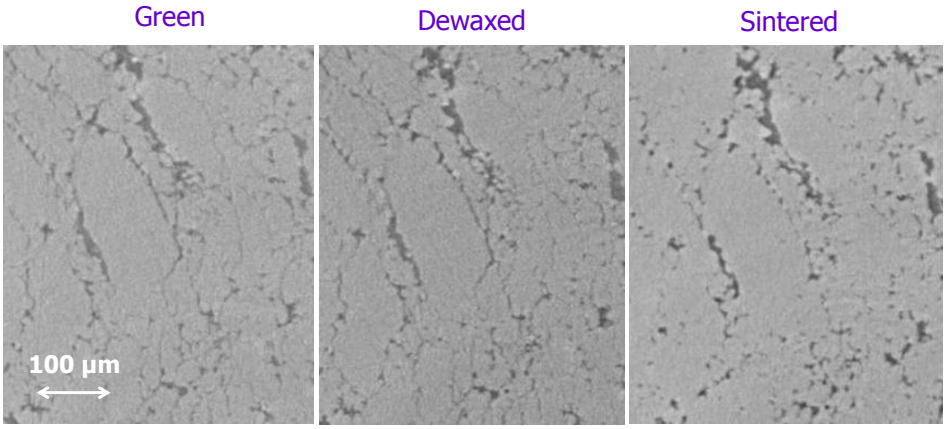

Figure 4 : Virtual slices of a Distaloy compact specimen extracted from 3D images obtained during in situ analysis. The direction of compaction is horizontal.

Original tools have been developed for a quantitative investigation of 3D images [5]. First, after a segmentation operation leading to binary images with separated porosity and solid phase, the changes in morphology and orientation of pores in 2D sections have been tracked. Secondly, local strains have been measured by correlating successive grey level images. From qualitative and quantitative information a description of the phenomena responsible for the deformation of steel powder compacts during sintering has been proposed. Two categories of pores have been found 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 pressing direction.

During dewaxing, 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. 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.

Conclusion

Fast microtomography at high X-ray energy appeared to be a very efficient tool to investigate in-situ and in the bulk the evolution of metallic powder during sintering at particle scale, above all when it is associated with 3D quantitative image analysis. This experimental method already provided precious information that has improved our knowledge of the sintering process and could not have been obtained by conventional methods. Most interesting results concerned particle clustering, interparticle neck growing and coordination, compact shrinkage anisotropy. This last issue, which is very critical from the applications point if view, will be investigated more deeply in the next experiments. Examples of other problems that could benefit from such 3D imaging are the transition from open to close porosity, the influence of defects (inert inclusions, macropores, etc.) on the densification, the role of particle rearrangement during liquid phase sintering.

The obtained data are also useful as a support for modelling. For example copper powder microtomographies have been sent to researchers of Sandia National Laboratories who simulated sintering with a Monte Carlo method. First comparison between simulation and experiments are very encouraging [6]. In a close future the same data will be used to validate discrete element modelling achieved in Grenoble [7] and at Fraunhofer Institute in Freiburg.

References

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