Introduction

Synchrotron radiation X-ray microtomography is used to investigate the sintering process of metallic powders. While the analysis of the data is still under progress, this work has already given rise to publications (1-2). Fast microtomography at high X-ray energy appears now to have spatial and temporal resolutions well adapted to investigate in-situ and in a non destructive way the sintering process of metallic powders. The principal aims of the experiments are: 1) to characterise the kinetics of the sintering process by in-situ imaging, 2) to investigate which is the origin of the anisotropic sintering of industrial alloys such as distaloy material (steel), and 3) to analyse the influence of defects (inert inclusions, macropores, etc.) on the densification. For these purposes, two kinds of materials have been studied: pure copper powder contained in a capillary which serves as a modelled material, and an industrial compacted alloy (distaloy) used as automotive components.

Results obtained on Copper powder

Figure 1 shows a 3D rendering of the copper powder contained in a 0.6 mm diameter quartz capillary. A part of this volume was virtually removed to have access to the interior of the specimen. The sample was only slightly sintered, but one can already observe some interparticle necks. Indeed the spatial resolution is high enough to exhibit the relevant information.

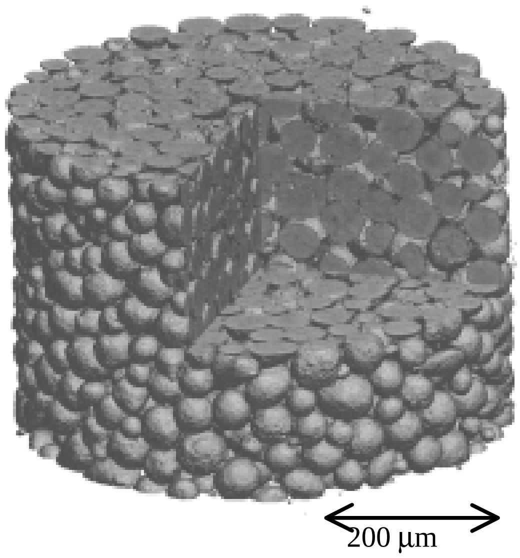

Figure 1

3D rendering of copper particles powder contained in a 0.6 mm diameter capillary.

Figure 2 exhibits four virtual slices perpendicular to the cylindrical axis at different times during sintering at 1050°C. One can clearly observe the drastic effects of the sintering: progressive neck formation and densification.

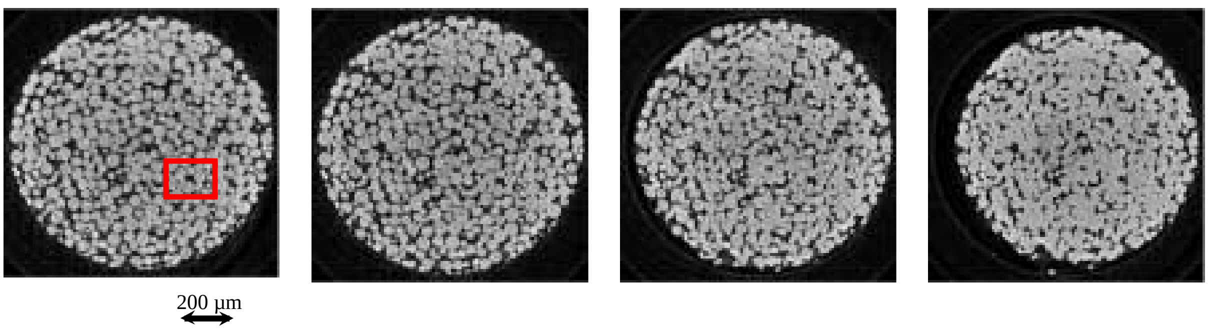

Figure 2

Virtual slices of the sample shown on Figure 1 at different sintering stages (from left to right: image before sintering, and during the sintering process: t=0, 1h10' and 2 h 40').

The data analysis allows stating the following trends related to copper sintering:

1) during the very first moments of the sintering, before strong necks have been formed, particle rearrangement is observed (even during one microtomography, movements of particles are observed);
2) the shrinkage is not at all uniform in the whole sample: while the average shrinkage is about 6.4%, local shrinkage up to 10% was observed between contacting particles. Such data, important to predict the non homogeneous mechanical properties of the specimen, could not be obtained with any other experimental method;

3) preliminary experiments showed that microtomography is also well suited to investigate the chemical changes for powder mixtures or alloyed powders.

Results obtained on distaloy samples

The distaloy material is more complex since this is an industrial steel alloy and the compaction prior to sintering leads to a dense material with low porosity. Anyway, interesting results were obtained. The goal of this investigation was mainly to identify the origin of the anisotropic shrinkage of such material. A very interesting clue can be extracted form the comparison of microtomography before and after the sintering as shown by Figure 3. After compaction the non sintered distaloy compact exhibits numerous interfaces mainly perpendicular to the compaction axis. Such interfaces disappear during the sintering process as shown by Figure 3. The data analysis will reveal when and how such interfaces disappear.

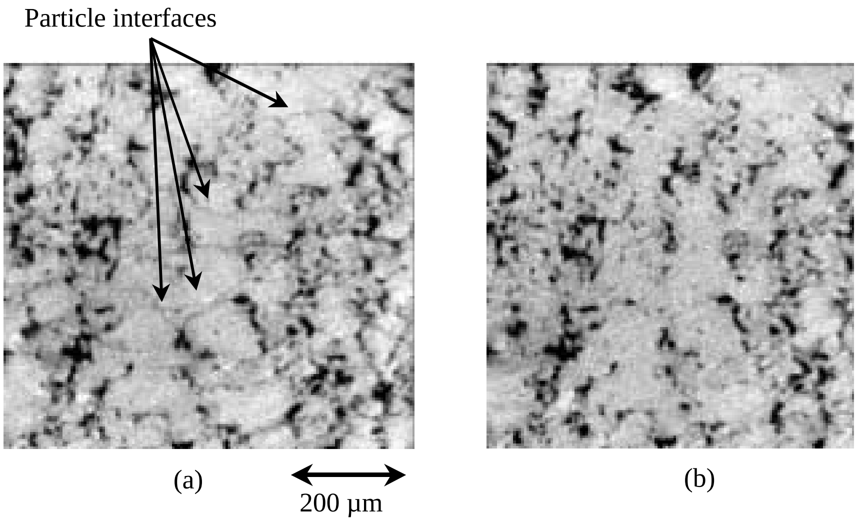

Figure 3 - Micro-tomography images of a Distaloy powder compact before (a) and after (b) sintering. The direction of compaction corresponds to the vertical direction in the plane of the sheet.

Conclusion

The first experiments on metallic powder sintering using fast microtomography show that this method will improve our knowledge of the sintering process. Indeed, interesting results were already obtained on the particles rearrangements, interparticle neck growing and particles interfaces.

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 sintering of powder mixtures including liquid phase sintering. The data, which is still under analysis (since rather sophisticated 3D software are necessary), will without any doubts draw original information relevant for testing existing models and developing more realistic models.

References

1. O. Lame, D. Bellet, . Di Michiel, D. Bouvard, *Nuclear Instrument and Methods in Physics Research B* **200**, 287 (2003)
2. O. Lame, D. Bellet, . Di Michiel, D. Bouvard, submitted to Acta. Materialia.