In-situ X-ray nano-tomography analysis of ceramic powder sintering

Aatreya Manjulagiri VENKATESH

Supervisors: Didier Bouvard, Pierre Lhuissier et Julie Villanova

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Jury :

- Elisabeth DJURADO, PROFESSEUR DES UNIVERSITES Grenoble INP	Examinateur
-Sabine ROLLAND DU ROSCOAT, PROFESSEUR DES UNIVERSITES UGA	Examinateur
-Fumihiro WAKAI, PROFESSEUR EMERITE Tokyo Institute of Technology	Examinateur
-François VALDIVIESO, PROFESSEUR DES UNIVERSITES Mines de Saint-Etienne	Rapporteur
-Dominique BERNARD, DIRECTEUR DE RECHERCHE EMERITE CNRS	Rapporteur

Abstract: Ultra-high resolution capabilities at the nano-imaging beamline of the upgraded ESRF synchrotron facility have now made investigation of ceramic powders at the individual particle length-scale feasible. These features were taken advantage of to investigate and enhance the understanding of sintering in two ceramic powder systems. Several 3D images with a voxel size as low as 25nm were obtained at different times of the thermal cycles for each powder system.

The first materials under consideration comprised two micron-sized alumina powders. The resolution attained allowed the depiction of particles and pores with enough details for subsequent quantitative analyses. Post-mortem analyses were initially carried out on these alumina powders sintered at 1500°C for various time periods. The phase-contrast nano-holotomography technique employed at the ESRF beamline allowed us to include large volumes of interest and examine various stages of sintering.

Furthermore, to monitor the evolution of sintering in real-time, nano-tomography experiments were attempted in-situ directly inside the synchrotron hutch. A compact high-temperature furnace was designed and fabricated for the same, providing us with images in the course of sintering at the nano-scale for the first time. Data resulting from quantitative image analyses were used to explore both densification and grain growth phenomena throughout the sintering cycle. Several sintering phenomena contributing to the collective behaviour of the particles were accurately observed at the local scale. These analyses included the grain size and shape, the pore size, the particle co-ordination number, the inter-particle neck size and, notably, the pore curvature for tracking the stages of sintering.

The experimental results were also confronted with a discrete element simulation to further compare and validate key sintering parameters.

The second investigated material is a sub-micronic zinc oxide powder mixed with 20 vol.% of larger alumina inclusions. In this case of constrained sintering, the 3D images allowed following up of the evolution of defects induced by zinc oxide aggregates and by the alumina inclusions throughout sintering.