Application of Digital Image Analysis to Characterize the Microstructure of Reactive Materials

Rachel Ferebee, Rensselaer Polytechnic Institute, SURF 2010 Fellow
Advisors: Dr. Arun Gokhale & Dr. Naresh Thadhani; Mentors: Ashok Gurumurthy & Anthony Fredenburg

Introduction

High velocity granular compaction is a processing technique relevant to the pharmaceutical and specialty materials industries. It has also been shown that highly-exothermic chemical reactions can occur in reactive powder mixtures.[1] The Ni+Al system is of interest, due to its use as a structural energetic material, a role in which the dense compact provides structural strength as well as contributes to the reaction.

Numerical simulations act as a means to investigate varying parameters of a system without the time and cost associated with physical experiments. Current microstructure simulations and models are unrealistic, making simplistic assumptions about particle shapes and orientations. The approach used in this research imports real microstructures to generate models representative of the actual material system.[2]

Procedure

Ni and Al powders were mixed in a 1:1 molar ratio, dried in a vacuum oven, and blended using a V-blender. Pellets 0.5 inches in diameter, 2 mm in height, and 60%, 70%, and 80% theoretical density were pressed from this powder mixed with a 24 hour cure epoxy representing the voids in the pellet. The pellets were cut in half and hot mounted exposing the cross section surfaces in the z plane. The samples were hand polished for varying durations on 240, 400, 600, and 800 grit paper followed by 15µm, 9µm, 6µm, 3µm, and 1µm diamond suspensions and colloidal silica. Indents were made at the corners of the samples with a microhardness tester to serve as reference points while taking micrographs.

Montages were created by stitching together adjacent fields of view at 20x magnification. The optical microscope settings were adjusted to optimize the contrast between the Ni and Al particles. Digital image analysis was then performed on the montages. The cropped, resized images were segmented to focus on one particular phase at a time. The binary images were then subjected to Two-Point Correlation Function measurements, effectively converting the image into mathematical form.

Quantitative characterization of the Ni+Al system at various densities was performed by manual and computational measurements of the first order properties: volume fraction, surface area, and triple phase boundary length.

Results and Discussion

The sample posed a challenge with respect to metallography. As a result of standard polishing procedures, pullouts of Ni particles were observed. Hand polishing was performed in order to avoid the creation of extra porosity not representative of the sample's natural microstructure.

The manual measurements performed on the montages shown in Figures 1a, 1b, and 1c revealed volume fractions of Ni, Al, and voids consistent with the theoretical calculations as well as those obtained from the Two-Point Correlation Function performed on the segmented images depicted in Figures 2a, 2b, and 2c.

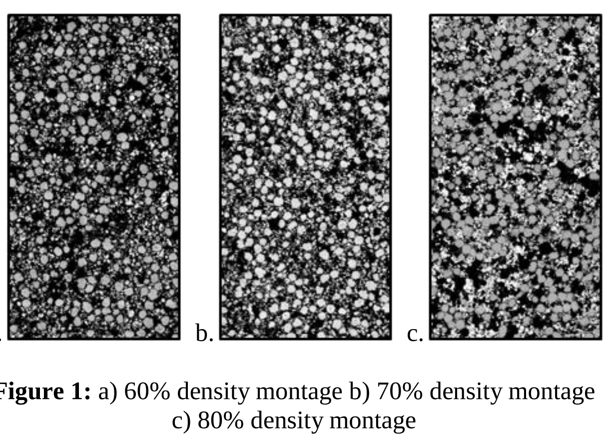

Figure 1: a) 60% density montage b) 70% density montage c) 80% density montage

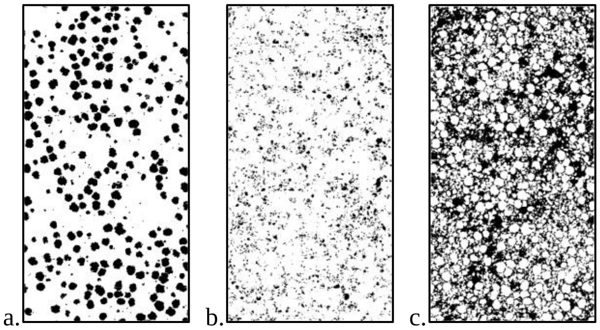

Figure 2: a) Segmented 60% with Ni in black b) Segmented 60% with Al in black c) Segmented 60% with voids in black

The surface area measurements led to mean intercept values for the Ni and Al particles of 46-62µm and 16-28µm, respectively, which were consistent with the expected particle sizes of 44-74µm for Ni and less than 44µm for Al.

The triple phase boundary lengths were measured by counting the number of times/area the Ni, Al, and void phases were in contact as a triple point. This two dimensional measurement is related to the length of the triple phase boundary in three dimensions extruded from the triple point in the z direction. A longer triple phase boundary contributes more to the reaction, and it was found that this length increased with density.

Summary and Conclusion

Three pellets of 60%, 70%, and 80% density of Ni+Al in a 1:1 molar ratio were fabricated to allow for advanced digital image analysis to be performed in an effort to import real microstructures for use in modeling and simulations of shock wave propagation experiments. The metallography was optimized to prevent particle pullout and to maximize the contrast between Ni and Al particles. Montages were created to provide a high resolution micrograph over a large surface area and were characterized quantitatively to determine the first order properties of the microstructure, including the volume fraction, the surface area, and the triple phase boundary length. A Two-Point Correlation Function was used to mathematically represent the microstructure, allowing for realistic modeling and simulations without relying on simplifications of the microstructure, such as the assumption of spherical particles.

Future work will involve serial sectioning of the samples to create a 3D representation of the microstructure through montages achieved after polishing away 1µm layers. The same digital image analysis will be applied to those montages, resulting in real imported microstructures as the input for modeling and simulations.

Acknowledgments

Special thanks to Dr. Gokhale and Dr. Thadhani for advising my research this summer and to graduate mentors Ashok Gurumurthy and Anthony Fredenburg who were tremendously helpful in the lab. Additional appreciation is extended to Dr. Milam, Shirley Manchester, and the NSF for facilitating the SURF Program.

References

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