Final Report of CRADA

Date: August 5, 2014

Principal Investigator: Iver E. Anderson

CRADA Agreement Number: AL-CRADA-2009-01

Partner Name(s): Carpenter Technology Corporation

Title: Development of Low Cost Gas Atomization of Precursor Powders for Simplified ODS Alloy Production

Results:
A novel gas atomization reaction synthesis (GARS) method was developed in this project to enable production (at our partner's facility) a precursor Ni-Cr-Y-Ti powder with a surface oxide and an internal rare earth (RE) containing intermetallic compound (IMC) phase. Consolidation and heat-treatment experiments were performed at Ames Lab to promote the exchange of oxygen from the surface oxide to the RE intermetallic to form nano-metric oxide dispersoids. Alloy selection was aided by an internal oxidation and serial grinding experiments at Ames Lab and found that Hf-containing alloys may form more stable dispersoids than Ti-containing alloy, i.e., the Hf-containing system exhibited five different oxide phases and two different intermetallics compared to the two oxide phases and one intermetallic in the Ti-containing alloys. Since the simpler Ti-containing system was less complex to characterize, and make observations on the effects of processing parameters, the Ti-containing system was selected by Ames Lab for experimental atomization trials at our partner. An internal oxidation model was developed at Ames Lab and used to predict the heat treatment times necessary for dispersoid formation as a function of powder size and temperature. A new high-pressure gas atomization (HPGA) nozzle was developed at Ames Lab with the aim of promoting fine powder production at scales similar to that of the high gas-flow and melt-flow of industrial atomizers. The atomization nozzle was characterized using schlieren imaging and aspiration pressure testing at Ames Lab to determine the optimum melt delivery tip geometry and atomization pressure to promote enhanced secondary atomization mechanisms. Six atomization trials were performed at our partner to investigate the effects of: gas atomization pressure and reactive gas concentration on the particle size distribution (PSD) and the oxygen content of the resulting powder. Also, the effect on the rapidly solidified microstructure (as a function of powder size) was investigated at Ames Lab as a function of reactive gas composition and bulk alloy composition. The results indicated that the pulsatile gas atomization mechanism and a significantly enhanced yield of fine powders reported in the literature for this type of process were not observed. Also it was determined that reactive gas may marginally improve the fine powder yield but further experiments are required. The oxygen content in the gas also did not have any detrimental effect on the microstructure (i.e. did not significantly reduce undercooling). On the contrary, the oxygen addition to the atomization gas may have mitigated some potent catalytic nucleation sites, but not enough to significantly alter the microstructure

vs. particle size relationship. Overall the downstream injection of oxygen was not found to significantly affect either the particle size distribution or undercooling (as inferred from microstructure and XRD observations) but injection further upstream, including in the gas atomization nozzle, remains to be investigated in later work.

Intellectual Property: No intellectual property was developed in this project.

Publications

J.L.L. Meyer, I.E. Anderson, FAPMI, and J.R. Rieken, "Novel Gas Atomization Approach for Nickel-Based Oxide Dispersion Strengthened (ODS) Powder Production," in *Advances in Powder Metallurgy and Particulate Materials-2012*, compiled by I. Donaldson, FAPMI, and N.T. Mares, (Metal Powder Industries Federation, Princeton, NJ), (2012) Part 2, pp. 132-146.

I.E. Anderson, J.R. Rieken, J. Meyer, D.J. Byrd, and A.J. Heidloff, "Visualization of Atomization Gas Flow and Melt Break-up Effects in Response to Nozzle Design Variations: Simulation and Practice," in *Advances in Powder Metallurgy and Particulate Materials-2011*, compiled by I.E. Anderson and T.W. Pelletiers, (Metal Powder Industries Federation, Princeton, NJ), (2011) Part 2, pp. 15-30.

J.L. Meyer, I.E. Anderson, J.R. Rieken, and D.J. Byrd, "Gas Atomization Precursor Powder Approach for Simplified Large-Scale Production of Oxide Dispersion Strengthened (ODS) Ni-Based Alloys," in *Advances in Powder Metallurgy and Particulate Materials-2011*, compiled by I.E. Anderson and T.W. Pelletiers, (Metal Powder Industries Federation, Princeton, NJ), (2011) Part 2, pp. 50-66.

I.E. Anderson, D. Byrd, and J.L Meyer, "Highly Tuned Gas Atomization for Controlled Preparation of Coarse Powder," MATWER, vol. 41, no. 7(2010), pp. 504-512.

J.L. Meyer, I.E. Anderson, J.R. Rieken, and D. Byrd, "Development of Large-Scale Gas Atomization of Precursor Powder for Simplified Production of Oxide Dispersion Strengthened (ODS) Ni-Based Alloys," in *Advances in Powder Metallurgy and Particulate Materials-2010*, compiled by M. Bulger and B. Stebick, (Metal Powder Industries Federation, Princeton, NJ), (2010) Part 2, pp. 112-131.