Specific Adaptation of Gas Atomization Processing for Al-Based Alloy Powder for Additive Manufacturing

The Ames Laboratory & Alcoa, Inc.

CRADA No. AL-C-2014-03

Investigators: Iver E. Anderson (Ames Laboratory)
John Siemon (Alcoa)

Period of Performance: August 1, 2015 – June 30, 2017 (with no-cost extension)

U.S. DEPARTMENT OF ENERGY

Ames Laboratory is a U.S. Department of Energy Laboratory managed by Iowa State University

Atomization Data Summary

The charge for each gas atomization experiment was provided by Alcoa and consisted of cast blocks cut into 1 inch by 1 inch square rods of the chosen aluminum alloys. The atmosphere in the melting chamber and connected atomization system was evacuated with a mechanical pump prior to backfilling with ultrahigh purity (UHP grade) Ar. The melt was contained in a bottom tapped alumina crucible with an alumina stopper rod to seal the exit while heating to a pouring temperature of 1000 – 1400°C. When the desired superheat was reached, the stopper rod was lifted and melt flowed through pour tube and was atomized with Ar from a 45-22-052-409 gas atomization nozzle (or atomization die), having a jet apex angle of 45 degrees with 22 cylindrical gas jets (each with diameter of 1.32 mm or 0.052 inches) arrayed around the axis of a 10.4 mm central bore. The Ar atomization gas supply regulator pressure was set to produce nozzle manifold pressures for the series of runs at pressures of 250-650 psi. Secondary gas halos of Ar+O_2 and He also were added to the interior of the spray chamber at various downstream locations for additional cooling of the atomized droplets, surface passivation, and to prevent coalescence of the resulting powder.

Powder Size Classification & Analysis

Powder size analysis and size classification were performed with a full set of ASTM sieves, starting with the 106 µm screen and proceeding down through the 20 µm screen. A Microtrac particle size analyzer with laser diffraction was used to verify the size classification results of the particulate resulting from the runs. The resulting powder size distribution results are provided with this report labeled with their respective run numbers. The size distribution results of the atomization runs were acceptable and atomization parameters were changed between runs and alloys in order to increase the percent yield of powder within the desired size range. The run parameters and results are included in the below table.

Chemical Analysis Results & Explosivity Testing

LECO analysis of the powders was performed for the initial laboratory scale run on a powder size range <45µm and showed average content of 800 ppm oxygen, 95 ppm carbon, 7 ppm nitrogen, and 3 ppm sulfur. These higher than usual numbers for the Ames Lab gas atomization process, especially for oxygen, are likely from the bulk casting charge materials. Auger electron spectroscopy analysis of representative powders showed an average oxide layer thickness of ~5nm. Explosivity testing, as performed by Chilworth Technology, Inc, deemed these powders to be not readily combustible substances.

Atomization Optimization

The initial three atomization attempts resulted in “freeze-outs” within the pour tubes in the pilot-scale system and yielded no powder. Re-evaluation of the alloy liquidus temperatures and melting characteristics, in collaboration with Alcoa, showed further superheat to be necessary to allow the liquid metal to flow through the pour tube to the atomization nozzle. A subsequent smaller run on the experimental atomization system verified these parameters and was successful, as were all successive runs on the larger pilot scale system. One alloy composition froze-out part way through the atomization on both pilot scale runs. SEM images showed needle formation and phase segregations within the microstructure. Analysis of the pour tube freeze-out microstructures showed that large needles formed within the pour tube during the atomization experiment, which eventually blocked the melt stream. Alcoa verified the needle formation in this alloy using theoretical modeling of phase solidification. Sufficient powder of this composition was still generated to allow powder characterization and additive manufacturing trials at Alcoa.

Run #	Pour Temp. (C)	Charge (kg)	% Yield	Gas/Metal Mass Ratio	Atm. Pressure (psi)	Pour rate (kg/min)	d50 (µm)
ASC-1-9	1100	15.4	NA	NA	250	NA	NA
ASC-1-11	1200	10.1	NA	NA	250	NA	NA
ASC-1-13	1050	15.0	NA	NA	300	NA	NA
GA-1-250	1100	2.5	30%	2.92	250	3.26	44
ASC-1-15	1000	16.5	87%	2.64	300	3.43	55
ASC-1-17	1400	16.8	62%	2.49	550	6.23	52
ASC-1-19	1400	13.0	66%	1.78	550	9.11	55
ASC-1-21	1380 (freeze out)	13.1	38%	4.59	650	4.92	48
ASC-1-23	1350 (freeze out)	13.2	29%	7.21	650	2.19	28
ASC-1-25	1350	13.6	NA	2.80	650	6.11	~45