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Characterization of Commercial Aluminum Powders for Use in Energetic Applications, Part II: Structural, Chemical, and Thermal Analyses

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14. ABSTRACT This work aims to study the structure, composition, and thermal behavior of several commercially acquired aluminum (Al) powders with varying sizes using transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and simultaneous thermogravimetric analysis and differential scanning calorimetry. Four samples of Al powders in the ranges of 40–60 nm, 60–80 nm, 100–130 nm, and less than 5 µm are specially examined by TEM and XPS to investigate their surface properties. Significant numbers of particles with their sizes outside the range of the given range are found for all samples. The sample labeled as “< 5 µm” in particular is essentially a mixture of nano- and micron-sized powders. All samples also contain significant crystalline alumina deposits that presumably are γ-Al ₂ O ₃ . X-ray photoelectron survey spectra revealed the significant carbon content for the 60–80-nm nanopowder sample. The results from thermal analyses suggest the active Al content of the nanopowders is as much as 15 wt% lower than that of the micron-sized powders.					
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1. Introduction and Background

As part of the ongoing development of energetic aluminum (Al) powders and optimization of the experimental approaches in functionalization toward maximized fast energy release and combustion effects, we conducted characterization of commercial Al powders of different sizes. Four samples of Al particles in the ranges of 40–60 nm, 60–80 nm, 100–130 nm, and less than 5 μm are of interest here. The reactivity of these samples has been reported in a previous study and compared via the laser-induced air shock from energetic materials (LASEM) method, spectroscopy, and time-resolved optical emission (Gottfried and Wainwright 2021). In that report, the time-to-peak for aluminum monoxide emission, temperature, and post-plasma combustion emission were found to decrease as the particle size decreases, suggesting higher reactivity for smaller Al particles at both the microsecond (μs) and millisecond (ms) timescales. The less-than 5- μm Al has the largest optical intensity (i.e., the strongest combustion) at the ms timescale.

In this report, we focus on characterization of the same materials with special focus on structural and thermo-oxidative properties via advanced electron microscopy (scanning electron microscopy [SEM] and transmission electron microscopy [TEM]), X-ray photoelectron spectroscopy (XPS), and simultaneous thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). For SEM and TEM, the goal is to examine the shape, qualitative size distribution, and morphology of the particles for each sample. The XPS survey spectra aim to examine any possible impurities. The purpose of thermal analyses is to first examine the exothermic-oxidation behavior for each sample and their correlation to the particle size and then compare their active Al contents.

The results documented in this report not only provide important additional information regarding the structural and thermal features of these materials, but also interpret the laboratory-scale energetic performance previously reported by Gottfried and Wainwright (2021). This work provides insights toward a complete description of the efficacy of these materials to be used as precursors in the development of novel reactives and energetics. In the interest of having a focused discussion on different aspects and meaningful cross-examinations among these samples, only key TEM data are reported herein. Further in-depth, nanoscale detailed TEM analysis will be reported in another technical report.

2. Experimental

2.1 Materials

Four Al powders of different particle sizes were characterized in this work. They were Sigma-Al powder with vendor-provided particle size no larger than 5 μm (herein labeled as “< 5- μm Al”) and three aluminum nanoparticle (nAl) samples purchased from SkySpring Nanomaterials, Inc. with vendor-provided particle sizes as 40–60, 60–80, and 100–130 nm. These three nAl samples are labeled as “40–60-nm Al”, “60–80-nm Al”, and “100–130-nm Al” in this report. Details of these Al powders have been described in a previous report (Gottfried and Wainwright 2021).

2.2 Electron Microscopy

SEM images were obtained using a Hitachi S4700 field-emission microscope operating at 30.0 kV. The powdered samples were sprinkled on a double-sided carbon tape placed on the stud. The excess powder was blown away with air. No coating was used.

TEM specimens were prepared via nanoparticle-suspension technique (Ayache et al. 2009). The standard procedures are as follows: an approximately 3-mL colloidal solution was prepared by dispersing a trace amount of the sample powders in acetone (VWR Analytical), followed by, at most, a 3-min gentle sonication (Super Scientific 100004 Ultrasonic Cleaner). A few drops of the colloidal solution were added onto a TEM grid (300 mesh, Ted Pella, Inc.) with the holey carbon support film side on top, and then the sample-containing grid was air dried before storage in the desiccator. All structural- and chemical-analysis data were obtained by using a JEOL JEM-2100F TEM operated at 200 kV (JEOL USA, Inc.). High-resolution bright-field images and relevant chemical analyses including X-ray energy-dispersive spectra (XEDS) were acquired using an Octane Elite T windowless detector with TEAM Analysis software (EDAX, Inc.). High-angle annular dark-field images at the scanning TEM (STEM) mode were obtained to attempt improved contrast for large Al particles for the “< 5- μm Al” sample. All TEM data were processed and analyzed using Gatan Digital Micrograph Suite software (Version 3.32.3403, Gatan Inc.).

2.3 XPS

Near-surface compositional measurements were performed using a Physical Electronics VersaProbe II Ultra XPS system equipped with a hemispherical analyzer and a take-off angle of 45°. The sample was irradiated by a focused beam of monochromatic Al K α X-rays (1,486.7 eV). The system's pressures are typically around 6.7×10^{-8} Pa when the instrument is idle without argon (Ar) flow and there is no sample in the main chamber. A survey scan was performed for each sample with the constant analyzer-energy mode with a passing energy of 93.5 eV. A low-energy neutralizer using both low-energy electrons and Ar ions was used to reduce surface charging, and the spectra were adjusted for any residual charging by adjusting the energy of the C1s peak corresponding to C-C to a binding energy of 285.0 eV. Surface compositions were calculated using relative sensitivity factors with the Casa XPS software.

2.4 Thermal Analysis

TGA/DSC were run in a TA Instruments SDT660 calorimeter using Trios software. Analyses were performed in an oxidizing environment (80% Ar + 20% oxygen [O₂]; 40 mL/min) with a heating rate of 10 °C/min. Samples were held in 90- μ m alumina (Al₂O₃) crucibles.

3. Results and Discussion

3.1 Characterization via SEM

SEM images of Al powders are given in Fig. 1. All are consistent with expectations based on information provided by the manufacturer, other than the Sigma Aldrich nanopowder in which significant numbers of μ m-sized particles were revealed (Fig. 1, top right).

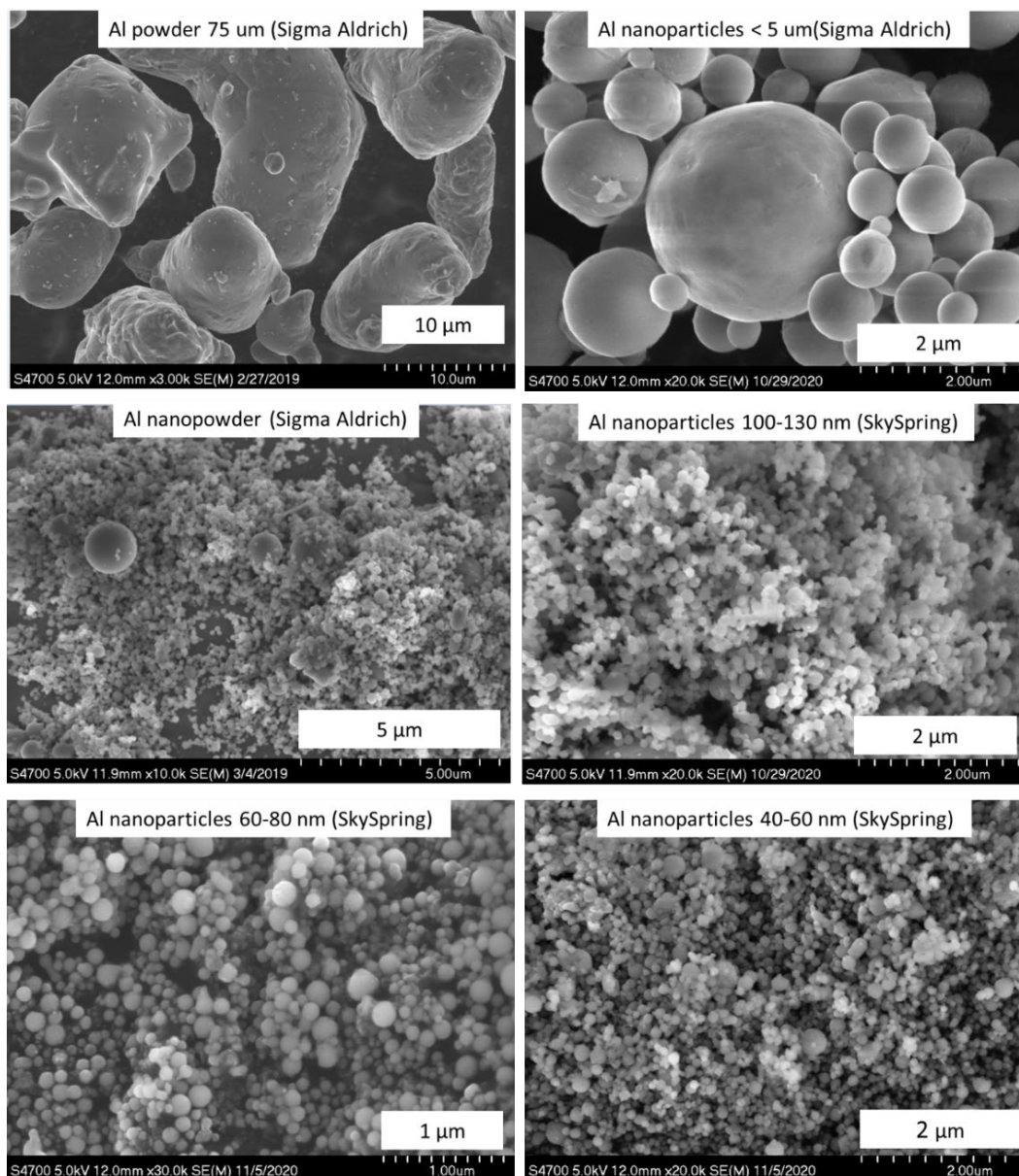


Fig. 1 SEM images of Al powders

3.2 Characterization via TEM

Herein we report the key structural and chemical properties of these materials obtained by TEM/STEM imaging and XEDS. Details of more in-depth nanoscale analyses will be described in a separate technical report.

Different from SEM images (Fig. 1), TEM imaging enables revelations of structural characteristics, in particular the crystallinity of materials and interfacial properties. In this work, we focus on the particles' shapes, sizes, core-shell structure, deposits, and crystallinity. As anticipated for most commercial Al powders, each sample exhibited a wide range of particle-size distribution. In particular, the < 5- μm Al

sample appeared to be a mixture of micron- and nano-sized particles varying from as small as 200 nm to as large as 2 μm . Figures 2 and 3 display two aggregates with individually measured particle sizes indicated by numbers (in the unit of nm). Figure 2 is an image showing a μm -sized oval Al particle surrounded by many nm-sized particles having a wide range of sizes, while Fig. 3 shows a collection of spherical μm - and nm-sized particles. Several deposits were also discovered as pointed by the black arrows in the figure but not in the focus condition. These TEM images of a $< 5\text{-}\mu\text{m}$ Al sample correspond well to the SEM image for the shape and size of particles (Fig. 1). Details of the surface morphology for the μm -sized particles cannot be revealed in TEM because of the extraordinarily large distance the electron beam must pass through, leading to significantly degraded spatial resolution due to the chromatic aberration effects (Williams and Carter 2009, ch. 20).

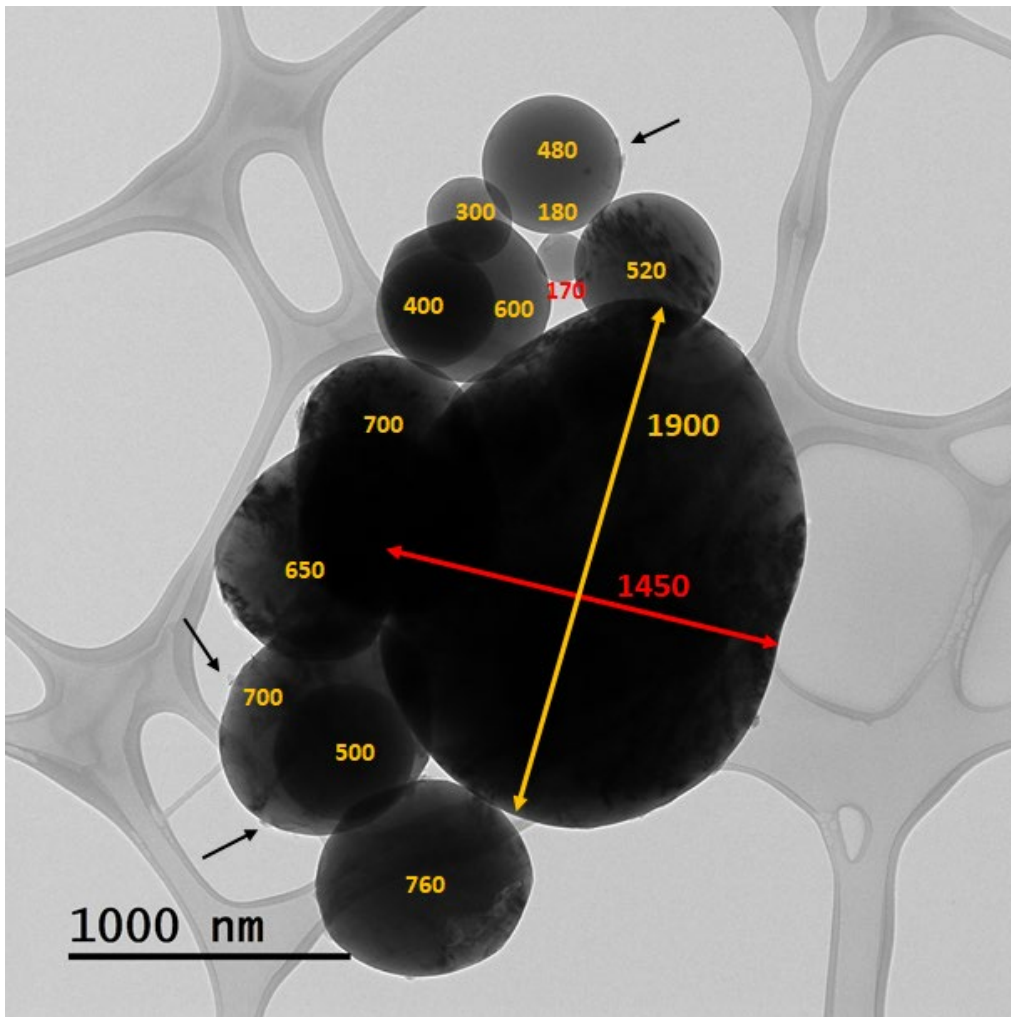


Fig. 2 TEM images of $< 5\text{-}\mu\text{m}$ Al sample showing μm -sized particle surrounded by nm-sized particles; numbers are in units of nanometer and arrows point to deposits

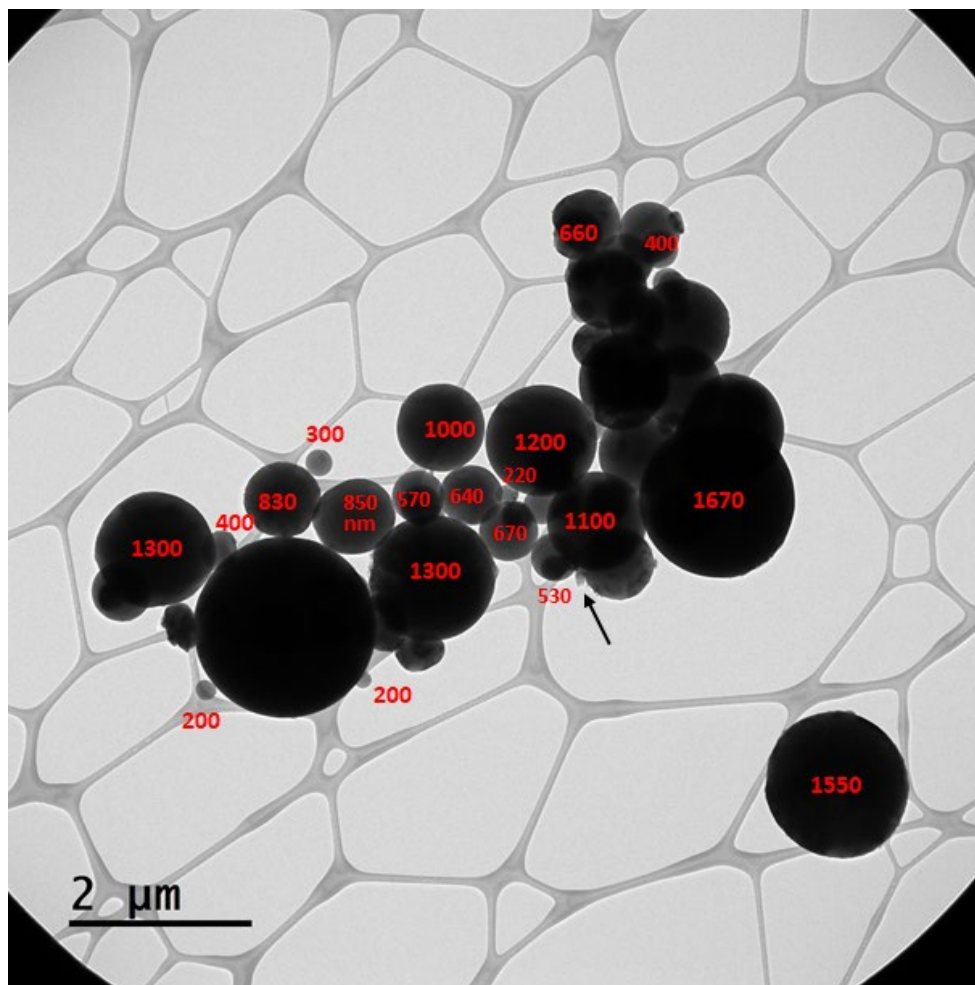


Fig. 3 TEM images of $< 5\text{-}\mu\text{m}$ Al sample showing a cluster of different particle sizes, 200 nm to almost $2\text{ }\mu\text{m}$; numbers are in units of nanometer and arrows point to deposits

Variations of the particle size for the nm-sized SkySpring samples (40–60-nm Al, 60–80-nm Al, and 100–130-nm Al) are not as significant as the Sigma-Aldrich $< 5\text{-}\mu\text{m}$ Al sample although they all show substantial numbers of exceptionally larger and smaller particles outside the nominal particle ranges provided by the vendor. Figures 4 and 5 display representative TEM images for these samples. As evident in Fig. 4, there are already many particles exceeding the 40–60-nm range only in this image for the 40–60-nm Al sample. The same trends are true for the 60–80-nm Al and 100–130-nm Al samples as well (Fig. 4). Thus, while particles for a μm -size labeled sample may statistically be larger than those for samples labeled in nm ranges, the collective effects due to small differences in the particle-size distribution may not be significant; for example, the several in the 10-nm range among our SkySpring samples examined here. Figures 4 and 5 essentially revealed indistinguishable size ranges among these samples. Some particles are also connected with each other.

Significant crystalline deposits, presumably γ -phase alumina (γ -Al₂O₃), were also discovered sporadically on particle surfaces and in most cases filled the interparticle spaces for all three samples. Representative deposits are specified by red arrows and dotted enclosures. Because these particles are spherical and randomly overlap with each other, it is not possible to have all constituents in the TEM field of view to be in excellent in-focus condition. Images shown in Fig. 4 were acquired with the majority of the constituents being in focus, including the deposits, although some of them were under- or over-focused as a result. In our previous studies of atmospheric plasma-treated and -coated nAl, similar γ -Al₂O₃ deposits were also found and hypothesized as the result of partial phase transformation from the amorphous Al₂O₃ shell in the plasma due to local heating (Wu et al. 2020a, 2020b, 2021). We suspect similar annealing phenomena may have also occurred in the synthesis and processing of commercial Al powders.

Moreover, a partially damaged particle was identified, as shown in Fig. 5 (top) and indicated by a black arrow for the 60–80-nm Al sample.

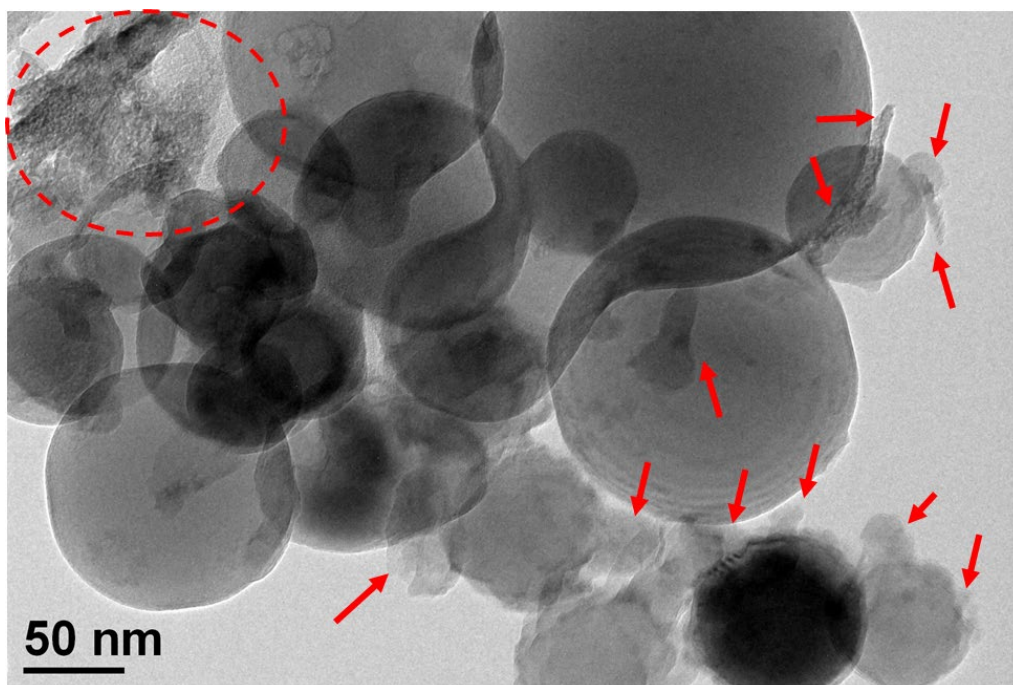


Fig. 4 TEM images of SkySpring nm-sized samples: 40–60-nm Al; red arrows indicate crystalline deposits, presumably γ -Al₂O₃

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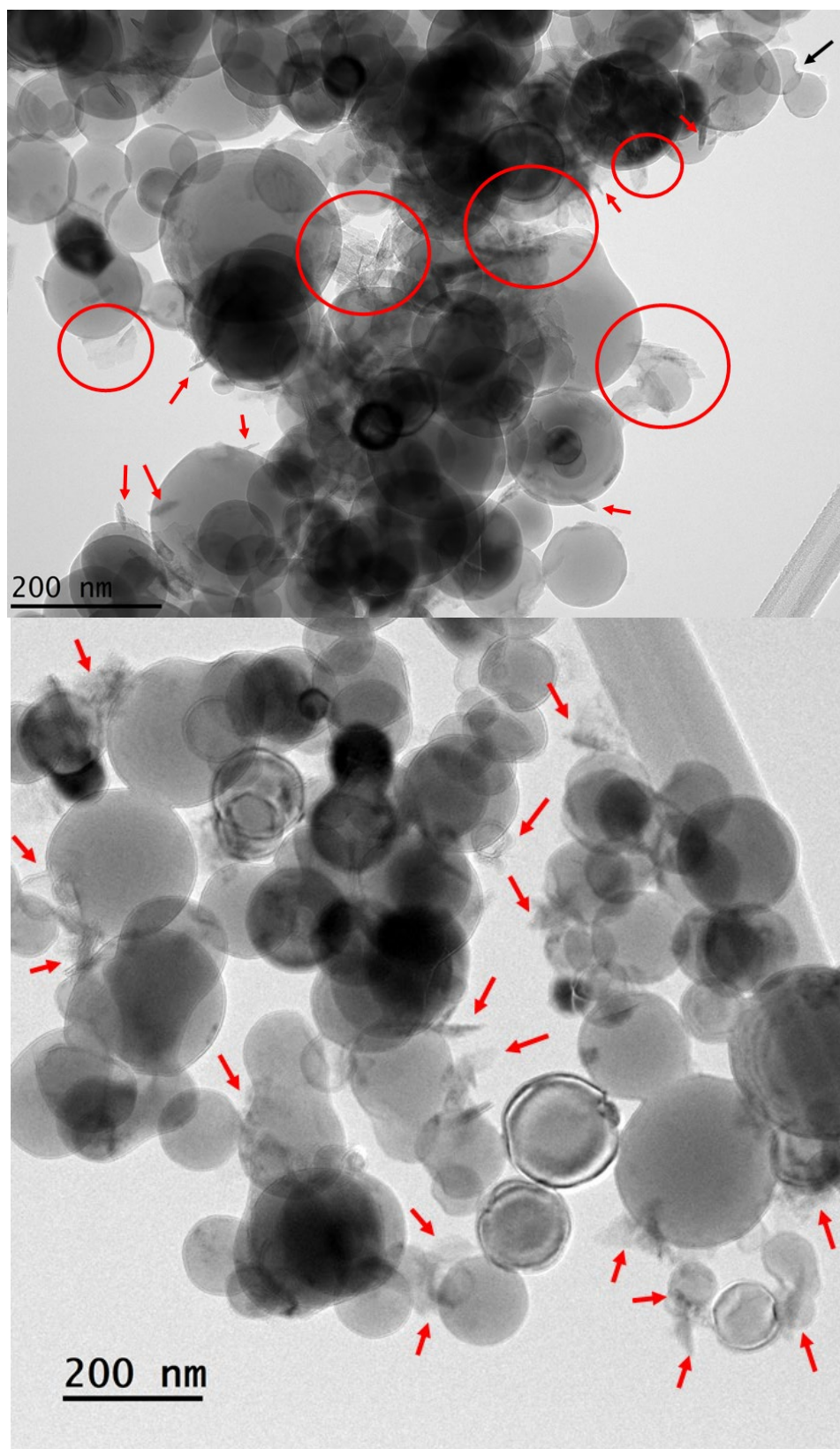


Fig. 5 TEM images of SkySpring nm-sized samples: (top) 60–80-nm Al and (bottom) 100–130-nm Al; black arrow in the top image specifies partially damaged particle and red arrows indicate crystalline deposits, presumably γ - Al_2O_3

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3.3 Characterization via XPS

Figure 6 displays the XPS survey spectra with the binding energy from 0 to 1300 eV. As anticipated, Al and oxygen (O) peaks were identified for all samples. By comparing the relative intensities among these samples, the SkySpring 60–80-nm sample had significantly higher content of surface carbon (C) with significantly reduced Al peaks. On the other hand, the 40–60-nm Al sample is “cleaner” with the lowest intensity of surface C peak along with distinct Al peaks. The Sigma-Aldrich < 5- μm Al sample had relatively higher C content as well. These findings are qualitatively consistent with the thermal behaviors (see Section 3.4) and the laboratory-scale energetic performance via LASEM (Gottfried and Wainwright 2021).

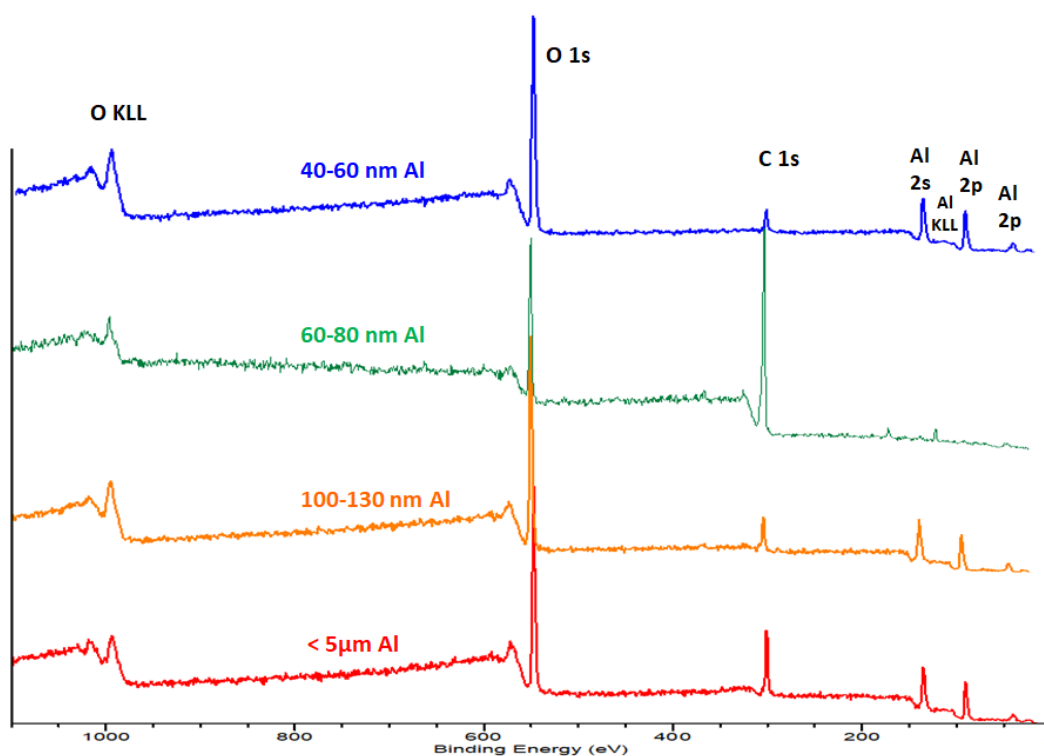


Fig. 6 XPS survey spectra of all samples

3.4 Characterization via Simultaneous Thermal Analysis

Traces for simultaneous thermal analysis via DSC/TGA for all but the < 75- μm Al powder are given in Fig. 7. Traces for the < 75- μm samples are given in Fig. 8; the irregular (but reproducible) DSC baseline made it impossible to determine an accurate enthalpy of oxidation. Onset temperatures for the first oxidation exotherm for nanoparticles generally occur near 600 °C and are very sharp. Additional exotherms near 800 and 1100 °C are much broader. Exotherms for the < 5- μm sample occur near 600 °C and at 800–1100 °C and are both broad.

Figure 9 summarizes the active Al content and enthalpy of oxidation for each sample in this study. The values may be compared with the laser-induced air shock velocities given in Fig. 10 (reproduced from Gottfried and Wainwright [2021]). It is observed that while the active Al content of the SkySpring nanopowders is as much as 15 wt% lower than that of the Sigma-Aldrich μm -sized powders, the observed LASEM velocity exceeds that of the larger particles, presumably because the nanopowders can oxidize so much faster than the micron-sized powders because of the collectively much-smaller particle sizes.

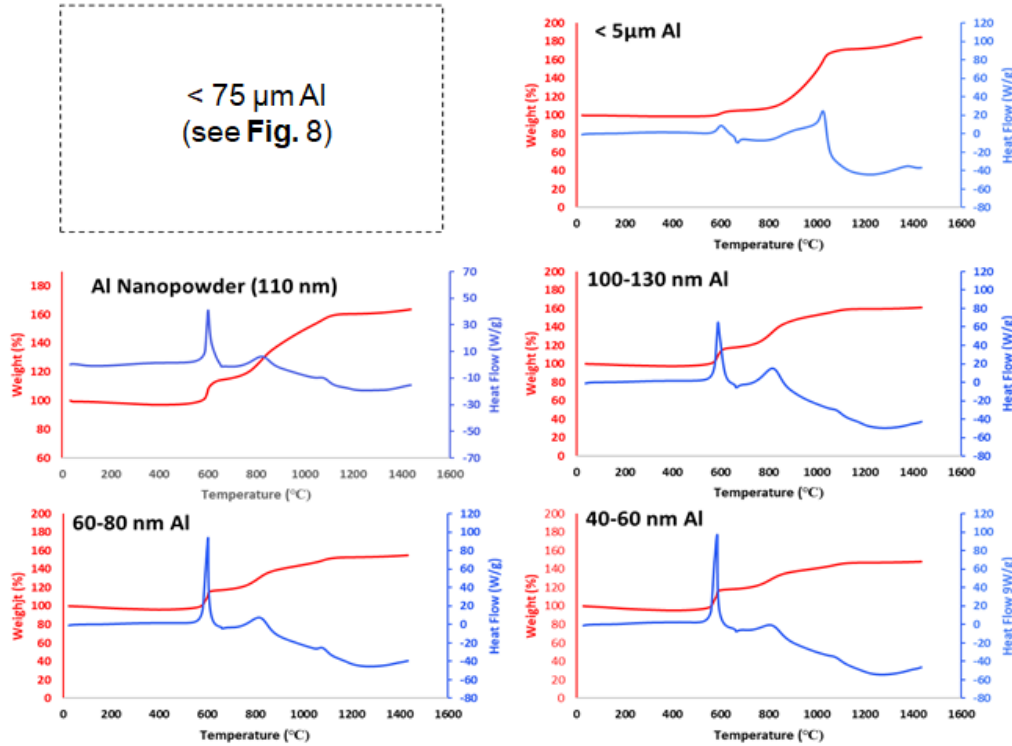


Fig. 7 Simultaneous DSC/TGA traces for Al samples as function of temperature

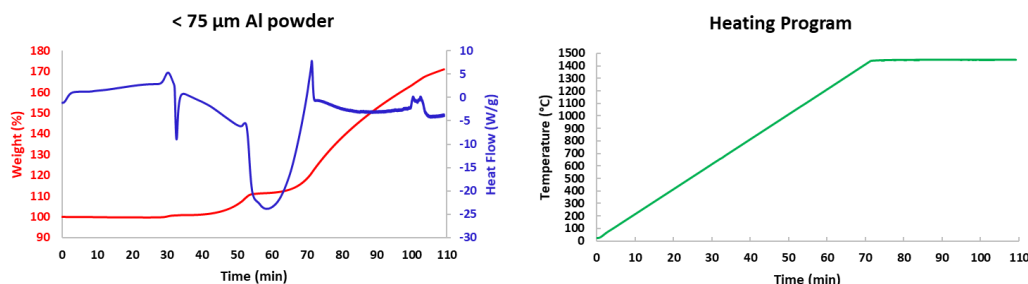


Fig. 8 Left, simultaneous DSC/TGA traces for < 75-µm Al sample as function of time; accurate determination of heat of oxidation from DSC trace was not possible due to baseline irregularities resulting from oxidation of very large Al particles; right, corresponding temperature profile for DSC/TGA data

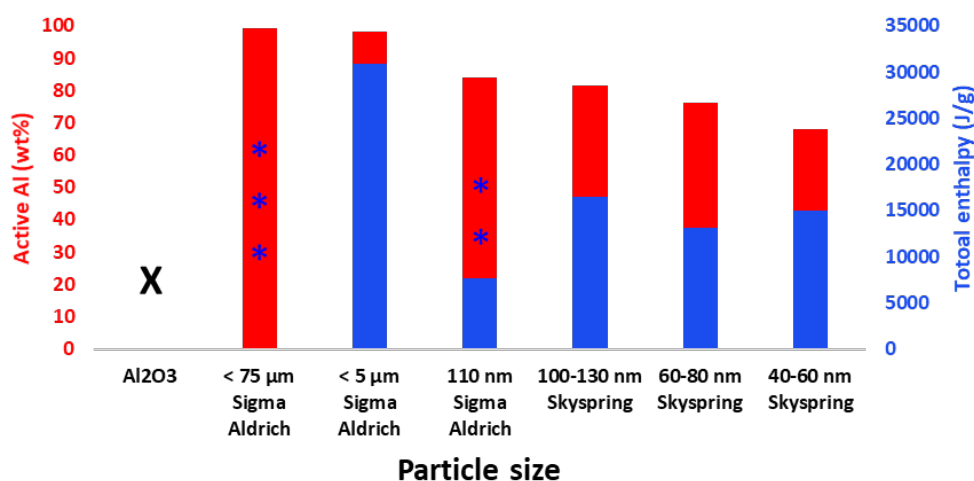


Fig. 9 Summary of active Al content and total enthalpy of oxidation for samples: asterisks for < 75-µm and 110-nm samples indicate accurate total enthalpy values not obtained; DSC trace with irregular baseline for 75-µm sample is in Figs. 4 and 5; nanopowder enthalpy is lower than expected, presumably from incomplete combustion of micron-size particles in powder (see Fig. 1)

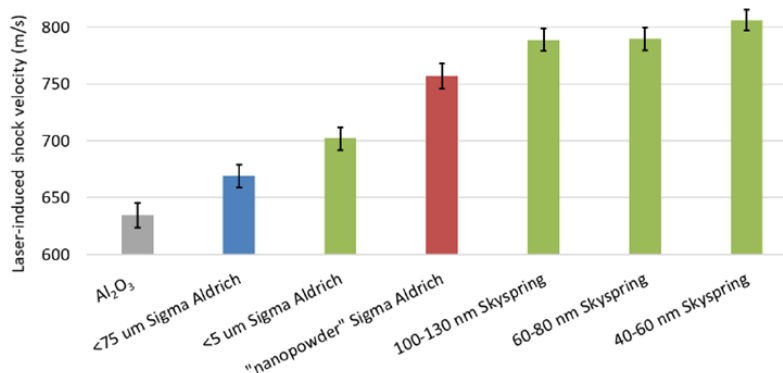


Fig. 10 Characteristic laser-induced shock velocities for four new Al samples (green); micron-sized Al₂O₃ (gray), < 75-µm-sized Al (blue), and Al "nanopowder" (red) previously characterized in lab shown for comparison (adapted from Fig. 2, Gottfried and Wainwright [2021])

4. Conclusions

This report describes our follow-up characterization tasks on commercial Al powders of particle sizes ranging from nanometer to micron. Samples of particle sizes comprising Sigma-Aldrich < 5- μm Al and SkySpring nanopowders, 40–60-nm Al, 60–80-nm Al, and 100–130-nm Al, were further characterized by TEM and XPS.

SEM images revealed the irregular geometry for the 10- μm Al sample, the high content of nm-sized particles for other μm -sized samples, and a variety of particle sizes for nm-sized samples. The findings were consistent with TEM characterization. TEM images of the < 5- μm Al sample revealed a high content of nm-sized particles as small as 200 nm. The μm -sized particles for this sample appeared to be at most 2 μm and scattered deposits were also found. All three nanopowder samples (40–60-nm Al, 60–80-nm Al, and 100–130-nm Al) contain significant numbers of particles outside the given size range, with larger particles in several hundred nanometers and the smallest particles in the range of only 10–20 nm. Thus, it is possible these nanopowder samples may essentially have indistinguishable particle sizes as a whole. This aspect can be further explored by conducting particle-size measurements via dynamic light scattering in the near future. Significant amounts of crystalline alumina deposits, presumably $\gamma\text{-Al}_2\text{O}_3$, were discovered for all samples. XPS survey spectra revealed a significantly higher content of surface carbon with correspondingly reduced Al peaks for the 60–80-nm Al sample. Compared with other nanopowder samples, the Sigma-Aldrich < 5- μm Al sample also had relatively higher C content.

Simultaneous thermal analysis successfully revealed the exothermo-oxidative nature of all samples but not the < 75- μm sample due to its irregular DSC baseline. It is determined that the active Al contents of all SkySpring nanopowders are 15 wt% lower than that of the Sigma-Aldrich μm -sized powders.

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List of Symbols, Abbreviations, and Acronyms

Al	aluminum
Al ₂ O ₃	alumina; aluminum oxide
Ar	argon
C	carbon
DEVCOM	US Army Combat Capabilities Development Command
DSC	differential scanning calorimetry
LASEM	laser-induced air shock from energetic materials
ms	millisecond
nAl	aluminum nanoparticle
nm	nanometer
O	oxygen
S&T	science and technology
SEM	scanning electron microscopy
STEM	scanning TEM
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
μm	micrometer
μs	microsecond
XEDS	X-ray energy-dispersive spectra
XPS	X-ray photoelectron spectroscopy

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