Investigating the Influence of Metallic Powder Handling and Storage Conditions on Flowability and Moisture Content

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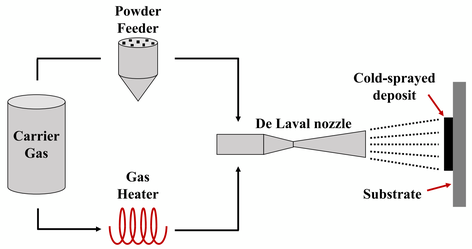
MOTIVATION



Powder-based additive manufacturing (AM) processes rely on **controlled feedstock powder** for successful processing.

For example, with **cold spray** processing, powders that have a narrow particle size distribution (PSD), low percentage of fine particles, and uniform morphology often spray better. Thus, it is essential to understand the **properties of the feedstock powder** in order to achieve predictable processing.

While most properties are fixed for a given batch of powder, properties such as **flowability** and **moisture content** can evolve during **powder handling and storage**, particularly when the powder is **exposed to ambient humidity and temperature**. Therefore, it is essential to understand how powder exposure to the environment affects flowability and moisture content so **property degradation** can be **mitigated**.

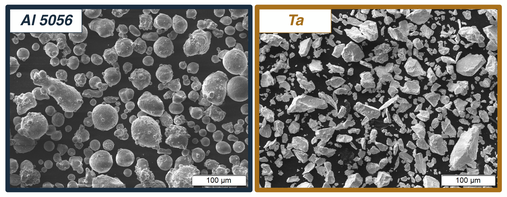
GOAL:

Evaluate the effect of **repeated environmental exposure** on powder *flowability* and *moisture content* for use in **AM** applications.

How can routine handling and storage of powder feedstock affect its in-process performance by altering powder properties?

PRELIMINARY EXPERIMENTATION

Two powders were investigated in this study: aluminum 5056 (***Al 5056***) and pure tantalum (***Ta***).



Both sets of powder were initially:
- **baked out** in a low-temperature oven to **drive off any moisture**
- **packaged** in separate vials in an **inert glovebox** (with argon gas) to **avoid environmental contamination**.

P = Packaged; = Exposed then Packaged; C = Characterized

	Day 0	Day 7	Day 14	Day 21	Day 28	Day 35	Day 42	Day 49	Day 56	Day 63	Day 70	Day 77	Day 84	Day 91
Lot #0	P													C
Lot #1	P	2 min 10 min												C
Lot #2	P	2 min 10 min	2 min 10 min											C
Lot #3	P	2 min 10 min	2 min 10 min	2 min 10 min										C
Lot #4	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min									C
Lot #5	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min								C
Lot #6	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min							C
Lot #7	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min						C
Lot #8	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min					C
Lot #9	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min				C
Lot #10	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min			C
Lot #11	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min		C
Lot #12	P	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	C
Lot #13	P												2 min 10 min	C
Lot #14	P											2 min 10 min	2 min 10 min	C
Lot #15	P										2 min 10 min	2 min 10 min	2 min 10 min	C
Lot #16	P									2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	C
Lot #17	P								2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	C
Lot #18	P							2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	2 min 10 min	C
Lot #19	C													

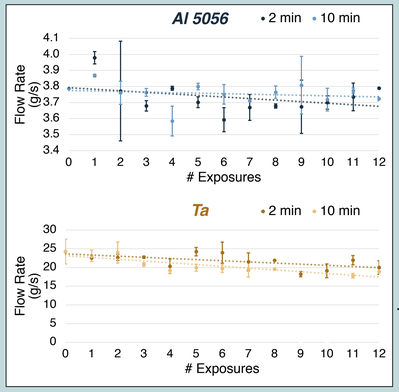
This study was conducted over a **3-month period**, from initial packaging to final characterization, whereby different lots of both powder types were subjected to **different environmental exposures** under standard laboratory conditions, according to the matrix on the left.

Through this experimentation, **various parameters** can be **altered** to see the effect on powder properties:
- Material type (***Al 5056*** vs. ***Ta***)
- # Exposures (0-12 exposures)
- Exposure duration (2 vs. 10 min)
- "Early" vs. "late" exposure
  - *"Early" = immediately after packaging (Lots #1-12)*
  - *"Late" = delayed after packaging (Lots #13-18)*

The powders were **characterized** both before *and* after exposure to observe the effect of powder exposure on flowability and moisture content.
- **Flowability** was characterized using the **Carney flow method** (ASTM Standard B964-16) in an **inert glovebox**, as an alternative method to the Hall flow method (ASTM Standard B213-20), which was unsuccessful with these powders.
- **Moisture content** was analyzed using **thermogravimetric analysis**, where a loss-on-drying measurement was taken after heating small amounts of powder at 250°C for 15 min after heating from room temperature at 20°C/min.

PRELIMINARY RESULTS

The results of the **flowability** and **moisture content** testing for the **initial 12 exposures** (Lots #1-12), as well as the **original powder sample** (Lot #19), are shown below for **both exposure durations**.

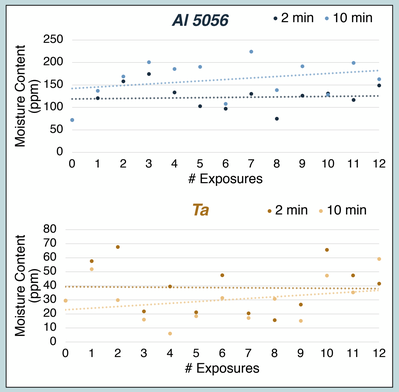


Flowability

Minimal changes in flow rate are observed as the number of exposures is increased and the exposure duration is changed; the values suggest a **slight decrease in flow rate as the number of exposures increases**. The results also demonstrate the gravity-driven nature of the Carney flow test, with an **innate flow rate difference** between ***Al 5056*** and ***Ta*** due to their material density differences. The only **"no flow" conditions** were seen with two ***Ta*** 10 min samples.

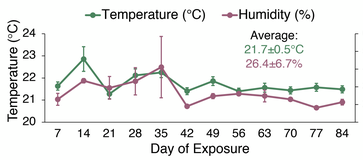
Moisture Content

The powder moisture content values for both materials show a **rather inconsistent trend** as the number of exposures is increased and the exposure duration is varied. As with flowability, it appears there is also a **material dependence** on the moisture content values within each powder sample.



The inconsistent and sometimes unexpected values of powder flowability and moisture content may potentially be explained by other influential factors: the **laboratory conditions** during exposure and the **powder size and morphology**.

Laboratory Conditions

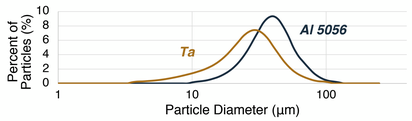


Over the course of the study, there were:
- **minute changes** in laboratory **temperature**
- **some spikes** in laboratory **humidity** that remained within a standard range (10-60%)

Given the data, **no direct correlation** can be made between the fluctuations in laboratory conditions and powder properties. Thus, **particle size-shape analysis** may prove particularly useful…

Inconsistency of powder properties may originate from the following powder sample **size-shape characteristics**:
- Large spread of particle size distribution
- Abundance of fine particles
- Irregular particle morphology
- High surface area-to-volume ratio

Particle Size Distribution



$$Span = \frac{D_{90} - D_{10}}{D_{50}}$$

$$Sphericity = \frac{4\pi \cdot Area}{Perimeter}$$

Median Value	*Al 5056*	*Ta*
Diameter (µm)	37.457 ± 0.519	26.356 ± 1.416
Span (-)	1.109 ± 0.065	1.616 ± 0.261
Volume % Under 20µm (%)	7.645 ± 0.364	31.418 ± 3.152
Sphericity (-)	0.965 ± 0.001	0.931 ± 0.001
Surface Area/Volume (µm^{-1})	0.233 ± 0.003	0.374 ± 0.013

PRELIMINARY CONCLUSIONS

- **Minimal effects** on powder properties from:
  - *Number of exposures*
  - *Exposure duration*
  - *Standard laboratory conditions*
- **Inconsistencies** in properties after exposure may stem from **particle size and shape characteristics**, though further work needs to be done to confirm this
- **Positive outcome** for powder handling and storage→ **little degradation of powder properties** with environmental exposure
  - ***Caution*** *should be taken when handling/storing powder under more* ***"extreme" conditions*** *(e.g., higher humidity) not covered in this study*

FUTURE WORK

- Further explore **complex relationship** between factors affecting powder properties
  - *Material dependence*
  - *Particle size-shape characteristics*
  - *Electrostatic charge and other surface effects*
- Evaluate more **"extreme" exposure conditions**
  - *Increased temperature and humidity*
  - *Longer exposure durations*
- **Alternative measurement methods** for powder properties
  - *Flowability: powder rheometer*
  - *Moisture content: oven desorption + Karl Fisher titration*
- Characterize any **compositional changes** in powder
  - *Oxygen, hydrogen, hydroxides, etc.*
- Consider influence on **processability** of powder in additive manufacturing
  - *Cold spray: deposition efficiency changes, critical velocity changes, etc.*

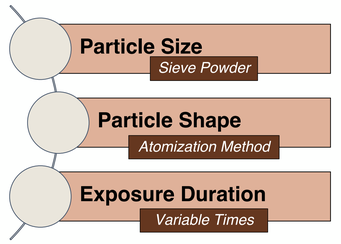
EXPLORING PARTICLE SIZE & SHAPE

Since preliminary experimentation has shown a **dependence** of powder flowability and moisture content on **particle size and morphology**, further controlled experimentation needs to be done to better understand this **complex relationship**. Other parameters will also be slightly modified from the preliminary study, including the **elimination of the number of exposures** as a variable, an **increase in the exposure duration**, and the use of an **environmental chamber** to control temperature and humidity.

FUTURE STUDY GOAL:

Evaluate the effect of **particle size and shape** on powder *flowability* and *moisture content* after exposure to controlled laboratory conditions.

Relevant Parameters to Vary



- **Powder Type**
- **Particle Size:** ***Sieve Powder***
  - Originally as-atomized (Unsieved)
  - Sieve: "Large", "Standard", "Fines"
- **Particle Shape:** ***Atomization Method***
  - Plasma Atomization
  - Gas Atomization
  - Water Atomization
  - Gas Atomization with Satellites
- **Exposure Duration:** ***Variable Times***
  - Original (Unexposed)
  - Short Exposure: 1 day
  - Medium Exposure: 7 days
  - Long Exposure: 30 days