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Optimization of Aluminum-Tin Ink Composition and Sintering in Atmospheric Conditions

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May 2019

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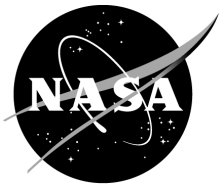

Optimization of Aluminum-Tin Ink Composition and Sintering in Atmospheric Conditions

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National Aeronautics and
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3D	three-dimensional printing
MSFC	Marshall Space Flight Center
SEM	scanning electron microscopy
SLM	selective laser melting

TECHNICAL MEMORANDUM

OPTIMIZATION OF ALUMINUM-TIN INK COMPOSITION AND SINTERING IN ATMOSPHERIC CONDITIONS

1. INTRODUCTION

This study will focus on the basics of generating an aluminum-tin ink that can sinter in air and exhibits properties near that of a solid aluminum-tin alloy. Sintering temperatures will also be assessed in this study. Once the optimal aluminum ink composition is determined, the optimal ink thickness for homogeneous sintering must be determined by additional experimentation.

Additive manufacturing is a rapidly developing and growing manufacturing process and has proven successful in many different ways. Processes, such as extrusion three-dimensional (3D) printing and selective laser melting (SLM), have proven to work but have limitations, such as material capabilities or density issues. SLM is a revolutionary process for additive manufacturing of metals but cannot be used in outer space due to the need for metallic powder which would diffuse into the atmosphere in a zero-gravity environment. For this reason, metallic ink additive manufacturing is a potential solution.

Work is being done on metallic ink additive manufacturing in a vacuum for electrical applications.[1] This project has focused on developing an aluminum-tin metallic ink that can sinter without the need of a vacuum or inert gas-purged atmosphere in order to prevent oxidation of the aluminum by adding flux.[2,3] Once a potential ink composition has been determined through sintering of small disks and thin layers of ink, the ink may be studied with a multimaterial 3D printer at NASA Marshall Space Flight Center (MSFC) in future experiments. If successful, this aluminum-tin ink will be capable for use on the International Space Station to make replacement parts quickly. Along with its zero-gravity advantages, this ink may also have applications on Earth because it may be extruded on a substrate with precise ceramic tips in a 3D printing process. This would allow the fabrication of precise, complex shapes and may generate a much faster and more efficient printing process as compared with traditional powder bed additive manufacturing processes. The process would not be limited by a small building volume because the system would not require an enclosed chamber.

2. EXPERIMENTAL PROCEDURE

Aluminum ink formulation materials were measured out according to the weights listed in table 1. These weights changed slightly for each variation in flux percentage. Measurements were made by adding one component to a beaker and then tarring the scale before adding the next constituent. Using a small metal spatula, the materials were mixed together. This hand-mixing process takes 2 to 3 hours and, once completed, all of the metal powder will be in an initial solution. Next, the mixed ink was milled on a three roll mill. This is a high-shear milling operation used to completely disperse the aluminum nanoparticles and form a homogeneous paste. (See photo of milling operation in fig. 1.)

Table 1. Example composition for 15% flux aluminum ink.

Material	15% Flux Amount (g)	(%)
Al 40 nm HongWu	62.9531726	52
Sn 70 nm HongWu	9.442975889	8
V3015 glass	1.628099291	1
1250 brazing powder	18.08999213	15
OC-40	1.085399528	1
6% N50 in terpineol	27.89476786	23

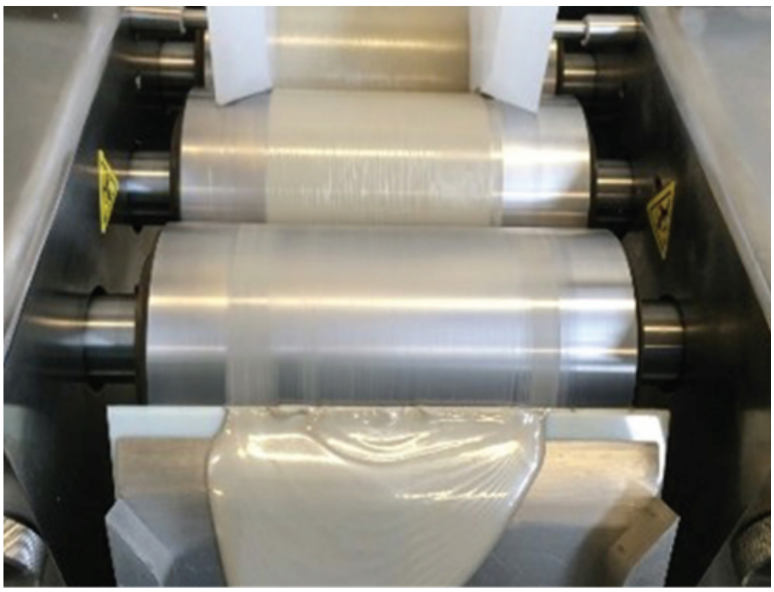


Figure 1. Three roll milling machine used to achieve sufficient mixing of aluminum ink.

The experiment was designed to begin with a lower percentage of flux material and then add flux to reach higher flux percentage. In this case, the new material must be hand mixed and remilled on the three roll mill.

Next, 1 to 2 grams of the aluminum ink was weighed out, put into a pressing die, and dried at 120 °C for 24 hours. The dried sample was compressed in the die by a punch into a pellet using a hydraulic press. The sample was compressed at approximately 15,000 psi. The pellet was then removed from the mold and the process repeated until two to three pellet samples of each flux percentage were generated.

The samples were sintered in a tube furnace in clean air with two different sintering procedures. One sintering procedure used a 600 °C furnace temperature while the other sintering procedure used a 400 °C temperature. The temperature was reduced to 400 °C after the 600 °C sintering cycle produced brittle samples. The 400 °C temperature was chosen because it was proven successful by Oakridge National Laboratory as documented in reference 1. The furnace sintering dwell time was 18 hours. This can be optimized later and reduced in time, but complete sintering must be ensured. Following sintering, the samples were submerged in deionized water, removed from the water, and air dried for approximately 30 minutes. The samples were weighed in air and then in deionized water in order to obtain density values. Many samples dissolved or broke during the density measurements. The consolidated samples were mounted to observe their microstructural cross section. Following mounting, they were hand polished and then machine polished down to a 0.5 micrometer finish. The samples were etched with a Keller's etchant and then observed using an optical microscope. Early samples were subjected to microhardness testing after optical imaging but results were nonhomogeneous in all cases so the data were not used.

After generating and testing cylindrical metallic ink samples, ink was applied in thin layers to a ceramic substrate. This test was done on both 14% and 15% flux inks because of the results found from the initial cylindrical samples. This was done by applying heat-resistant tape to a ceramic substrate and then using a spatula to apply a layer of ink with equivalent thickness as the tape. Multiple single-layer samples were produced and then dried in a 120 °C furnace prior to sintering at 400 °C. One of the single-layer samples had a second and third layer added to it with the same method as the first layer. These samples were mounted in epoxy in order to show their cross section and then imaged using an optical microscope.

3. RESULTS AND DISCUSSION

The materials shown in table 1 each contributed to the success of the aluminum-tin ink. The tin was added to the ink in order to fill interstitial gaps in the aluminum. The V3015 glass powder was added in order to increase the ink's ability to adhere to a substrate. Future studies will compare an ink with and without the addition of tin to see if an ink without the tin will sinter properly. The glass will be removed in subsequent studies focused on structural ink printing because adherence to a substrate will not be paramount. The 1250 brazing powder was added to the ink to provide protection from oxide formation. The OC-40 was added to increase the flow of the ink during 3D extrusion printing processes. Lastly, the 6% N50 in terpineol was added to allow the materials to mix into solution and evaporated from the ink during drying and sintering.

When comparing figures 2 through 6, the samples made at 15% flux, shown in figure 6, appeared to exhibit the greatest consolidation after sintering. As shown in figures 2 and 5, samples with 5% flux charred completely at both sintering temperatures of 400 °C and 600 °C. Table 2 shows the density measurements and observations made for samples made with flux percentages between 5% and 25%. The samples with flux percentages of 13%–15% degraded the least during submersion in deionized water. Although the 13% and 14% samples, shown in figure 7, had a higher average density than the 15% samples, the 15% samples performed better in subsequent sample preparation.

Figure 2. Ink samples sintered at 600 °C for 18 hours with flux percentages of 5%, 10%, 15%, 20%, and 25% in order from left to right (two samples at each compostion).

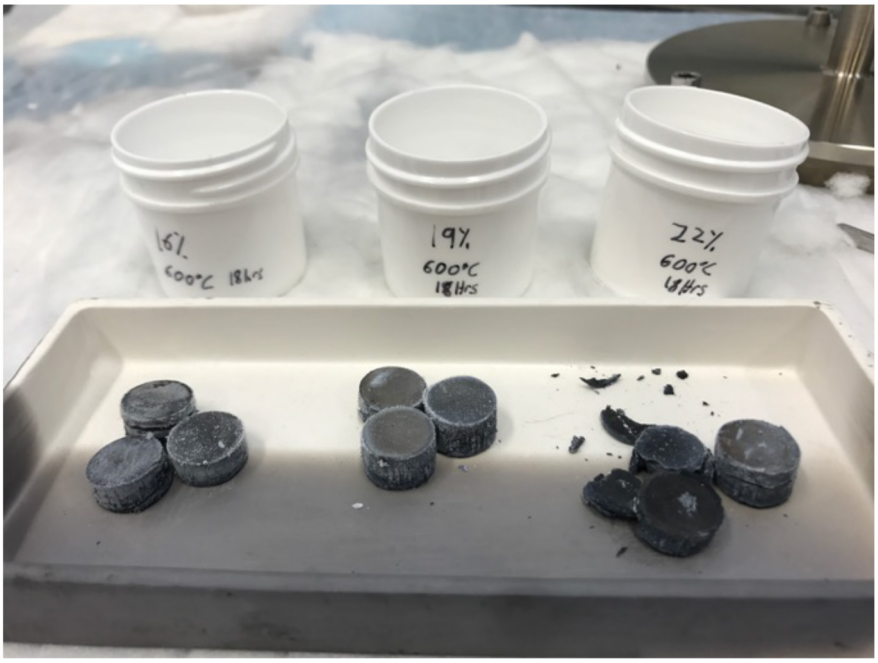


Figure 3. Ink samples sintered at 600 °C for 18 hours with labeled flux percentages of 16%, 19%, and 22%.

Figure 4. Ink samples sintered at 400 °C for 18 hours with labeled flux percentages of 16%, 19%, and 22%.

Figure 5. Ink samples sintered at 400 °C for 18 hours with labeled flux percentages of 5% and 10%.

Figure 6. Samples sintered at 400 °C for 18 hours with a 15% flux composition ink.

Table 2. Density measurements.

Density Measurements (g/cm^2)						
	400 °C			600 °C		
Flux (%)	S1	S2	S3	S1	S2	S3
5						
10	2.36	2.36	2.35			
12	Broken	2.43	Broken	N/A	N/A	N/A
13	2.42	2.45	2.45	N/A	N/A	N/A
14	2.46	2.42	2.52	N/A	N/A	N/A
15	2.4	2.42	2.42	N/A	N/A	N/A
16	2.3	2.26	2.32	2.38	2.45	2.52
19	2.32	2.43	2.37	2.37	2.39	2.46
22	2.36	2.4	2.56	2.32	2.33	2.39
25	N/A	N/A	N/A			
	Samples broke down on submersion for density measurement but stabilized eventually (density measurement accuracy in question)					
	Samples bubbled and dissolved very little					
	Samples bubbled on submersion but did not dissolve					
	Samples too degraded for density measurement or broke down completely when submerged.					

Figure 7. Samples sintered at 400 °C for 18 hours with a 12%, 13%, and 14% flux composition.

Figure 8 shows optical images of thin samples that better represent the extent that sintering can penetrate into an aluminum ink sample. When comparing figure 8(d)–(f), the 14% flux sample exhibits cracking, the 16% flux sample exhibits extensive cracking, and the 15% sample has no apparent cracking. These three images were taken at × 100 with the region of least cracking observed under × 25 magnification. Figure 8(a)–(c) shows a similar result; the 14% flux sample in figure 8(a) has extensive cracking but also has a region of consolidated material in the bottom right corner. The 16% flux sample in figure 8(c) shows extreme cracking and has no evident area of consolidated material. The 15% flux sample in figure 8(b) exhibits large internal cracks which propagate to the edge of the sample on the top and bottom. These cracks are not observed all over the sample, and it has a greater region of consolidated material towards the left side of the image and some on the top and bottom of the sample with respect to the other two samples. Other samples of the same flux percentage were generated earlier in the study and were of greater thickness. These samples tended to break down during processing and testing which can be attributed to a greater sample thickness, causing a lack of internal sintering. Thinner samples appeared to mitigate this issue, leading to the conclusion that a thickness limitation is necessary for aluminum ink samples to be successful.

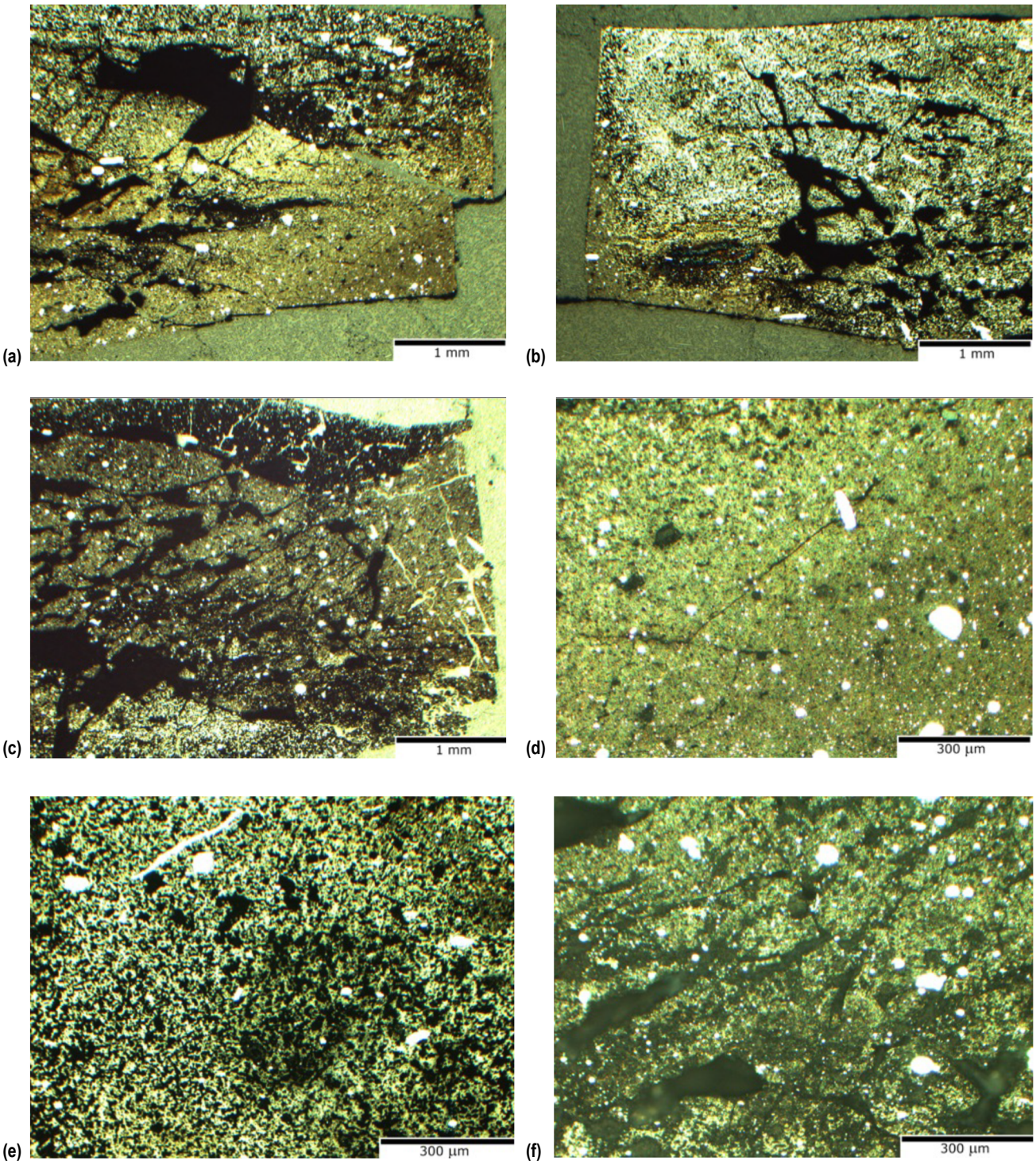


Figure 8. Cross section of thin, cylindrical samples at 400 °C for 18 hours with various flux percentages: (a) 14% S4, × 25, (b) 15% S4, × 25, (c) 16% S4, × 25, (d) 14% S4, × 100, (e) 15% S4, × 100, and (f) 16% S4, × 100.

The samples shown in figure 9 were created by coating a ceramic 2 inch by 2 inch substrate with sections of the metallic ink. Heat-resistant tape was applied to the ceramic substrate prior to the application of the ink so multiple separate lines of metallic ink could be applied to one ceramic plate. A flat-edged plastic spatula was used to remove excess ink so the remaining ink would be approximately the thickness of the heat-resistant tape. The samples were put into a drying furnace with a temperature of 120 °C in order to dry off the solvent. The tape was removed and the samples were put into a sintering furnace for 18 hours at a temperature of 400 °C. These samples were then removed from the furnace and mounted in epoxy so they could be imaged under an optical microscope. One 15% flux sample was also imaged with scanning electron microscopy (SEM).

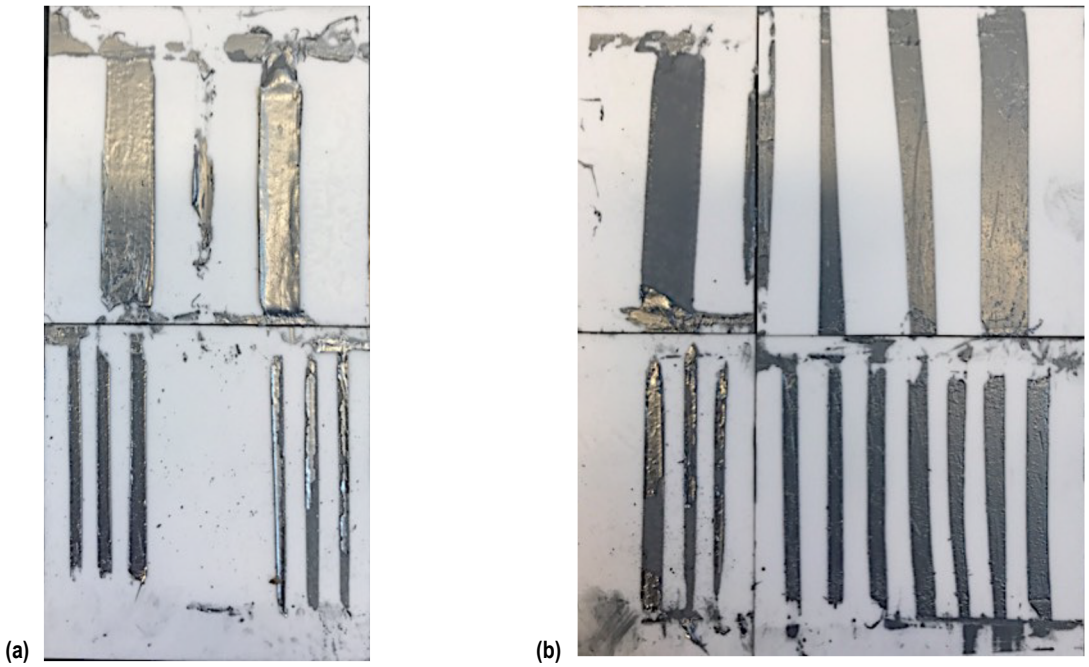


Figure 9. Overview images of (a) 14% and (b) 15% flux ink applied in thin layers to a ceramic substrate.

Figure 10(a)–(c) shows the cross sections of a single layer of the aluminum-tin ink. The sample appeared to sinter evenly and adhered to the ceramic substrate surface. The cross section had a uniform microstructure throughout but contained some inclusions as shown in figure 10(b), most likely due to the segregation of metallic particles, and may be avoided in the future by increasing the milling time to ensure a more uniform ink mixture.

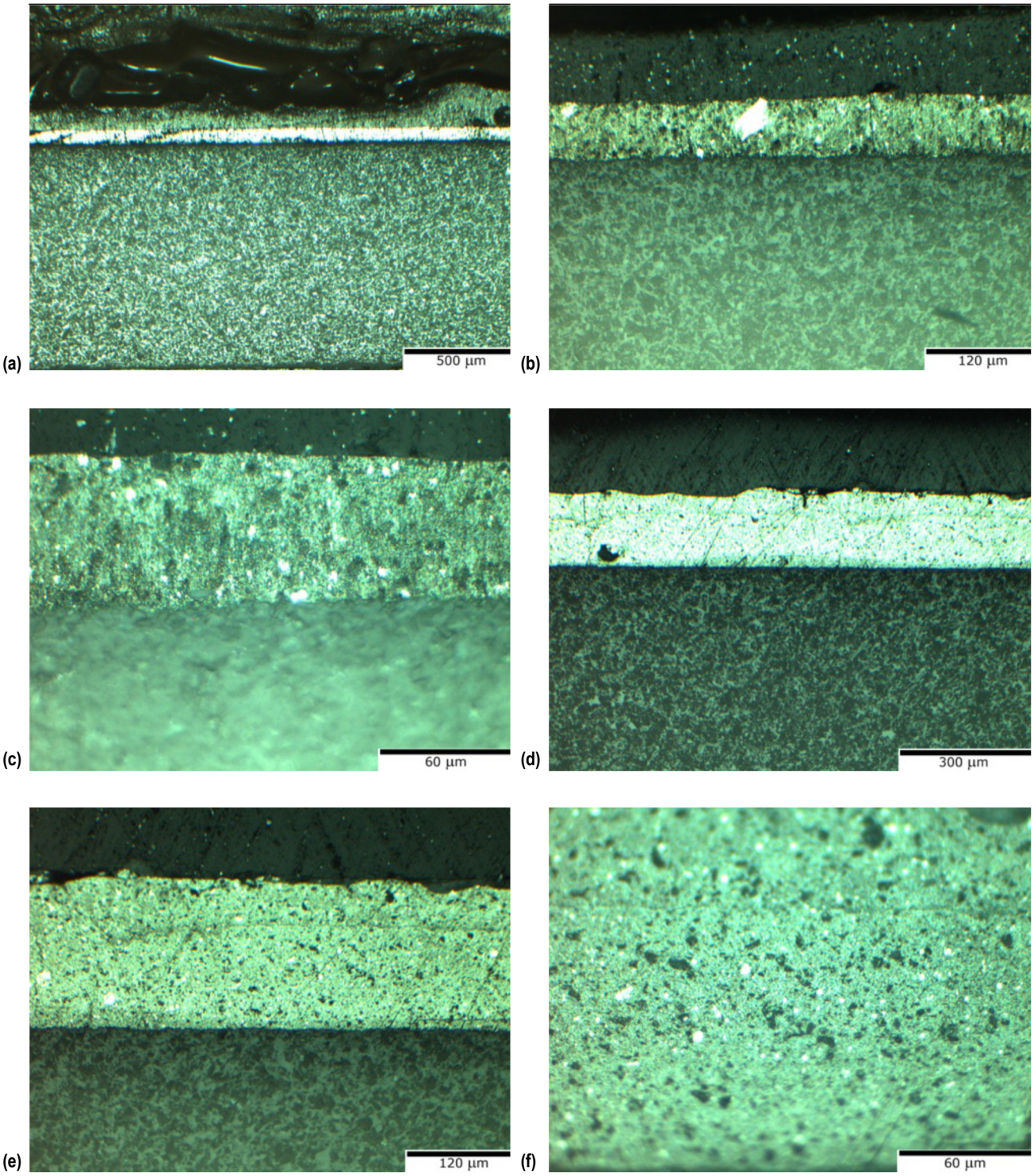


Figure 10. Optical images of cross sections of 15% flux ink applied in thin layers to a ceramic substrate: (a) Sample 1 at × 50, (b) sample 1 at × 200, (c) sample 1 at × 500, (d) sample 2 (three layers) at × 100, (e) sample 2 (three layers) at × 200, (f) sample 2 (three layers) at × 500.

Figure 10(d)–(e) shows cross sections of ink samples containing three layers of ink. Each layer was applied and sintered prior to the application of the next layer. The surface of each layer was not fully uniform due to the application process. Although layer thickness and shape was not uniform, the layers adhered to each other, and in some locations, were indistinguishable. This proves that the 15% flux aluminum ink may have potential applications in an additive manufacturing setup. Future experimentation must be done using the nScrypt 3D printer (shown in fig. 11) located at MSFC in order to validate this ink composition for additive manufacturing uses. Initial printing trials on the nScrypt printer indicated that this material will print effectively with the 3D direct-write deposition process.

Figure 11. Image of nScrypt 3D printer.

Figure 12 shows a 15% ink sample that was sintered and imaged with SEM. Figure 12(a) shows the sample at a magnification of × 50 and has multiple cracks present. Those cracks likely propagated due to the brittle nature of the material at such low thickness. Further experimentation is necessary to determine if cracks will propagate in thicker samples consisting of many layers. Figure 12(b) shows the sample at × 500 magnification and exhibits multiple precipitates throughout the image. These precipitates appear to be uniformly distributed and further experimentation is necessary to determine if they will have detriment on material properties.

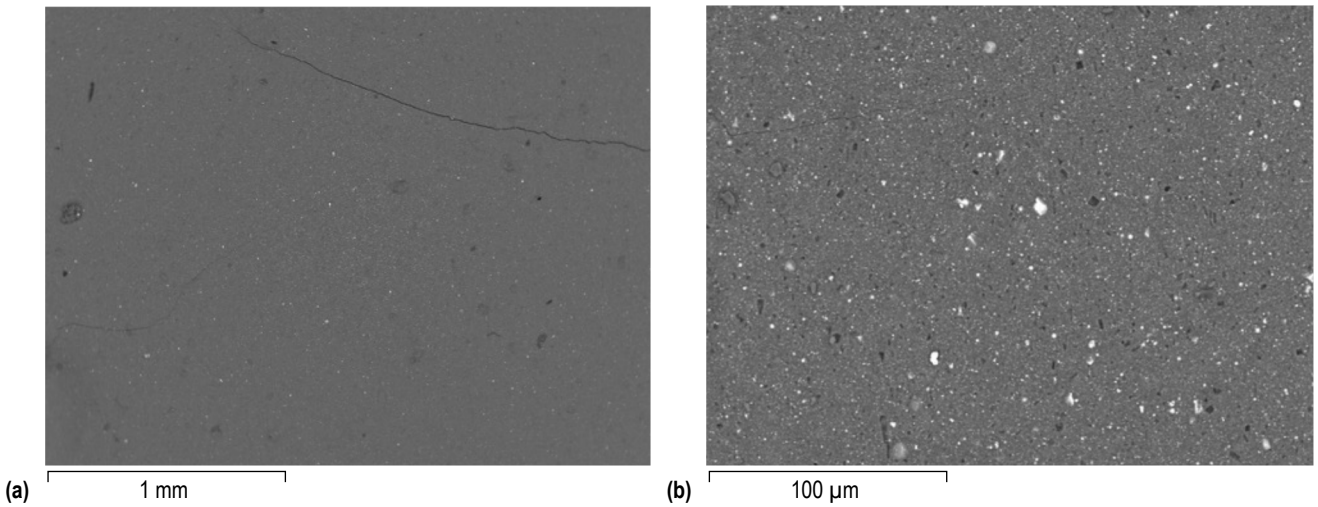
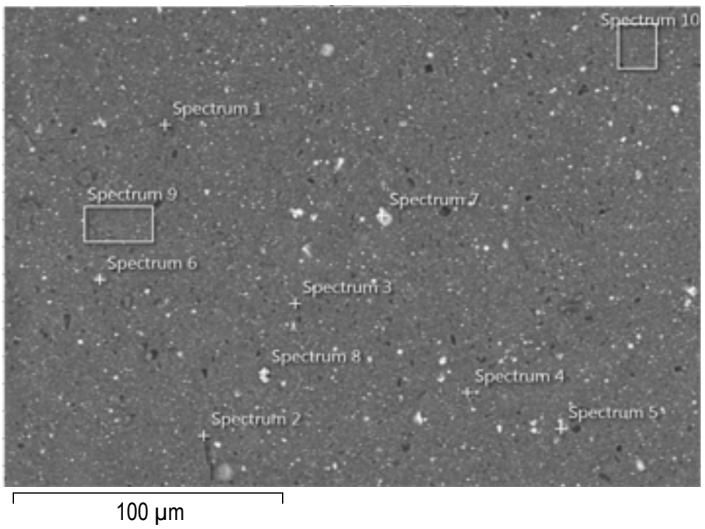


Figure 12. SEM surface images of 15% flux ink applied in thin layers to a ceramic substrate: (a) × 50 and (a) × 500.

The composition of one sample of the 15% flux ink was determined by SEM and is shown in figure 13. Aluminum and tin concentration changes throughout the sample, and it may be attributed to insufficient milling of the ink. This may also be due to separation of the ink during extended storage. To prevent this in the future, milling shall be done shortly before the ink is deposited on the substrates and sintered. Other elements, such as carbon, oxygen, fluoride, and potassium, were found throughout the sintered aluminum-tin ink sample. These elements likely deposited during the sintering process due to their presence within the atmosphere.



Composition (Weight %)

Label	C	O	F	Na	Al	Si	K	Fe	Sn	Te	Pb
Spectrum 1	5.61	17.97	26.51	0.08	33.75		13.92		2.17		
Spectrum 2	5.51	19.10	16.50		47.47		8.81		2.62		
Spectrum 3	3.78	14.27	6.80		62.15		3.08	0.13	9.78		
Spectrum 4	3.64	11.36	4.73		65.98		2.14	0.23	11.91		
Spectrum 5	2.83	18.05	10.37		20.35		2.31		46.09		
Spectrum 6	3.47	14.76	8.64		52.43		5.35	0.16	15.20		
Spectrum 7	4.76	15.82	9.29		9.34	2.82	3.46		2.02	13.52	38.96
Spectrum 8	5.31	19.03	15.51		40.66		7.49		12.01		
Spectrum 9	5.69	17.55	9.30		56.34		4.46		6.67		
Spectrum 10	5.62	16.78	8.35		59.02		3.98		6.25		

Figure 13. Composition of aluminum-tin ink sample obtained from SEM, × 500.

All samples, aside from samples made with 15% flux and sintered at 400 °C, bubbled rapidly in deionized water, as the excess flux salts reacted with the water and released carbon dioxide. This was caused by internal porosity, observed later in the process, and was known to be present because density values were lower than that of the material constituents. The best results came from samples made at 400 °C for 18 hours and with composition between 10% and 15% flux.

After making samples at flux percentages of 5%, 10%, 12%, 13%, 14%, 15%, 16%, 19%, 22%, and 25%, all samples with flux greater than 15% and lower than 13% were observed to either burn up during sintering or fail at some point between sintering and polishing. Samples made at 12% and 400 °C exhibited a metallic surface appearance like 13%, 14% ,and 15%, as shown in figures 5 and 6. The 12% samples dissolved greatly in deionized water, and upon drying, burst into pieces as shown in figure 14. Similar observations were seen in all flux percentages below 13% and can be seen in figure 15 with the apparent dissolving of the 10% flux samples. It appeared as if the samples heated in some way during submersion in deionized water.

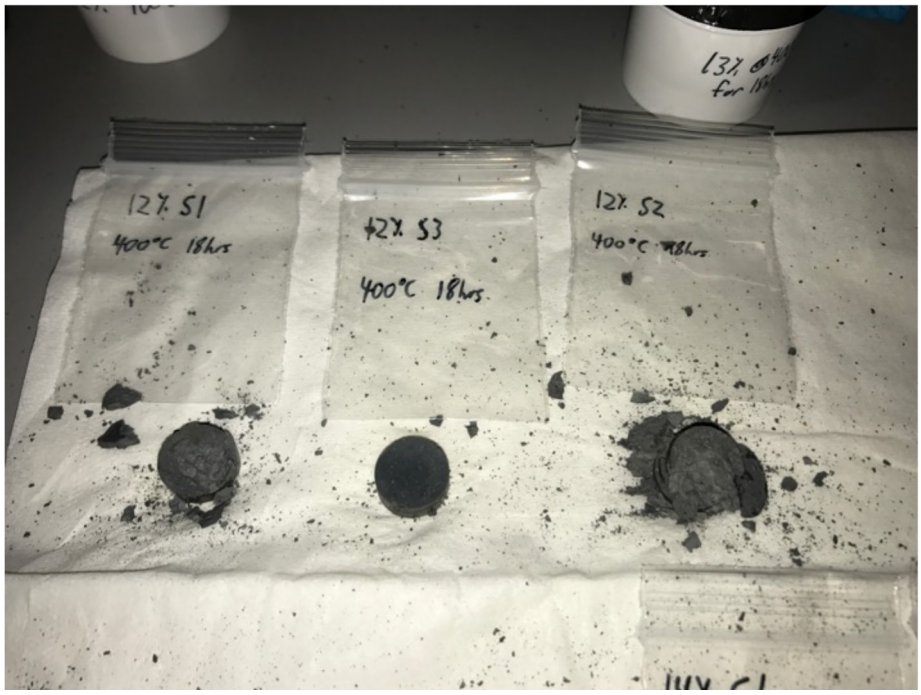


Figure 14. 12% flux samples following submersion in deionized water.

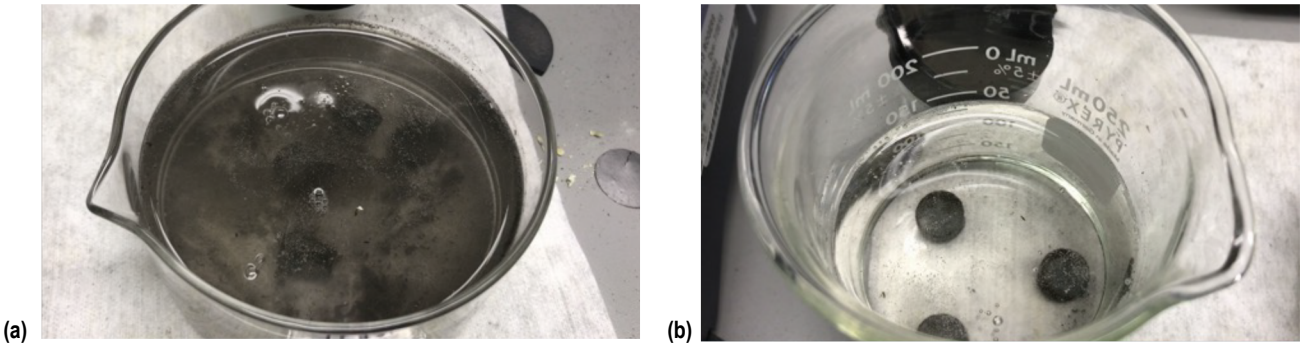


Figure 15. Samples sintered at 400 °C for 18 hours and then submerged in deionized water prior to density measurements: (a) 10% flux and (b) 15% flux.

All samples that were sintered at 600 °C for 18 hours failed by crumbling during polishing or dissolved in deionized water during density measurements. They were extremely brittle and dissolved, releasing a black dross (impurities from the sintering flux) into the water. This black substance was unsintered material within the pellets which came into contact with the deionized water through penetration of internal porosity. Hardness tests were attempted on samples that could withstand mounting and polishing but yielded inhomogeneous results. Although samples made at 600 °C for 18 hours were extremely brittle, the samples that were made at 16% and 20% flux were capable of withstanding polishing. Two to three samples were made at each flux percentage and some of them made it further into the process. Samples of 15% and 20% sintered at 600 °C

dissolved partially upon submersion in deionized water but made it past polishing. Since two out of three samples from each sample set made at 600 °C failed, the conclusion that 600 °C is a poor sintering temperature was reached.

Samples made at 400 °C for 18 hours with flux percentages of 13%, 14%, and 15% exhibited the best surface appearance and all samples survived submerged density measurements. During density measurements, these samples bubbled slightly but did not discolor the water like all samples outside this flux percentage range. This indicates that the samples were not fully dense but did not dissolve greatly in the water. According to table 2, all three of these flux percentages exhibited consistently greater density than any of the samples outside this flux range. Hardness testing results of all samples from 5% to 25% flux were inhomogeneous and were discarded due to the inability to ensure accuracy.

The samples generated at 13%, 14%, 15%, and 16% at 400 °C had a hard exterior, and when cross sectioned, showed a softer, inhomogeneous interior. This proved that for sintering to work properly, the samples need to be much thinner. For this reason, samples of these four flux percentages were generated again at lower thickness to improve sintering. These samples were then cross sectioned, the extent of sintering was assessed, and some of them showed positive results. The thickness of the homogenous exterior was shown to be greatest in the 15% S4 sample shown in figure 8(b) and (e). As discussed above, when thin layers of the ink were deposited and sintered, they exhibited consolidated structures without the extensive cracking seen in the pellet samples. Of the thin-layered samples, those made at 15% flux exhibited a more consolidated and crack-free cross section than the 14% flux samples.

4. CONCLUSION

Samples sintered at 600 °C were brittle, oxidized, and partially sintered, resulting in poor density. Optimal results were observed at a sintering temperature of 400 °C. At 400 °C, samples containing 13%–16% flux exhibited the most consistent density and stayed consolidated. Samples of 13% flux bubbled the least of all samples when submerged in deionized water, indicating a more complete reaction of the flux during the sintering process. Although these samples were much more consolidated and exhibited a more metallic surface finish and consistent density, they still had very weak internal properties. This study has helped to pinpoint a narrow flux range between 13% and 16% and a sintering cycle around 400 °C. The ideal flux percentage was determined to be 15% due to consistent density, ability to withstand the experimental procedure, and because that composition exhibited the most homogeneous metallic exterior with the least internal cracking.

5. FUTURE WORK

In order to utilize the material developed with the research described in this TM, it must be developed further. The material must be tested for desired properties, such as density, electrical conductivity, and hardness. It must also be tested for material properties and consolidation over a range of different thicknesses in order to determine the window of acceptable deposition thicknesses. The thicknesses discovered must then be validated for use on the nScrypt 3D printer. Then the material can be printed and tested for an array of electrical properties along with any other properties that may pertain to the use of an oxide-resistant aluminum ink. Potential applications for the material throughout industry must also be researched and established to justify additional experimentation.

Sintering techniques, such as photonic and laser sintering, shall also be explored as methods to harden the aluminum ink into a useable material. The material may also be tested for deferent deposition methods such as various extrusion 3D printing processes.

Oak Ridge National Laboratories has already been collaborated with on the method of photonic sintering. Further funding will be needed to create a basic photoelectric additive manufacturing setup at MSFC so that further analysis of build layer thickness and sintering methods can be achieved. Aluminum additive manufactured samples will be tested for hardness, tensile strength, density, and microstructure.

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14. ABSTRACT

Traditional manufacturing techniques for aluminum use lengthy machining processes that produce large amounts of waste and require many parts to complete an assembly. With additive manufacturing, near-net-shape parts can be created. One method for metallic additive manufacturing uses a metallic ink mixture. This method has potential advantages both on the ground and in zero gravity. Aluminum ink readily forms an oxide layer when exposed to the atmosphere. An approach to avoiding oxidation of aluminum during additive manufacturing is to add a flux which prevents oxidation. An optimal ink composition has been found using an aluminum-tin mixture and has proven successful with regard to oxidation prevention. Additional experimentation must study this material in an additive manufacturing setup to test for density, tensile strength, and microstructure.

15. SUBJECT TERMS

aluminum, additive, metal, sinter, manufacture, ink

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