3D Printing with Mixed Powders of Boron Carbide and Al Alloy

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Abstract: Laser additive manufacturing with mixed powders of boron carbide and aluminum alloy is investigated. Parameters such as laser power, scan speed, scan pattern, and hatching space are systematically evaluated and optimized to obtain the desired density and porosity. These results show that the AM part contains 20 wt% boron carbide and 80 wt% aluminum alloy, which are well mixed and synthesized during the melting process. Its mechanical properties are close to those of aluminum. A thin-wall structure based two dimensional and three dimensional radial collimators were fabricated with well-controlled geometry for neutron scattering measurement.

Keywords: additive manufacturing; fiber laser; boron carbide; aluminum; neutron scattering radial collimator

1. Introduction

Using neutron scattering measurement, materials at the atomic level can be probed, which is essential to develop innovations like faster computers, tougher armor, and effective medicines. Radial collimators are used when a neutron scattering instrument employs a large area of detectors to cover a wide range of scattering angles [1,2]. A radial collimator consists of a series of absorbing septa radially oriented from a fixed point. These collimators will often oscillate several degrees at the sample position. Conventional neutron collimators use thin foils, typically made from mylar, aluminum (Al), or stainless steel, coated in an absorbing material, such as gadolinium (Ga, melting temperature 1312 °C, thermal conductivity 10.6 W/m.K) or enriched boron (B, melting temperature 2076 °C, thermal conductivity 27.4 W/m.K), to absorb divergent incident neutrons, or those scattered from unwanted sources. The foils are held together and tensioned to ensure uniformity, using bolted frames. The unit for gripping and tensioning the foils limits how closely they may be placed together, their angular separation, their proximity to the sample, and therefore the efficiency of the collimator. Modelling software, in combination with ray tracing simulations, shows that there are designs that can greatly improve the performance of the collimator but are not possible to implement using conventional assembly or machining techniques.

Additive manufacturing (AM), especially laser-based AM, becomes a powerful tool to replace conventional methods such as casting and hot spray, due to its cost effectiveness and capability of making complex structures and compositions [3–9]. Specifically, AM shows significant advantages in parts fabrication with mixed powders, metals, and/or ceramics, to achieve desired performance [7–9]. However, direct thin-wall (2D and 3D) fabrication for radial collimators for neutron scattering instrument is still a challenging field [1,2], especially for multimaterials such as a mixture of boron carbide (B₄C) and Al alloy (AlSi10Mg). This is mainly due to a limited understanding of composition control, laser melting control, melted structure formation, and phase transition.
In this paper, research is extended from the fabrication of functional ceramic components to directly make radial collimators for neutron scattering measurement without binders and post process. To the best of our knowledge, this is the first publication demonstrating additive manufacturing using mixed powders of B₄C and Al alloy.

2. Materials and Methods

Boron carbide (melting temperature 2763 °C, thermal conductivity 29 W/m.K, density 2.52 g/cm³, and thermal expansion coefficient \(3 \times 10^{-6}/K\)) is a very common material used for radial collimators, because it is relatively cheap, and safe in terms of handling. However, its brittleness makes it hard to use in fabrication of thin-wall radial collimators with 3D printing technology. It is found that by adding Al (melting temperature 660 °C, thermal conductivity 237 W/m.K, density 2.70 g/cm³, and thermal expansion coefficient \(23 \times 10^{-6}/K\)) to B₄C, the strength and thermal handling capability can be improved significantly and the brittleness can be reduced without increasing in weight. In this paper, B₄C powder (American Elements, Los Angeles, CA) and Al alloy (AlSi10Mg) powder (CNPC, China) are used to make radial collimator parts. These powders are readily available standard commercial products. The mixing ratio of 20% B₄C and 80% AlSi10Mg is selected to make thin walls matching the performance of neutron absorption (B [10] concentration) by using the conventional method, whose Myler septa are coated with Boron [2].

Microscopic images and size characterization are given in Figures 1 and 2 for B₄C powder and AlSi10Mg powder, respectively. The size of the B₄C particles is generally in the range of 10 to 45 µm. However, the shapes are irregular, which is not ideal for AM process. In reality, we have no other choice since this is the best quality we could find from all suppliers. AlSi10Mg particle size is finer than B₄C, since 80% of the particles are measured within 10 to 30 µm. Moreover, it has spherical shape, which has better flowability. Practically, different powder sizes can help make dense components [10].

![Figure 1. Characterization of boron carbide (B₄C) powder.](image1)

![Figure 2. Characterization of Al alloy (AlSi10Mg) powder.](image2)
80 wt% of AlSi10Mg alloy powder was mixed with 20 wt% B₄C powder before the AM process, by using a jar mill machine to run 24 h. A series of cubic samples were printed to measure the density and surface roughness. Powder bed fusion (PBF) system made by PolarOnyx was used to do the AM process. This system was modified to fit for low-mass ceramic 3D printing and filled with Argon gas.

The process parameters are listed as follows: laser average power: 100, 150, and 200 W on targets; laser type: fiber laser; laser operation mode: pulse repetition rate of 200 MHz, pulse width of 1 ns; laser wavelength: 1070 nm; scan speed: 100 mm/s to 500 mm/s; patterning type: lines; hatching order: bidirectional; hatching space: 50 to 150 µm; and powder layer thickness: 50 µm. Fiber laser can provide stable operation and reasonable absorption, which is essential to the AM process.

All of the measurement data on roughness and density are summarized in Figure 3. It is noted here that some of the data are not complete due to the fact that those samples are damaged during polishing process. However, the rest of the data are sufficient enough to make a conclusion on process optimization. Over 99% relative density (image-J is used) and acceptable roughness (<50 µm, top surface) were obtained when the average power on target was greater than 150 W. Further study on uniformity was carried out by fixing 150 W average power and scan speed of 100 and 150 mm/s. The hatching space varies from 130 to 150 µm (or 0.13 mm to 0.15mm) at a step of 10 µm. Figure 4 shows that all of the samples have good uniformity of relative density (<2% variation, from 97–99%).

**Figure 3.** Top surface roughness (left column) and density measurement (right column) of mixed B₄C (20 wt%) and AlSi10Mg (80 wt%).
Figure 4. Uniformity test for samples using different parameters. All samples on the top use scan speed 100 mm/s, whereas on the bottom uses scan speed 150 mm/s. From the bottom to the top rows, the hatching space is 0.13, 0.14, 0.15, 0.16, and 0.17 mm, respectively.

3. Results and Discussion

Hardness, Young’s modulus, SEM, and energy dispersive x-ray spectroscopy (EDX) were tested for a sample printed with 150 W power and 150 mm/s scan speed. Figure 5 shows the load versus displacement for a cube sample. Nanovea PB1000 Mechanical Tester is used for the hardness test. It appears that the hardness (110 MPa) and Young’s modulus (11.7 GPa) of mixed B\textsubscript{4}C/Al are close to those of Al. This is mainly due to a large portion of Al powder in the sample. An important feature is that the synthesized part is less brittle and has better elastic property than B\textsubscript{4}C.

Figure 5. Load curve.

Figure 6 shows the SEM and EDX results of a polished cubic sample with 99% density. EDX element analysis over an area of 1 mm x 1 mm indicates that Boron increases by 7 wt% and Al decreases by 30 wt% after the AM process. This has been verified with different testing areas and samples. One reasonable explanation is that a significant portion of Al was evaporated during the process due to its low melting temperature (660 °C) and boiling temperature (2740 °C). However, this will need to be further studied and confirmed in on-going research activities.

Figure 6. Cont.
This provides strong evidence that AM can not only melt powders to form 3D structures but also can carry on in situ synthesis to form new compounds to change their properties (such as mechanical, electrical, etc.) for specific applications. As a note, from the phase diagram of Al and B₄C, aluminum carbide (Al₄C₃) and aluminum diboride (AlB₂) forms at a temperature over 1400 °C and aluminum diboride (AlB₂) at a temperature over 600 °C. Detailed study of AM synthesis is beyond the scope of this paper and will be present in a separate publication.

**Figure 6.** SEM and EDX test results of a cubic sample with mixed B₄C (20 wt%) and AlSi10Mg (80 wt%).

Mapping of element test using EDX was done with the same testing area shown in Figure 6 to identify the mixing quality of B₄C and Al. As shown in Figure 7, Boron element is well distributed in the Al structure. Again, it shows that Boron appears to have a larger percentage after the AM process.

**Figure 7.** EDX mapping test of boron (left) and Al (right). Boron is well distributed in the Al structure.
Two types of collimators were made with the same parameters for hardness test (150 W power and 150 mm/s scan speed). One is the honeycomb type (3D), of which geometrical parameters were taken from reference [2]; the results are shown in Figure 9. In control of the geometry and tolerance (10 µm level), very consistent results have been achieved. Another type is the thin-wall collimator (2D). A small version of the optimized thin-wall type of collimator was printed and is given in Figure 10. It is under evaluation at Oak Ridge National Labs (ORNL). Part of the sample was cut and tested with EDX and XRD. It is consistent with the data from cubic samples (Figure 8) in terms of synthesis of Al\(_4\)C\(_3\) and AlB\(_2\). Especially, we observed that Boron concentration has been increased significantly and Al has been decreased due to evaporation during the collimator printing.

![Figure 8. XRD test of mixed B\(_4\)C and AlSi10Mg sample.](image)

| Collimator | H x W (mm) | L (mm) | T (mm) | b (mm) |
|------------|------------|--------|--------|--------|
| Paper ref. | 58x58      | 33     | 0.5    | 3.4    |
| Our design | 58x58      | 33     | 0.5    | 3.4    |
| Printed part. | 57.978  | 33 | 0.51  | 3.39 |

![Figure 9. 3D collimator structures (honeycomb).](image)

![Figure 10. A small version of the optimized collimator design.](image)
4. Conclusions

For the first time, 3D printing using PBF system was demonstrated to manufacture radial collimators using mixed B$_4$C (20 wt%) and AlSi10Mg (80 wt%) powders. The processed samples were examined and characterized by optical microscope, SEM, EDX, and XRD. It is found that Boron element is well distributed in Al structure, and the synthesized parts demonstrated excellent mechanical strength and brittleness improvement. We observed a significant portion of Al was lost during the AM process, for which further investigation is on-going to understand the mechanism.

Moreover, newly formed crystal phases such as aluminum carbide (Al$_4$C$_3$) and aluminum diboride (AlB$_2$) are observed during the melting and solidification process. By control of melt pool temperature and composition, different level of synthesis can be achieved to match specific application. This opens a new frontier for 3D printing to synthesize new compounds and explore new properties. We believe there will be a great potential for the aerospace and defense, automobile, semiconductor, and energy industries.

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