Femtosecond Fiber Laser Additive Manufacturing of Tungsten

(Invited Talk)

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ABSTRACT

Additive manufacturing (AM) is promising to produce complex shaped components, including metals and alloys, to meet requirements from different industries such as aerospace, defense and biomedicines. Current laser AM uses CW lasers and very few publications have been reported for using pulsed lasers (esp. ultrafast lasers). In this paper, additive manufacturing of Tungsten materials is investigated by using femtosecond (fs) fiber lasers. Various processing conditions are studied, which leads to desired characteristics in terms of morphology, porosity, hardness, microstructural and mechanical properties of the processed components. Fully dense Tungsten part with refined grain and increased hardness was obtained and compared with parts made with different pulse widths and CW laser. The results are evidenced that the fs laser based AM provides more dimensions to modify mechanical properties with controlled heating, rapid melting and cooling rates compared with a CW or long pulsed laser. This can greatly benefit to the make of complicated structures and materials that could not be achieved before.

Keywords: Laser 3D manufacturing; Additive manufacturing; Tungsten; Fiber lasers; Femtosecond fiber lasers; Ultrafast fiber lasers.

1. INTRODUCTION

Laser additive manufacturing 1-7 (e.g., selective laser melting SLM) uses material powders or small parts to build three dimensional parts with complicated structures. It has been proved to be an efficient, robust, and cost effective way for the next generation manufacturing. AM processes for many industrial metals like titanium Ti (~1668°C melting temperature) and aluminum Al (~650°C melting temperature) are well established and many remarkable results have been published ¹⁻⁷, by using Continuous-Wave (CW) or long-pulsed lasers. However, there are several limitations for current laser AM processes in Tungsten 3D manufacturing to battle with challenges such as residual stresses, density, uniformity, and variation in mechanical strengths: 1) CW or long-pulsed lasers can only process materials with low to medium thermal conductivity and melting temperature, due to low peak intensity (\sim kW). Processing high temperature and high thermal conductivity materials such as Tungsten (melting temperature 3422 °C, thermal conductivity 173 $W/(m \cdot K)$ needs much higher power laser to deposit high energy in short period of time against fast thermal drain. 2) Further increasing power of CW or long pulsed lasers will induce thermal diffusion outside the focal volume (i.e., HAZ) and residual stress. That can cause damage (e.g., cracks and fatigue) of the joint part. 3) Formation of unstable intermetallic phases can further degrade the quality of joining and reduce the hardness and strength. 4) Separate post processes are always required to polish, cut, trim, structure the AM components. This will induce extra labor and cost. 5) Current CW laser AM system uses material absorption to bond powders. It can only bond materials that absorb at the laser wavelength.

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Recently, the world's first fs laser AM process was successfully demonstrated for melting and shaping Tungsten powders, Hafnium Diboride (HfB₂), Zirconium Diboride (ZrB₂),⁸⁻¹⁰ and for welding dissimilar metals such as Al alloy, SST, and Mg alloy.¹¹ In this paper, we report our latest research on systematically investigating the capability of fs fiber laser based AM. Various shaped parts were made and density as high as 99% of Tungsten parts was demonstrated. Hardness, microstructure and grain size comparison were carried out by varying pulse width and tuning to CW mode. The fs fiber laser based AM shows a clear path toward fine microstructure making and provides unprecedented way to modify material functions and mechanical properties.

2. FEMTOSECOND FIBER LASER ADDITIVE MANUFACTURING: MECHANISM AND SETUP

The main characteristic of the ultrashort laser pulse is the high peak intensity that results in rapid (ps) delivery of energy into the material (independent of material absorption characteristics) to cause ionization, which is much faster than the plasma expansion (ns to µs), therefore the local temperature is rapidly increased to over 6000°C (controllable through energy and pulse number) and the thermal damages to surroundings are reduced or eliminated.^{12,13} Compared with CW laser AM, the fs laser AM system creates instantaneous high temperature to melt high temperature and high thermal conductivity metals (e.g., Tungsten, melting temperature 3422°C, thermal conductivity 173 W/(m·K)) and form much stronger micro-scale welding/bonding between similar or dissimilar refractory metal powders to form various shapes and sizes.⁸⁻¹⁰ This multi-functional capability (SM and AM, controllable melting temperature and HAZ) will significantly reduce the building time and cost, which is not achievable for CW laser AM. Figure 1 gives a self-explained comparison for AM using CW or ns laser, fs laser (low power), and fs laser (high power).



Figure 1. Comparison of fs laser AM process versus CW or ns laser process

Many parameters can have impact on fs laser AM quality.¹⁻¹¹ The AM process is very complicated and involves many variables related to not only material properties, laser parameters, etc., but also human operation and intervention. In terms of laser parameters, there are energy, pulse width, average power, pulse repetition rate (PRR), peak power, beam quality, focal spot size, hatch, scanning speed and contour, and mode of operation. In terms of powder quality, there are powder size, shape, residual stress, etc. In terms of powder welding dynamics, there are heat flow, chemical reaction,

metal evaporation, thermal diffusion and transfer, and stress and fatigue. In terms of metallurgy, there are melting, solidification and cooling, grain/microstructure formation, phase transformation, cracking, and femtochemistry. Figure 2 gives a simplified evolution diagram to summarize the mechanism of the AM process. The ablation of fs fiber laser incurs ionization and recombination (in ps regime) of materials to form new grains and microstructures during supercooling rate and solidification from a few ps to a few ms.



Figure 2. Temporal evolution of fs laser AM process

The experimental setup has been established based on the existing powder bed AM and SM facilities in the application lab of PolarOnyx (Figure 3). It mainly comprises a fs fiber laser system, beam delivery components, a beam shaper to form desired shapes (flat top, donuts), an automated motion system, a scanning system, a powder delivery system, and a control system. The output pulse width from the mode locked fiber laser is ~750 fs and the PRR is tunable between 100 kHz and 100 MHz. The laser beam wavelength is centered at 1030 nm. The maximum output pulse energy is 500 μ J and average power is 1 kW.



Figure 3. Experimental set up used for 3D AM: (a) Parts and layout of experimental setup. AOM: acoustic optical modulator; M: galvanic mirrors; (b) Sketch of powder bed distribution

3. TUNGSTEN PARTS MAKING AND COMPARISON

3.1. Tungsten Parts Making

By varying process parameters such as fs fiber laser parameters (energy, power, PRR) and scanning speed and pattern, all types of samples can be made with controllable porosity, density (up to 99%), shapes, and structures. Figure 4 shows some samples we made with **Tungsten powders on Tungsten substrates**. Figure 5 gives two examples to show how small the feature size can be done for Tungsten powder with fs fiber laser AM. As small as 10 μ m line feature was achieved. And a small cylinder is made with a side hole as small as 0.3 mm. Complicated parts such as gear were also made in PolarOnyx. Figure 6 shows an example of Tungsten gear AM. The gear is $\frac{1}{2}$ inch in diameter.



Figure 4. Samples of Tungsten parts on Tungsten substrates with various porosity, structure shapes, and density. Thin wall (right photo) has a thickness of 100 micron.



Figure 5. (Left) fs fiber laser AM of Tungsten powders, indicating complete melt of 10 micron size Tungsten powders and recrystallization. (Right) A small side hole is additively made along with a small cylinder.



Figure 6. Tungsten gear AM with fs fiber laser.

3.2 Performance comparison by varying pulse width

Cube is used as testing structure for comparing the performance under different conditions. A series of Tungsten cubes were made by varying the pulse width to be 750 fs, 20 ps, 200 ps, and CW mode, while keeping other AM parameters unchanged. After making the Tungsten cube samples, all of those samples including Tungsten substrate were polished first, then etched by solution (1 g NaOH and 1 g $K_3Fe(CN)_4$ into 10 mL Water) for 3 minutes. After water rinse, alcohol

rinse, and dry, microscopic image (10x) for both sample and substrate surface were taken to measure the grain size. The samples are further polished for taking SEM images. Figure 7 illustrates the convention we used to describe the measurement results.



Figure 7. Convenstion used in interpretation of test results

Comparison between fs laser and CW laser was carried out. <u>Figure 8</u> shows the grain size comparison. Interestingly, the fs fiber laser based AM can manipulate the microstructure and grain size and obtain as much as 4x smaller grain size than that of CW laser.



Microstructure is modified. Top Surface

Figure 8. fs fiber laser AM of Tungsten powders has smaller grain size and controllable micro-structures.



Figure 9. Grain structure comparison for 20 ps (left) and 200 ps (right)

Further detailed comparison has been done by tuning the pulse width to 20 ps and 200 ps while keeping other parameters same. <u>Figure 9</u> gives example of the grain structure photos. Detailed grain structures related to both top and cross section of all samples are given in <u>Figure 10</u> and <u>Figure 11</u>, respectively. Using ImageJ, we calculated the average grain areas of fs, 20ps, 200ps, CW laser AM samples and show the results in Table 1 and Table 2. <u>Figure 12</u> gives a summarized plot illustration for both top surface and cross section. It clearly indicates that by tuning the pulse width, the grain size can be

manipulated. And shorter pulse width produces smaller grain size, which is related to better mechanical strength (inversely proportional to the grainsize¹⁴).



Figure 10. Top surface comparison of Tungsten cube samples. Left: microscopy image (10x); Right: highlight the boundary/outline of the grain size. (From top to bottom: fs sample; 20 ps sample; 200 ps sample, CW sample.)



Figure 11. Cross section comparison of Tungsten cube samples. Left: microscopy image (10x); Right: highlight the boundary/outline of grain size. (From top to bottom: fs sample; 20 ps sample; 200 ps sample, CW sample.)

Table 1. Using ImageJ to calculate the average grain size area (top surface) of fs, 20 ps, 200 ps, CW laser samples.

	fs sample	20 ps sample	200 ps sample	CW sample
Average grain size area (um ²)	564	855	1042	1109

Table 2. Using ImageJ to calculate the average grain size area (cross section) of fs, 20 ps, 200 ps, CW laser samples

	fs sample	20 ps sample	200 ps sample	CW sample
Average grain size area (um ²)	595	700	1011	1135





SEM images of tungsten samples made with fs, 20 ps, 200 ps, and CW laser were taken. The cross section SEM images were shown in <u>Figure 13 -8</u>. It does shows that the fs laser based AM generates less cracks and defects compared with those AM parts made with longer pulsed or CW lasers. This is mainly because the melting rate (and possible cooling rate) induced by higher peak power of fs laser is much higher than long pulsed lasers. Further investigation is needed to see if there are any other factors involved.



Figure 13. Cross section of tungsten sample made by fs laser, from left to right, SEM image of 200x, 1000x, 5000x.



Figure 14. Cross section of tungsten sample made by 20 ps laser, from left to right, SEM image of 200x, 1000x, 5000x.



Figure 15. Cross section of tungsten sample made by 200 ps laser, from left to right, SEM image of 200x, 1000x, 5000x.



Figure 16. Cross section of tungsten sample made by CW laser, from left to right, SEM image of 200x, 1000x, 5000x.

Hardness testing was carried out with the micro hardness testing instrument: Buehler Micromet 2400; Model: 1600-4987 (testing method: 200g load and 10s dwell time). Both the top layer and cross section of tungsten samples made with fs, 20 ps, 200 ps, CW laser, and the tungsten substrate (three samples for each set) were measured and averaged over 5 different locations for each sample and five samples for each case. The data is summarized in Table 3. The hardness number of the fs sample is in general larger than those of long pulsed and CW samples.

Hardness (HRC)	fs sample	20 ps sample	200 ps sample	CW sample	Tungsten substrate
Тор	45.4	44.1	42.4	44.7	44.9
Cross section	47.7	41.8	45.1	44.9	45.8

Table 3. Summary of the hardness testing results for different samples.

4. CONCLUSIONS

In conclusion, we have demonstrated, for the first time, that fs fiber laser are not only able to make 3D parts but also able to modify microstructures and grain sizes to have better overall performance compared with those parts made with long ps lasers and CW lasers. It has a great potential for future laser 3D manufacturing in making high melting temperature materials and complicated structures that could not be achieved before.

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