

Effects of Processing Parameters on the Mechanical Properties of CMSX-4[®] Additively Fabricated through Scanning Laser Epitaxy (SLE)

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The work aims to characterize the effects of processing parameters on the mechanical properties of CMSX-4[®] fabricated using a laser-powder bed fusion (LPBF)-based additive manufacturing (AM) process, scanning laser epitaxy (SLE). The laser power, the scan speed, and the number of repeat scans are varied in SLE. These parameters are combined to define a new measure such as the energy density. Microstructures of the samples are investigated using optical microscopy and scanning electron microscopy. Uniaxial tension tests are performed on samples in longitudinal direction and relevant data is extracted. This work is sponsored by the Office of Naval Research through grant N00014-14-1-0658.

Keywords: Additive Manufacturing, Scanning Laser Epitaxy (SLE), Nickel-base, Superalloys, CMSX-4[®], Mechanical Testing

Introduction

In order to increase the efficiency of gas turbine aero engines, investment cast equiaxed (EQ) turbine component parts are increasingly being replaced with directionally solidified (DS) components that have either columnar or single crystal (SX) microstructures. However, due to prolonged period of operation, these DS blades suffer from damage and wear limiting their lifetime. Such reduction in their effective life necessitates the replacement of the numerous airfoils within the engines. Due to the high cost of producing DS/SX cast turbine blades, the cost of replacing each blade is several thousand dollars. With each engine containing several hundred blades, the cost to replace everyone can reach hundreds of thousands of dollars. Hence, it is of great interest to develop an additive manufacturing (AM)-based process which will restore the SX/DS microstructure at the damaged location allowing the blades to be reused rather than discarded.

Scanning laser epitaxy (SLE) is a laser-based manufacturing process for deposition of EQ, DS, and SX structures onto superalloy substrates through the controlled melting and re-solidification of superalloy powders. In this particular implementation of the process, a laser beam guided by a set of high-speed galvanometer scanners allows for tight control of the amount of energy being applied to the top of the powder bed as well as the speed at which the melt pool moves across the substrate. Under the proper operating conditions and with enough substrate re-melt, the solidification of the powder melt pool will follow the microstructures of the underlying substrate, allowing for directional and even SX growth [1]. The SLE process has shown significant potential for one-step repair of CMSX-4[®] [2-4], René N5 [5, 6], René 142 [7], MAR-M247 [8], René 80 [9], and IN100 [10, 11].

CMSX-4[®] was developed through the joint efforts of Canon-Muskegon and Allison in the 1990s [12]. This “second generation” SX superalloy alloy showed an improvement of 35 °C in the turbine airfoil temperature capability compared to CMSX-2[®] and CMSX-3[®] [13]. CMSX-4[®] contains 3 wt. % of rhenium (Re) similar to other “second generation” SX superalloys. Re was shown to delay coarsening of the γ' phase by partially segregating to the γ matrix [14]. CMSX-4[®] also enjoys solid-solution-strengthening due to the presence of chromium (Cr), tungsten (W), and tantalum (Ta) and precipitation-hardening due to the presence of aluminum (Al) and titanium (Ti) [12].

Current work focuses on establishing an optimal operating range for the various process parameters in SLE. The effects of these different processing parameters on the deposit characteristics are examined. The laser power, the scan speed, and the number of repeat scans are varied in SLE. These parameters are combined to define a new measure such as the energy density. Microstructures of the samples are investigated using optical microscopy and scanning electron microscopy. Uniaxial tension tests are performed on samples in longitudinal direction. The yield strength, ultimate tensile strength, modulus and ductility values are extracted

Experimental

The CMSX-4[®] powder was produced by Praxair Surface Technologies using an atomization process and had a particle size ranging from 85-150 μm . The SLE process was conducted on rectangular investment-cast SX CMSX-4[®] substrates having dimensions of 38.10 mm x 11.85 mm x 2.54 mm. Each substrate was placed into a 38.10 mm x 11.85 mm recess cut into an IN625 base plate. The CMSX-4[®] powder was placed above the substrates using rectangular wells cut into an Aluminum mask plate.

Table I. Chemical composition of the CMSX-4[®] powder (wt. %)

	Cr	Co	Mo	Re	W	Al	Ti	Ta	Ni
CMSX-4 [®]	6.5	9.7	0.4	3.0	6.4	5.6	1.0	6.5	Bal

After the samples were prepared, they were placed into a Terra Universal atmospheric glove box which was then purged with argon. A 1kW Ytterbium fiber laser (IPG Photonics, Model: YLS-1000) was used in conjunction with a Cambridge Technologies galvanometer scanner to focus the beam to a diameter of 40 μm at the top of the substrate. A consistent raster scan pattern across the width of the sample was used to propagate a linear melt pool along the substrate.

In order to facilitate reduced order parameter space, the SLE process parameters namely the laser power, the scan speed, the number of repeat scans, and the scan spacing are combined to define a new measure, the scan energy density (E) [4]. E is defined as follows:

$$E = \frac{P}{SS \times V \times t_p} \quad (1)$$

Here, P is the laser power, N is the number of repeat scans, V is the raster scan speed, SS is the raster scan spacing, and t_p is the powder thickness and.

Microstructural Investigation and Mechanical Testing

Each sample was sectioned along the length and width. Using a Buehler automated disc saw, each sample was first sectioned lengthwise, and then sectioned by multiple widthwise cuts, as shown in Figure 1. Each section was mounted in Bakelite and polished to a mirror finish; starting with 80 grit paper and progressively increasing the size to 1200 grit. The samples were then rough-polished using 5 μm and 3 μm diamond solutions. Finally, the samples were fine polished using a 0.5 μm colloidal alumina suspension. The polished samples were then etched with Marble's reagent (50 ml HCl, 50 ml H₂O, and 10.0 gm CuSO₄) to eliminate the γ' phase and reveal the dendritic microstructure.

A Leica DM6000 optical microscope was thereafter used to take the images. The microstructural investigation of the SLE processed CMSX-4[®] was carried out on a Hitachi SU8230 SEM. Vickers microhardness measurements were carried out using a Buehler microhardness indenter with a fixed load at 2000 gf. The hardness values were extracted in Vickers microhardness scale (HV). Details on hardness measurements may be found elsewhere [11]. The tensile properties of the CMSX-4[®] specimens were determined according to ASTM D638 using an Instron 33R 4466 equipped with 10 kN load cell.

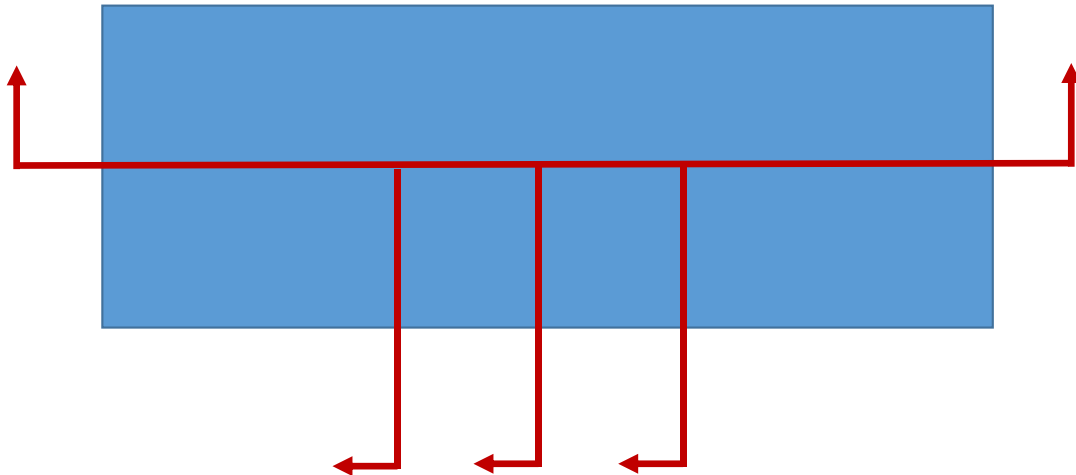


Figure 1. Sections used in metallographic analysis

Results and Discussion

As illustrated in Figure 2 and 3, the longitudinal sections showed a full metallographic bond along the entire length of the samples. The transition from the investment cast CMSX-4[®] substrate to the SLE-deposited material is indicated by the sharp change in the microstructure such as the dendrite size. The dense deposit is free from porosity, hot tearing or stress cracking. The characteristics of the samples illustrated in Figure 2 and 3 are reported in Table II.

Sample 1 was processed with E of 52.73 J/mm³ while Sample 2 was processed with E of 18.75 J/mm³. A previous study on CMSX-4[®] showed that higher values of E showed lower SX ratio

(defined as the ratio of average total height and the average deposit height) [4]. Following the previous results, the current study also showed that Sample 1 had lower SX ratio.

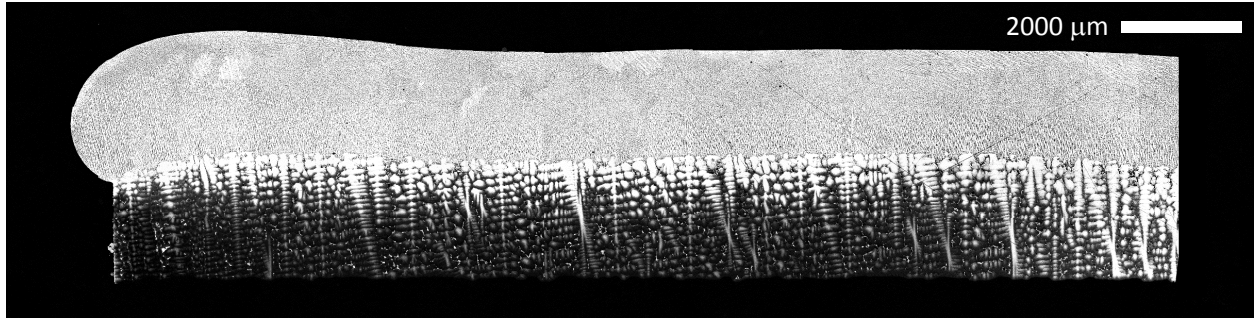


Figure 2. Representative longitudinal cross-section of the as-deposited Sample 1. This sample is processed at $E = 52.73 \text{ J/mm}^3$.

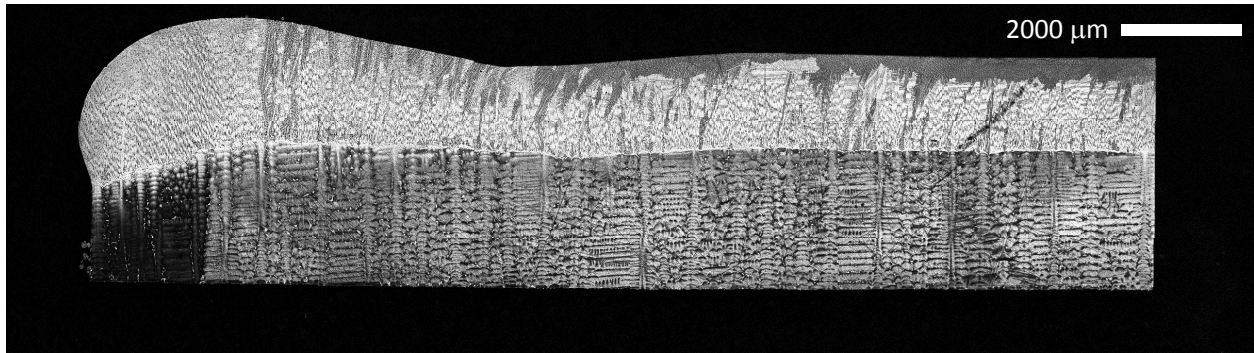


Figure 3. Representative longitudinal cross-section of the as-deposited Sample 2. This sample is processed at $E = 18.75 \text{ J/mm}^3$.

Table I. Characteristics of Sample 1 and Sample 2

Sample #	# of Repeats	Power (W)	Scan Speed (mm/s)	Scan Spacing (μm)	Powder Thickness (mm)	Energy Density (J/mm^3)	SX Ratio
1	600	750	800	12.7	1.4	52.73	0.39
2	400	500	750	25.4	1.4	18.75	0.75

The orientation of the columnar growth in the SLE-deposited material showed little to no angular misorientation as compared to the underlying substrate as shown in Figure 4(a). Figure 4(b) illustrates the transition from SX growth with [001] directionality to a shift in orientation. This change is known as oriented to misoriented transition (OMT). On some samples, the top of the deposit suffered from EQ growth. This is known as the columnar to EQ transition (CET).

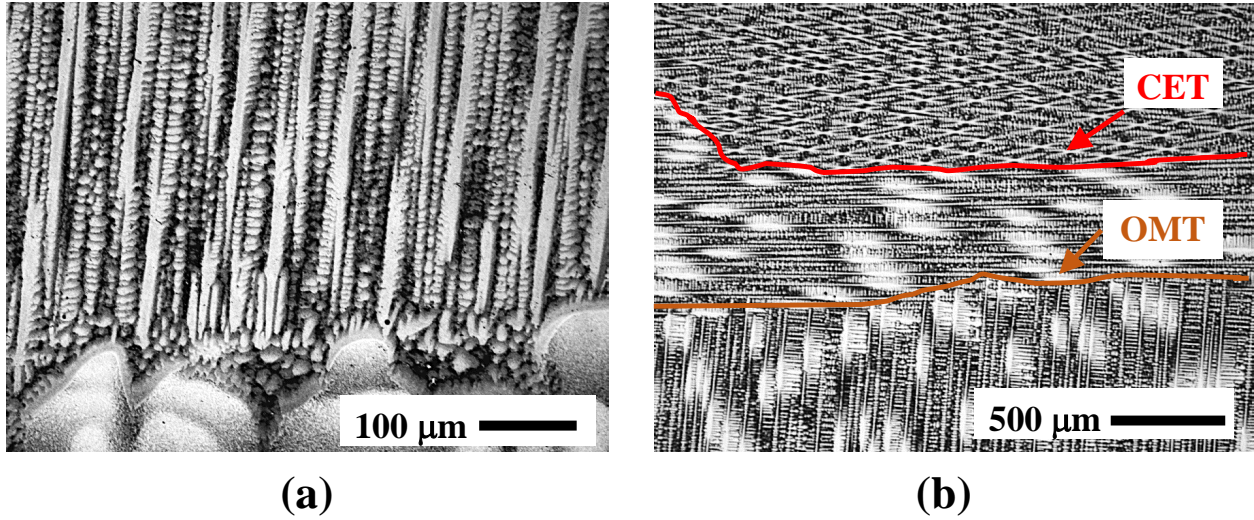


Figure 4. (a) Transition from substrate to deposit and (b) transition regions in the deposit.

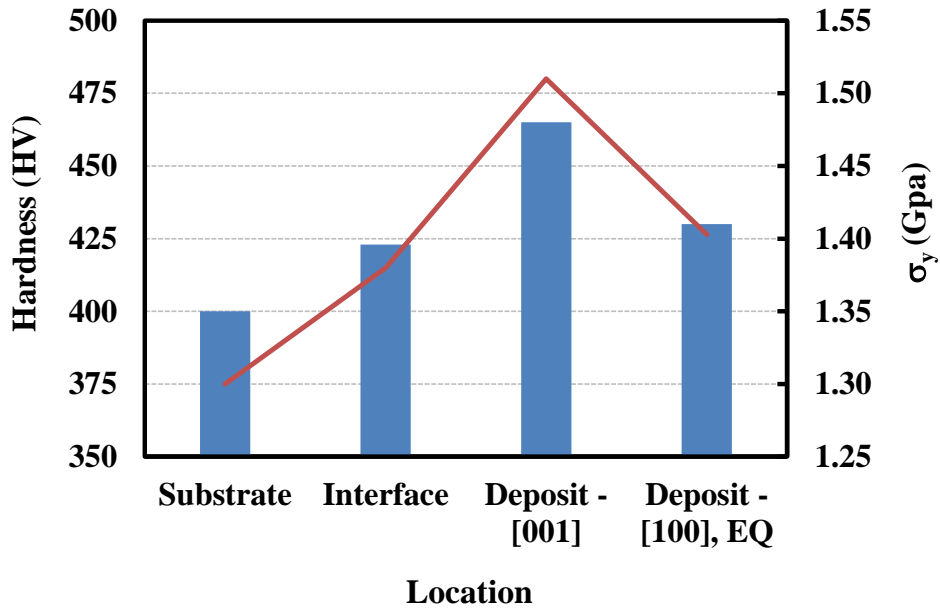


Figure 5. Summary of microhardness results for CMSX-4[®].

In the present study, the [001] columnar region typically showed higher hardness than either the [100] columnar zone or the EQ region. The results indicated a hardness value in the range of 430-500 HV in the deposit region. The substrate region showed hardness values of order 400 HV. Hence, the hardness in the deposit region is about 10% higher than the substrate region. The yield stress was approximated as, $\sigma_y \sim HV/3$ (HV measured in GPa) [8].

Figure 6 shows the load vs. displacement curves for Sample 1 and Sample 2. Sample 2 showed higher values of modulus, lower values of elongation, and higher yield strength as compared to Sample 1 as illustrated in Figure 6. This difference is due to the fact that Sample 2 had higher SX

ratio compared to Sample 1 and therefore, it has higher microhardness. It is noted that the results are presented as load-displacement instead of stress-strain as the cross-section (thickness of the samples was different for the two samples and was varying along the length within the same sample). That is the reason that no absolute values of the modulus or strength are mentioned.

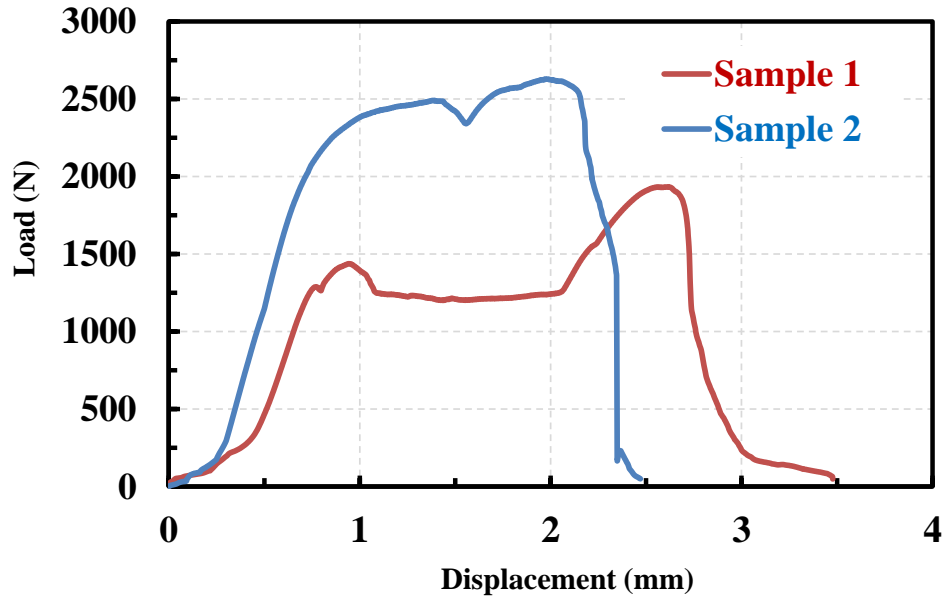


Figure 6: Load vs displacement curves for samples 1 and 2 based on tensile testing

Conclusions

In the present study, two different samples deposited by SLE at two different energy densities were explored. The sample produced at higher values of E showed lower SX ratio and therefore, lower yield strength. It is concluded from the present research that higher SX ratio is desirable for the purpose of obtaining higher yield strength and modulus.

Acknowledgments and Disclosures

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References

- [1] Basak A, Das S. Epitaxy and Microstructure Evolution in Metal Additive Manufacturing. Annual Review of Materials Research 2016;46:125-49.
- [2] Acharya R, Bansal R, Gambone JJ, Das S. A Coupled Thermal, Fluid Flow, and Solidification Model for the Processing of Single-Crystal Alloy CMSX-4 Through Scanning Laser Epitaxy for

- Turbine Engine Hot-Section Component Repair (Part I). Metallurgical and Materials Transactions B 2014;45:2247-61.
- [3] Acharya R, Bansal R, Gambone JJ, Das S. A Microstructure Evolution Model for the Processing of Single-Crystal Alloy CMSX-4 Through Scanning Laser Epitaxy for Turbine Engine Hot-Section Component Repair (Part II). Metallurgical and Materials Transactions B 2014;45:2279-90.
- [4] Basak A, Acharya R, Das S. Additive Manufacturing of Single-Crystal Superalloy CMSX-4 Through Scanning Laser Epitaxy: Computational Modeling, Experimental Process Development, and Process Parameter Optimization. Metallurgical and Materials Transactions A 2016;47:3845-59.
- [5] Basak A, Das S. A Study on the Effects of Substrate Crystallographic Orientation on Microstructural Characteristics of René N5 Processed through Scanning Laser Epitaxy. Proceedings of the 13th International Symposium of Superalloys 2016:1041-9.
- [6] Basak A, Das S. Additive Manufacturing of Nickel-Base Superalloy René N5 through Scanning Laser Epitaxy (SLE) – Material Processing, Microstructures, and Microhardness Properties. Advanced Engineering Materials 2017;19:1600690.
- [7] Basak A, Das S. A Study on the Microstructural Characterization of René 142 Deposited Atop René 125 Processed through Scanning Laser Epitaxy. Materials Science Forum 2016;879:187-92.
- [8] Basak A, Das S. Microstructure of nickel-base superalloy MAR-M247 additively manufactured through scanning laser epitaxy (SLE). Journal of Alloys and Compounds 2017;705:806-16.
- [9] Acharya R, Bansal R, Gambone JJ, Kaplan MA, Fuchs GE, Das S, et al. Additive Manufacturing and Characterization of René 80 Superalloy Processed Through Scanning Laser Epitaxy for Turbine Engine Hot-Section Component Repair. Advanced Engineering Materials 2015;17:942-50.
- [10] Acharya R, Das S. Additive Manufacturing of IN100 Superalloy Through Scanning Laser Epitaxy for Turbine Engine Hot-Section Component Repair: Process Development, Modeling, Microstructural Characterization, and Process Control. Metallurgical and Materials Transactions A 2015;46:3864-75.
- [11] Basak A, Das S. Additive Manufacturing of Nickel-Base Superalloy IN100 through Scanning Laser Epitaxy (SLE). TMS JOM (Submitted).
- [12] Harris K, Erickson G, Sikkenga S, Brentnall W, Aurrecochea J, Kubarych K. Development of the rhenium containing superalloys CMSX-4® & CM 186 LC® for single crystal blade and directionally solidified Vane applications in advanced turbine engines. Superalloys 1992 1992;297.
- [13] Frasier D, Whetstone J, Harris K, Erickson G, Schwer R. Process and alloy optimization for CMSX-4 superalloy single crystal airfoils. High Temperature Materials for Power Engineering 1990:1281-300.
- [14] Giamei A, Pearson D, Anton D. γ/γ' : The Key to Superalloy Behavior. MRS Proceedings: Cambridge Univ Press; 1984. p. 293-308.