Journal of Alloys and Compounds 697 (2017) 174–181

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Experimental optimization of laser additive manufacturing process of single-crystal nickel-base superalloys by a statistical experiment design method

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A R T I C L E I N F O

Article history:
Received 9 July 2016
Received in revised form
17 October 2016
Accepted 8 December 2016
Available online 10 December 2016

Keywords:
Laser processing
Metals and alloys
Microstructure
Orthogonal experiment method
Epitaxial growth

A B S T R A C T

Experimental process optimization is essential to obtain reliable processing conditions prior to performing actual laser additive manufacturing (LAM) of single-crystal (SX) nickel-base superalloys. The influence of processing parameters on deposited productivity and epitaxial SX growth in powder-feeding LAM process was systematically investigated by the orthogonal experiment (OE) method, a statistical experiment design method. This method can rapidly and economically estimate the effect of each processing parameter by a small number of experiments. Resulting relationship between the processing variables and each of deposited productivity and microstructure formation contributes to the selection of detailed processing conditions to balance the factors crucial to successful SX LAM, which means that appropriate adjustment of the processing parameters during actual SX LAM is easy to be performed. On the basis of the analyses of the OE results, a combination of relatively high power, low scanning velocity and moderate powder feeding rate is beneficial to both deposited productivity and epitaxial SX growth during powder-feeding LAM, and allows the preparation of good multilayer SX deposits with fine dendrites.

1. Introduction

Single-crystal (SX) nickel-base superalloys are widely used in turbine engines due to their excellent properties at elevated temperatures [1,2]. Since laser processing, *e.g.*, laser additive manufacturing (LAM), can add controlled amounts of material to required locations with low heat input and high cooling rate [3–8], it has exhibited a significant impact on the precise repair and fabrication of these SX components. Numerous research results on the solidification behavior of the laser-processed SX alloys suggested that successful SX laser processing needs to ensure columnar dendrites epitaxially growing from the SX substrate and suppress the nucleation and growth of equiaxed grains ahead of solid/liquid interface, *i.e.*, columnar-to-equiaxed transition (CET) [9–19]. Although these studies have contributed to the selection of the processing variables, their conclusions were proposed based on modeling methods and almost without considering powder feeding [9–16]. However, systematic experimental research on the processing-microstructure relationship in actual powder-feeding SXLAM process has not yet been reported; some experiments were performed just to verify the accuracy of the models and their simulated results. This may be because the optimization procedure based on traditional experiment design methods requires abundant experiments to study the effect of each processing variable, leading to high optimization cost for Re-containing SX alloys. For instance, experimental design of three factors and three levels requires $3^3 = 27$ sets of experiments using traditional methods. One effective way to reduce the cost of process optimization is to decrease the number of required experiments. For a long time, the orthogonal experiment (OE) method, a statistical experiment design method proposed by Taguchi [20–22], has been widely applied to the optimization of processes and experiments [23–28] because it only requires a small number of experiments, *e.g.*, the OE design of three factors and three levels just requires nine sets of experiments. It was therefore employed in this work to economically understand the complex relationship between the processing parameters and microstructure/deposited productivity.

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http://dx.doi.org/10.1016/j.jallcom.2016.12.109
0925-8388/

After obtaining an appropriate processing window from reported literature or by theoretically analyzing/calculating, this OE design method is useful for the experimental optimization prior to preforming actual SXLAM process. This is because the OE method can rapidly and economically estimate the effect of each variable within the selected processing window on microstructure development by a small number of experiments. Resulting processing-microstructure relationship contributes to performing appropriate adjustment of the processing parameters during actual LAM and preparing good SX deposits. Consequently, the main objective of this study is to (1) apply the OE method to systematically investigate the influence of laser processing parameters on deposited productivity and epitaxial SX growth during powder-feeding LAM of superalloy by a small number of experiments, and (2) optimize SXLAM process according to the OE results.

2. Experimental procedure

LAM experiments were conducted by a 6 kW LAM system with a coaxial powder delivery system and an oxygen content less than 50 ppm. In this work, the powder of a second-generation SX superalloy René N5 was used as the deposited material and a first-generation SX superalloy SRR99 was chosen as the substrate. The nominal compositions of the René N5 and SRR99 SX alloys are Ni−6.2Al−7Cr−8Co−2Mo−7Ta−3Re−5W−0.2Hf and Ni−5.5Al−8Cr−5Co−3Ta−2.2Ti−10W (in *wt.* %), respectively. All the substrates were machined from conventional DS SX cast ingots with the [001] orientation normal to the deposited surfaces. For all experiments the substrate surfaces were ground with 600-grit SiC paper and cleaned in methanol prior to LAM.

Deposited productivity and epitaxial SX growth during LAM are affected by various processing parameters including laser power P, scanning velocity V_b, beam diameter D_b, and powder feeding rate m. However, D_b maintains generally constant and Gäumann et al. [11] found the effect of D_b is weaker than that of P and V_b. Therefore, the effects of three supreme parameters, *i.e.*, P, V_b and m, on deposited productivity and epitaxial SX growth were systematically investigated and a constant D_b of 2 mm was used. Each parameter was designed with three levels to avoid nonlinear (or non-monotonic) effects and an $L_9(3^4)$ OE array (Table 1) was employed to provide the minimum numbers of required experiments [20–22]. The microstructure was characterized by a Leica DM4000 optical microscope (OM) to evaluate the optimization degree. Each index value was measured at least three replications. After analyzing the OE results, a thin-wall SX specimen was prepared by LAM process using a set of optimized processing parameters (P: 1000–2000W, V_b: 10 mm/s and m: 14 g/min) to verify the conclusions of the OE analyses.

3. Results and discussion

Fig. 1 shows the transverse-section microstructure of a laser cladding, *i.e.*, a single-layer laser deposit, as an example. The dendrites epitaxially grow from the SX substrate along the [001] crystallographic orientation. When growing close to the top, the columnar epitaxial growth is replaced by the equiaxed growth. These dominating solidification features are similar to previous research on laser remelting [11–15] as well as LAM [17–19]. In addition, several geometric dimensions crucial to deposited productivity and epitaxial crystal quality of SXLAM process (including the width W, deposited height H_D, remelted height H_R, and height of epitaxial growth H_E of the laser claddings) are marked in Fig. 1 as the quantified indices to systematically compare the optimization degrees.

Table 1
Orthogonal experiment array of the laser processing parameters (laser power, P, scanning velocity, V_b, and powder feeding rate, m).

No.	P (W)	V_b (mm/s)	m (g/min)
A	500	10	12
B	500	15	16
C	500	25	24
D	1000	10	24
E	1000	15	12
F	1000	25	16
G	1500	10	16
H	1500	15	24
I	1500	25	12

It should be noted that successful SXLAM depends primarily on the control of the dendritic growth pattern. Additionally, the crystal growth patterns of all of the laser claddings are similar, and the difference lies in the values of these quantified indices. Therefore, the microstructural analyses in this work emphasize on the comparison of the values of these indices crucial to successful SXLAM, rather than the explanation of the issues that have been widely discussed such as dendrites how to epitaxially grow or stray grains how to nucleate and grow.

3.1. Influence of laser processing parameters on deposited productivity

To improve deposited productivity and epitaxial growth during SXLAM, the effects of many indices, which may be nonlinear or even non-monotonic, should be carefully considered. Therefore, a method based on the analysis of range and trend was used to rapidly and visually analyze the OE results. Such a method is able to simplify the analysis procedure and conveniently obtain an optimized processing combination that can balance each index. As shown in Fig. 2a, an increase in P significantly increases W whereas increased V_b and m reduce W. This is because the width of the laser claddings depends mostly upon the total heat input of the laser track. In other words, the changes in the processing parameters reducing the total heat input can narrow down the cladding width.

Similarly, the variation trends of H_D and H_R with P and V_b are similar to those of W (see Fig. 2b). Therefore, an increased P and/or a decreased V_b that enhance the heat input always enlarge the size of the laser claddings, *i.e.*, deposited productivity. Different from P and V_b, the influence of m on the total heights of the claddings, *i.e.*, $H_D + H_R$, is invisible though an increased m can heighten H_D and simultaneously lower H_R. These observations confirm again that the cladding geometry is primarily determined by the total heat input. Change to m can only affect the distribution of energy between substrate and deposit, and does not visibly vary the total heights of the claddings. Therefore an increase in m exhibits a visible decrease in the dilution ratio, R_D. In comparison, the influences of P and V_b on R_D are relatively weak. Although low P and V_b are able to decline R_D, the further increased P will not significantly increase R_D (see Fig. 2c).

In Fig. 2d, the influence of the processing parameters on the powder using efficiency, η, can be clearly observed. Firstly, an increase in P improves η, and this effect weaken with further increasing P, which implies that η can only be improved limitedly by simply increasing P. Secondly, a decrease in V_b can effectively raise η especially for relatively low velocities, which means that a low V_b always improves deposited productivity. Moreover, a high m can prominently increase η and hence enhance deposited productivity.