Phase Transformation Temperature Analysis in the Additive Manufacturing of Nickel-Titanium Shape Memory Alloys

Introduction

Nickel Titanium (NiTi) is a Shape Memory Alloy (SMA) which means that it has the unique property of Shape Memory; the ability to remember its original shape after becoming deformed [1]. Unlike other SMAs, NiTi is biocompatible and resistant to corrosion; these additional properties have made it popu-



lar across many industrial sectors, including the biomedical and aerospace industries such as implants, actuators, etc. [2].

The manufacturing process of NiTi is difficult, this has limited its application to simple shapes such as wires, sheets, and foils [3]. Conventional manufacturing processes require tight control due to NiTi's sensitive atomic composition [1]. Changes in the atomic composition can lead to shifts in the phase transformation temperatures which are responsible for the shape memory effect [5].

The rise of Additive Manufacturing (AM) technologies offers additional methods to manufacture NiTi. Selective Laser Melting (SLM) is an AM method that offers the best control of process parameters [6]. By modelling the phase transformation temperatures as a function of process parameters, it is possible to create a response surface model which will provide a reasonable approximation of the phase transformation temperatures of NiTi.

Methods & Materials

NiTi Powder Preparation

NiTi Composition – 50.9% Ni Average Particle Size – 19.7µm Maximum Particle Size - 45µm



Differential Scanning Calorimetry

This technique is used to determine the phase transformation temperatures of the NiTi sample by heating and cooling the sample to two extreme temperatures.

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Edgar Palapa
               Faculty Advisor: Dr. Alaa Elwany
            Graduate Student Advisor: Bing Zhang
Department of Industrial & Systems Engineering, Texas A&M University
               101 Bizzell St, College Station, TX 77843
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Design of Experiments

A 2⁵⁻¹ fractional factorial design of experiments (DoE) was used to minimize the number of runs while maximizing the number of interactions between all factors. By doing a fractional factorial DoE, the number of runs required is reduced from 32 runs to only 16 runs.

Runs	p [W]	v [mm/s]	h [mm]	t [mm]	no. passes	As [°C]	Af [°C]	Ms [°C]	Mf [°C]
1	35	80	0.035	0.030	1	54	78	62	-24
2	50	80	0.035	0.030	2	79	91	60	50
3	35	160	0.035	0.030	2	53	77	64	-9
4	50	160	0.035	0.030	1	71	90	62	32
5	35	80	0.120	0.030	2	52	83	64	-49
6	50	80	0.120	0.030	1	36	80	61	5
7	35	160	0.120	0.030	1	-61	4	-3	-62
8	50	160	0.120	0.030	2	28	73	54	-7
9	35	80	0.035	0.050	2	59	83	63	12
10	50	80	0.035	0.050	1	71	85	62	32
11	35	160	0.035	0.050	1	-63	38	21	-54
12	50	160	0.035	0.050	2	65	80	64	12
13	35	80	0.120	0.050	1	-71	24	1	-105
14	50	80	0.120	0.050	2	65	82	64	26
15	35	160	0.120	0.050	2	-58	-1	2	-52
16	50	160	0.120	0.050	1	-56	46	28	-53

Table 2.– Fractional Factorial DoE Results

Results

Linear Regression Model

The data was fit into four linear regression models using MATLAB. Interactions terms were either added or removed depending on the Root Mean Square Error (RMSE), p-value, R, and adjusted R^2 values. Below is one of the four results along with the residual plots that determine the fitness of the data.

 $A_s = 203.34 + 0.35276(v) - 822.91(h) - 8878(t) - 3.3824(v)(h) + 139.83(p)(t) - 16.562(v)(t)$ -1.5479(p)(no. passes) + 374.1(h)(no. passes) + 2031.9(t)(no. passes)





Response Surface Model

The lack of center points in the DoE made it difficult to fit a response surface model to the responses. A fractional factorial design is limited to linear models which makes it difficult to detect curvature in the surface response model.

SLM offers a better control of the NiTi shape memory effect and for this reason, a response surface model of the phase transformation temperatures as a function of process parameters would facilitate the optimization of laser settings for the desired shape memory behavior. A fractional factorial DoE was used to conduct the experimental runs to minimize the number of runs and NiTi powder usage. A linear regression model fit to the collected data before attempting to plot the response surface. The RMSE, pvalues, F-statistic, R^2 , and adjusted R^2 values of all the phase transformation temperatures indicate that there is a good fit between the data and the regression models. However, the residuals plot did not follow a normal distribution for the martensite start temperature. Therefore, only the austenite start, austenite finish, and martensite finish temperature models are good fit for the data. The absence of center points and additional levels in the DoE made fitting a surface response plot to the data unfeasible. A response surface model may have been feasible through the use of multi-level DoEs such as Box Behnken or Central Composite designs.

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Conclusion

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