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A FACTORIAL DESIGN STUDY OF AGEING HEAT TREATMENT INFLUENCE ON PHASE TRANSFORMATION OF Ni-44,8wt%Ti ALLOY

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Abstract. *Shape memory alloys (SMAs) represent a unique class of materials that undergo a reversible phase transformation (martensitic transformation) allowing these materials to display dramatic pseudoelastic stress-induced deformations and shape memory temperature-induced deformations that are recoverable. Among the known shape memory alloys, NiTi is the most commonly used because of its excellent mechanical properties, corrosion resistance and biocompatibility. This work studied the influence of two parameters of heat treatment (temperature and time) on martensite phase transformation temperature (M_S) in a Ni-Ti (48,8 wt % Ti) shape memory alloy, using a factorial design (2^2). The aim of our research was to establish a mathematic model of the technological process, useful for controlling of martensite phase transformation temperature. The two factors, temperature and time, have an important influence on M_S.*

Keywords*: Ni-Ti alloy, Shape memory, Factorial design.*

1. INTRODUCTION

In recent years shape memory alloys (SMAs) have been recognized as effective and promising means for application in various branches of engineering, e.g. space technology, medicine, robotics and actuator technology. For the usage of SMAs in thermosensitive devices, it is very important to increase the speed of response and the sensitivity of shape memory elements, as well as to widen essentially the application fields of thermosensors through their miniaturization. Hence, there is a trend to very small-dimensioned shape memory elements, which could be used as microactuators. One of the perspective materials for this purpose is SMA rapid solidified ribbon (Mehrabi *et al*., 2012)

In spite of more than 40 years of researches regarding SMA, many investigations are still necessary. The mathematical design of experiments, combined with more accurate investigations are important in order to get a complete and clear understanding of SMA behavior. There is a huge amount of literature about TiNi SMA, but it can be observed a lack of using mathematical design of experiments, hence a poor data regarding applications that request high precision (Cheng *et al*., 2012).

In this paper the authors use a factorial design (2^2) for studying the influence of ageing heat treatment on start martensite (M_S) phase transformation temperature of TiNi SMA (44,8wt%Ti).

In general, factorial experiments are designed such that a range of variables (factors) at a fixed number of 'levels' are compared in all possible combinations. The simplest forms these can take are those performed at two levels, i.e. where each variable is set at two different values. Designs of this type have a number of advantages:
1. They can estimate the main effects and interaction effects of several variables simultaneously;
2. Using estimates of variance and noise effects, the significance of each effect and interaction may be estimated and subsequently compared.

The full methodology of this type of experimental design and associated analysis may be found in various books dedicated to the subject (Box *et al*., 1978).

The initial purpose of our research was to determine suitable heat treatment parameters for controlling the initial phase transformation temperature (M_S) as an important future application in microactuators. The results can be used in many other applications (e.g. self-expandable stents), and the method is useful for studying the behavior of other types of SMA.

2. EXPERIMENTAL PROCEDURE

The 48,8wt%Ti ingot with 19 mm in diameter by 100 mm long was prepared using the conventional vacuum arc-remelting method. High purity Ti and Ni raw materials were repeatedly melted six times in an argon atmosphere for homogenization. Then they were cut into small pieces, each of which has a weight in the range of 10 to 30 grams to use in melting spinning. The melt-spun ribbons were produced under a 200 mbar Ar atmosphere using quartz-glass crucibles with a nozzle diameter of 0.9 mm, coated internally with Y_2O_3. By applying an Ar overpressure of ~90 mbar within the crucible, the melt was ejected onto the surface of a polished Cu wheel (200 mm diameter) having a circumferential wheel speed of 50 m/s. The distance between the nozzle and the wheel surface was from 0.6 to 2 mm.

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The final ribbons of the melt-spinning process have 41 μm in thickness and 1 mm in width. Subsequent heat treatment was carried in the ribbons.

In the present work, two factors (temperature and time) were analyzed at two levels. Thus, 4 experiments with three center points could be used to fully explore all possible combinations. The actual variables and levels associated with each sign are contained in Tab. 1.

A 2^2 complete factorial design plus central point (three replicates) was performed in order to evaluate the influence of time and temperature on martensite phase transformation temperature (M_S). Tab. 2 presents the set values of the evaluated parameters at high, medium, and low levels. These levels are presented in tab. 2 as +1, 0, and –1, respectively. We simultaneously varied parameters between a low and a high level in order to obtain all possible combinations. Four (2^2) different samples were prepared to investigate the experimental domain. In addition, three samples with medium level parameters were prepared in order to compare the experimental and the theoretical mean values and to check the model validity. A total of 7 experiments were performed during the optimization.

Table 1. Processing and operational variable levels.

LEVEL	TEMPERATURE (^{O}C)	TIME (h)
-	350	1
0	400	3
+	450	5

Table 2. Experimental design matrix.

RUN	TEMPERATURE (^{o}C)	TIME (h)	TEMPERATURE x TIME
1	-	-	+
2	+	-	-
3	-	+	-
4	+	+	+
5	0	0	0
6	0	0	0
7	0	0	0

By arranging Tab. 2 in this order, it is possible to analyse the significance of an individual variable on any particular response (martensite phase transformation temperature). The main effect of each variable is calculated from the difference in the high level $< Y^+ >$ and the low level $< Y >$ averages.

$$\text{Main effect} = < Y^+ > - < Y^- > \quad (1)$$

The significance of each main effect is then expressed as a percentage of the overall average response from all 4 tests, i.e.:

$$\text{Significance (\%)} = (\text{Main effect}/\text{Overall mean response}) \times 100 \quad (2)$$

where, the Overall mean average for a particular response (M_S) is the sum of that response for all 4 samples divided by 4. If the calculated percentage is greater than the experimental error, the effect of the variable is considered to be significant. An advantage of arranging the table in this standard form is that it also allows interaction effects between variables to be analyzed in a similar manner to individual main effects. The interactions can be obtained by simply multiplying the signs of the respective variables to obtain a secondary column. Thus, the column for the interaction between Temperature (T) and Time (t) is obtained by multiplying the signs in their respective columns. The significance of the interaction can then be calculated as before.

An empirical mathematical model was built using Statistica 7.0 software. The data were modeled in a linear equation, and the results were evaluated by analysis of variance. Response surfaces were built in the optimized condition.

Transformation temperatures of ribbons were determined by differential scanning calorimetry (DSC).

3. RESULTS AND DISCUSSION

The experiments were carried out randomly. The property measured for TiNi (48,8wt%Ti) heat-treated ribbon was M_S temperature. The values of the initial temperature of Martensitic transformation were obtained from each experimental condition shown in Tab. 3.

Table 3. Experimental design matrix with natural values of independent variables and the measured values of transformation temperature (M_S).

RUN	TEMPERATURE (°C)	TIME (h)	TEMPERATURE x TIME	M_S
1	-	-	+	31,7
2	+	-	-	33,5
3	-	+	-	33,9
4	+	+	+	38,8
5	0	0	0	34,3
6	0	0	0	34,0
7	0	0	0	34,1

The calculated factor influences, factor interactions are shown in Tab. 4. Results indicate that all variables, including the interaction temperature x time, had influence on the value of the initial temperature of martensitic transformation (M_S).

Table 4. Estimate of the variables in the initial temperature of martensitic transformation.

FACTOR	VARIABLES EFFECT	p FACTOR (95%)
AVERAGE	34,33	0,000
(T) TEMPERATURE (°C)	3,35	0,001348
(t) TIME (h)	3,75	0,000966
T*t	1,55	0,012421

The variation of M_S temperature (tab. 4) can be expressed by Eq. (3):

$$M_S = 34,33 + 1,68.T + 1,88.t + 0,78.T.t \quad (3)$$

where "M_S" is martensite transformation initial temperature, "T" is heat treatment temperature, and "t" is heat treatment time. The above equation was calculated using the linear method.

If we chose T= 350 ºC and t= 60 minutes (1h) and replace in Eq. (3) we'll get M_S = 31,55 ºC. The result was checked using a sample made of same alloy, same dimensions, and the experimental result determined by DSC method was M_S = 31,70 (Tab. 3).

The three-dimensional response surface plots for M_S (Fig. 1). The temperature M_S increase with the increasing of the temperature and time heat treatment. It is observed in Fig. 1 that to increase M_S is necessary to conduct experiments with higher temperatures and times, and to decrease M_S is necessary to conduct experiments with smaller times and temperatures. In some cases the M_S was less than the temperature of the human body. In this case the thermal treatments are suitable for use in stents. In the event that the M_S was greater than the temperature of the human body, the most appropriate application would be in microsensors and mcroatuadores.

The temperature M_S increase with the increasing of the temperature and time heat treatment is supposed to appear as a result of increasing of the precipitates amount and decreasing of Ni content in the matrix phase. The amount of precipitates increases with increasing of ageing temperature, but this will lead to a decreasing of Ni content in the matrix phase, hence the chnages of Af temperature. The heat treatment is based on phase transformations and precipitation of new phases (Otsuka and Ren, 2002; Vojtˇech *et al.*, 2011; Zheng *et al.*, 2013). According with the binary phase diagram equilibrium (Otsuka and Ren, 2002) and the experimental results (Otsuka and Ren, 2002; Batalud 2005), the heat treatments have a significant influence over the physical and mechanical characteristics of TiNi shape memory alloys with more than 50.5 wt. % Ni. As a result, the properties of TiNi can be fine-tuned for obtaining the designed physical and mechanical characteristics.

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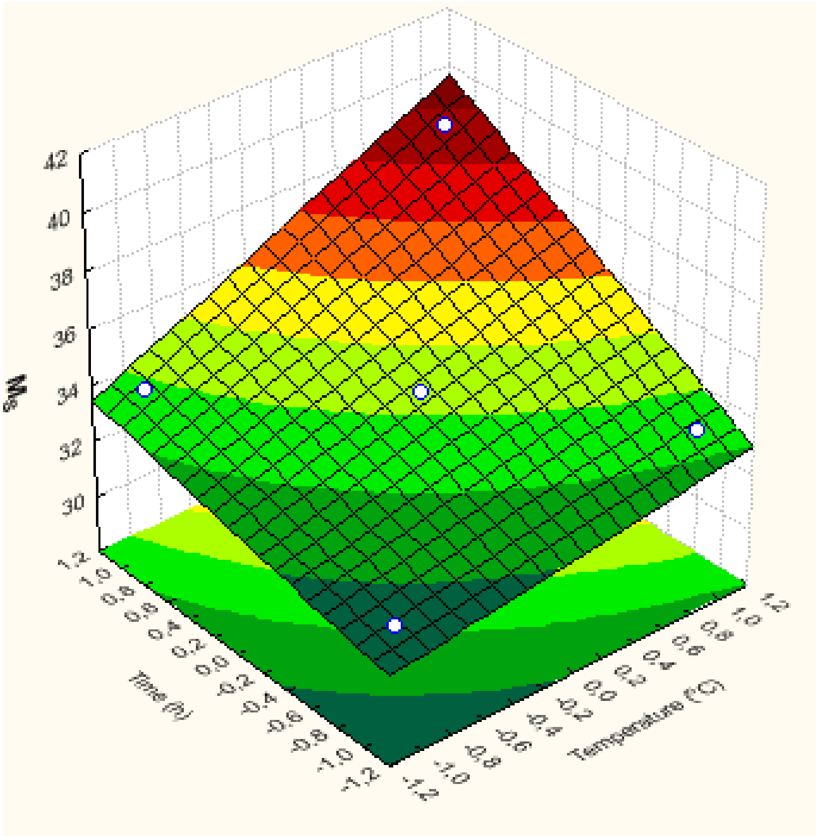

Figure 1. Response surface plot showing effect of the temperature and time in M_S.

The analysis of variance (ANOVA) was performed. ANOVA was an important statistical analysis and diagnostic tool which helped us to determine the statistical significance of the regression. Results of analysis of variance were summarized in Tab. 5. They indicated that the $F_{\text{-ratio}}$ = 103,6 was neatly higher than the critical value $F_{0,05}$ (3; 3) = 9,28 and this proved the satisfactory quality of the model. Moreover, the square of correlation coefficient (R^2) for the model computed as:

$$R^2 = \frac{\text{sum of squares attribueted to the regression}}{\text{total sum of squares}} \tag{4}$$

and equal to 0.99 implied that 99% of the variability in the data for each response were explained by the models.

Table 5. ANOVA results (optimization design) for M_S response.

SOURCE	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	$F_{\text{-RATIO}}$	$F_{0,05}$	R^2
Regression	27,68	3	9,23	103,6	9,28	0,99
Residual	0,27	3	0,09	-	-	
Total	27,93	6	-	-	-	

4. CONCLUSIONS

The results of this study are concluded as follows:

(i) In our research we studied the effect of two factors of influence on M_S phase transformation temperatures. A three level factorial design (2^2) plus central point (three replicates) was used to observe the influence of heat treatment parameters on M_S;

(ii) M_S depends on heat treatment temperature and time. The ageing temperature and time are proper for controlling the M_S temperature. The parameters for obtaining a certain M_S temperature, according with the application's requirements can be easily determined using Eq. (3);

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(iii) Equation (3), determined by using the two level factorial design plus central point (three replicates), is a powerful instrument for controlling with a good precision the temperature;
(iv) More tests accompanied by a study of microstructure and phase transformation are necessary for a reasonable explanation of the occurrence of a maximum on ageing temperature and time versus M_S variation surface;
(v) The paper provides an example of how to use the design of experiment for analyzing the influence of two factors on characteristics of a material.

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6. RESPONSIBILITY NOTICE

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