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ScienceDirect
Computational Materials Science 40 (2007) 359–365
COMPUTATIONAL MATERIALS SCIENCE
www.elsevier.com/locate/commatsci

Prediction of the effect of vacuum sintering conditions on porosity and hardness of porous NiTi shape memory alloy using ANFIS

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Received 4 June 2006; received in revised form 8 October 2006; accepted 22 January 2007
Available online 13 March 2007

Abstract

A neuro-fuzzy model was utilized to predict the hardness and porosity of NiTi shape memory alloy produced by vacuum sintering of powder mixture. Compaction pressure, sintering time and sintering temperature were chosen as input nodes. This procedure allowed successful prediction of porosity and hardness of the NiTi SMA samples. Absolute relative errors were at most 6.3% for hardness and 4.8% for porosity. Mean relative values were 3.4% for hardness and 3.3% for porosity. Results showed that the increasing of the values of input parameters affected outputs, linearly. The most significant parameters influencing the porosity content and the hardness of the under-vacuum combustion-synthesized NiTi specimens were sintering temperature and compaction pressure.

Keywords: Fuzzy clustering; ANFIS method; Powder metallurgy; Combustion synthesis; Sintering; NiTi; SMA

1. Introduction

NiTi shape memory alloy (SMA) has excellent sensing–actuating property [1], acceptable biocompatibility [2] and admissible corrosion resistance [3]. Porous NiTi has newly been introduced as a biomaterial with great applications since promising osteointegration behavior has especially been observed with it [4]. An important feature of NiTi is its delicate response to changes of temperature and mechanical load [5,6]. These variations are due to thermoelastic B2 to B19′ [7,8] and as stress induced austenite to martensite (A → M) phase change [8]. Temperature-dependent first-order displacive transformations of higher symmetry NiTi cubic crystals to lower symmetry rhombohedral, tetragonal, orthorhombic or monoclinic phases result in one-way, two-way and all round shape memory behavior [9]. Previous authors have mathematically modeled response of superelastic NiTi porous samples [9–11]. These researchers have proposed nano-mechanical atomic potential models to find the role of temperature on perfect crystal equilibrium paths in a stress-free bi-atomic alloy system [9–11]. Rapid strain variations across the A–M interface are reported in the nano-sized NiTi grains [12].

Traditional vacuum induction melting (VIM) accompanied with ingot casting has previously been used to produce NiTi objects [13]. Formation of intermetallic nonequilibrium phases together with oxide, nitride and carbide impurities embedded within the system is the matter of much concern [14–16]. Many investigators have focused on combustion synthesis (CS or SHS) as well as powder metallurgy (PM) for production of bulk NiTi specimens [17–24]. Comprehensive information concerning the effects of powder metallurgical procedures on properties of the NiTi SMA samples is still needed for further development of the practical applications. Significant interest exists, for example, on liquid phase sintering for production of porous NiTi objects [25,26]. Large dimensional change and low packing density after sintering are two drawbacks that need to be considered during liquid phase sintering [26]. Fixtured sintering has

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0927-0256/$ - see front matter © 2007 Published by Elsevier B.V.
doi:10.1016/j.commatsci.2007.01.007

previously been proposed as a means of reducing the dimensional change of the sintered porous NiTi objects [25]. The improvements obtained in this method can help near-net-shape technology via powder metallurgy that overcomes the traditional casting problems.

Porous NiTi implants appear to allow significant cranial bone ingrowth and transportation of body fluids through its interconnected pores after appropriate time. The general muscle and bone reaction to porous NiTi were evaluated by Rhalmi et al. [27]. They reported excellent bone remodeling phenomenon characterized by osteoclastic and osteoblastic activity in the cortex. As a successful implant material, the basic demand is that NiTi SMA must have adequate biomechanical properties such as hardness, tensile, compression, fatigue and torsion strength and wear resistance. Various researchers investigated the mechanical properties of the porous NiTi SMA which can be easily adjusted to match those of replaced hard tissues by obtaining different porosity contents through controlling the synthesis conditions. Krone et al. [20] compared the mechanical behavior of porous NiTi parts fabricated by powder metallurgical methods with those prepared by common melt processes. Chu et al. [28] investigated the effect of heat treatment on the microstructure and mechanical properties of porous NiTi SMA fabricated by self-propagating high-temperature synthesis and found that the subsequent aging treatment after solution treatment can lead to the precipitation of the discrete Ni_4Ti_3 phase in NiTi matrix grains, which increase the brittleness of porous NiTi SMA. Sadrnezhaad et al. [25,26] investigated the effects of compaction pressure and sintering conditions on hardness, dimensional change, porosity, and morphology of micro and nano-crystalline NiTi intermetallic alloy and concluded that suitable thermoelastic and superelastic effects are achievable via appropriate selection of fixtured sintering conditions. So simultaneously the desired porosity content and the resulted mechanical properties such as indentation hardness should be considered to achieve the perfect biomedical characteristics of the NiTi implants. That is why finding the optimum sintering conditions in order to obtain the maximum hardness and the critical value of porosity content has been the aim of the present investigation.

Artificial neural networks (ANNs) are flexible modeling tools with capabilities of learning the mathematical mapping between input and output variables of nonlinear systems. They have recently been used for property prediction in materials systems [29–32]. Fuzzy Inference System (FIS), which is based on expertise expressed in terms of 'IF–THEN' rules, can thus be employed to predict the behavior of many uncertain systems. FIS advantage is that it does not require knowledge of the underlying physical process as a precondition for its application.

The purpose of this study is to determine the accuracy of ANFIS method which is a combination of ANN and FIS for estimation of porosity and hardness of the produced NiTi samples after compaction and fixtured sintering of the metallic powders. The mathematical approach employed here can be well devised for flattening of the future further developments in manufacturing of NiTi shape memory implants with acceptable mechanical and biomedical characteristics.

2. Experimental procedure

Commercially pure elemental powders of titanium (~50 μm size and 99.9% purity) and nickel (~9 μm size and 99.6% purity) – both purchased from Merck Inc. of Germany – were blended in a Turbula mixer with a 50.0 at.%Ti and 50.0 at.%Ni ratio for up to 2 h. No attempt was made to eliminate surface oxides from the elemental powders. All samples were stored in evacuated containers to prevent their oxidation.

Cylindrical pills of 1.5 cm diameter and around 0.2 cm thickness were produced by bi-axial cold pressing of the as-mixed nickel–titanium powders into stainless steel molds. A hydraulic press was used to apply different pressures (400, 500 and 600 MPa) on the powder samples. The pills were placed inside an accurately machined fixture, made of DIN 1.4821 heat resistant steel. They were sintered for 2, 3, 4 and 5 h at 950, 1000 and 1050 °C and a vacuum pressure of lower than 10 torr. Schematic of the fixture are given in Fig. 1. Use of the fixture diminishes both the rates of axial and radial changes of the specimens [25]. Fixtured sintering can only reduce the dimensional changes of the specimens to some extent. A small change is therefore observed in dimensions of the samples with dimensional limitations imposed by a fixture. Imperfect seating of the fixed pins plus gradual wearing of the container with time could also result in a slight increase in dimensions of the samples.

Size, shape and distribution of the Ti and Ni particles affect on porosity, density and mechanical properties of

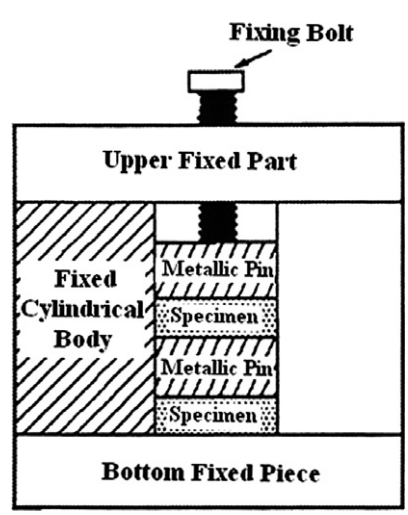

Fig. 1. Schematic side view representation of the fixture used for dimensional stability of Ni–Ti compacted sample during sintering.

the sintered samples. Uniform distribution combined with the differences in size and shapes of the metallic powders appropriates the formation of a condensed tablet suitable for subsequent sintering operation. An Olympus optical microscope was used to observe the microstructure of the sintered specimens. Densities of the sintered samples were measured with a Sartorius-CP324S densitometer, using Archimedes' principle. Fractional porosity, P, defined as the ratio of pore volume to the total volume can be determined as

$$P = \left(1 - \frac{\rho}{\rho_{\text{th}}}\right) \times 100\% \tag{1}$$

where ρ is the density of the porous material and ρ_{th} is the theoretical density of the corresponding bulk material (6.45 g/cm^3 for Ti–50 at.% Ni alloy [26]).

The surfaces of the samples were ground with 1200 grit silicon paper. Samples were then characterized by X-ray diffraction (XRD), Vickers hardness measurement and differential scanning calorimetry (DSC) tests and effects of compaction pressure, sintering time and sintering temperature were determined. The Vickers hardness was measured using an ERNST hardness tester (with using a force of 49 N). The indentation hardness tests were performed on the sintered specimens for three times and the averages were used for analysis. Most of the results were reported in the previous papers [24–26]. New results were included for assessment of the ANFIS model described here. The mathematical approach employed here to help prediction of the sintered NiTi behavior is devised for flattening of the future further developments.

3. Fuzzy inference system

Fuzzy logic starts with the concept of a fuzzy set. Fuzzy set theory has been developed for modeling complex systems in uncertain and imprecise environment. A fuzzy set is an extension of a classical set whose elements may partially belong to that set. Suppose X is the universe of discourse (input space) and its elements are denoted by x, then a fuzzy set A in X is defined as a set of ordered pairs:

$$A = \{x, \mu_A(x) | x \in X\} \tag{2}$$

where $\mu_A(x)$ is called the membership function (MF) of x in A. MF is a function that defines how each element x in the input space is mapped to a membership value (or degree of membership) between 0 and 1.

One of the most useful tools presented within the context of fuzzy set theory to deal with nonlinear, but ill-defined, mapping of input variables to some output ones is what is known as fuzzy inference system (FIS). FIS is a framework, which simulates the behavior of a given system as IF–THEN rules through knowledge of experts or past available data of the system. It is a process of how to map a set of given input variables to an output variable using fuzzy logic. A fuzzy inference system is composed of five functional blocks:

1. A rule base containing a number of fuzzy IF–THEN rules.
2. A database which defines the membership functions of the fuzzy sets used in the fuzzy rules.
3. A decision making unit which performs the inference operations on the rules.
4. A fuzzification inference which transforms the crisp inputs into degree of match with linguistic values.
5. A defuzzification interface which transforms the fuzzy results of the inference into a crisp output.

A rule base and a database are jointly referred usually to as a knowledge base. A fuzzy IF–THEN rule involves generally of two parts. The first part is IF. The second is THEN. These are called premise and consequent, respectively. The general form of a fuzzy IF–THEN rule is as follows:

Rule: If x is A; then z is B.

Several types of FIS have been proposed in the literatures which are due to the differences between the specification of the consequent part and the defuzzification schemes [33]. One of these types is the so called Takagi and Sugeno FIS [34], in which the consequent variable of each rule is defined as a linear combination of input variables. The final output is then the weighted average of each rule's output. A Sugeno FIS including three input variables x, y and z are, for example, a one output variable f and three following fuzzy rules:

Rule 1: If x is A_1, y is B_1 and z is C_1 then $f_1 = p_1x + q_1y + r_1z + s_1$
Rule 2: If x is A_1, y is B_1 and z is C_3 then $f_2 = p_2x + q_2y + r_2z + s_2$
Rule 3: If x is A_1, y is B_1 and z is C_2 then $f_3 = p_3x + q_3y + r_3z + s_3$

where p_i, q_i, r_i and s_i are the consequent parameters of i_{th} rule. A_i, B_i and C_i are the linguistic labels which are represented by fuzzy sets.

The so called firing strength or degree of fulfillment of a (x, y, z) to rule i, which measures the degree to which that pair belongs to rule i, can be defined as

$$w_i = \mu_{A_i}(x) \wedge \mu_{B_i}(y) \wedge \mu_{C_i}, \quad i = 1, 2, 3 \tag{3}$$

where $\mu_{A_i}(x)$, $\mu_{B_i}(y)$ and μ_{C_i} are membership functions of x and y in fuzzy sets A_i, B_i and C_i. '$\wedge$' denotes a fuzzy T-norm operator which is a function that describes a superset of fuzzy intersection (AND) operators, including minimum or algebraic product. In this study algebraic product was used as a T-norm operator. The final output of the system is the weighted average of all rules outputs as

$$\text{Final output} = \frac{\sum_{i=1}^{n} w_i f_i}{\sum_{i=1}^{n} w_i} \tag{4}$$

In the hardness prediction problem, x, y, z are time, temperature and pressure of sintering, respectively and f is the hardness of the sample. The fuzzy IF–THEN rules used may, therefore, have the following form.

The parameters p_i, q_i, r_i and s_i are estimated by using the available input – output data. Similar IF–THEN rules are used in the prediction problem of the porosity content. There is no systematic way to know what type and shape of membership functions of premise variables have the best performance in a defined FIS. An efficient way for doing this is using an artificial neural networks (ANNs) model trained by input–output data. This method is called adaptive-network-based-fuzzy inference system (ANFIS) which is explained in the next section.

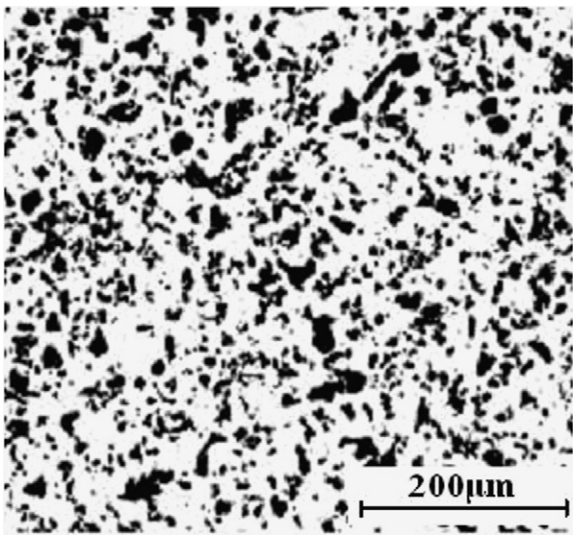

Fig. 2. Metallographic image of the specimen produced at 400 MPa and sintered for 4 h at 1000 °C with a fixture.

4. Combining neural nets and FIS

An adaptive-network-based-fuzzy inference system (ANFIS) [35] is a Sugeno type FIS in which the problem of fine-tuning membership functions of premise variables is carried out by a feed-forward neural network. ANFIS combines the advantages of both neural networks (e.g. learning capabilities, optimization capabilities and connectionist structures) and fuzzy inference systems (e.g. human like 'IF–THEN' rule thinking and ease of incorporating expert knowledge). The basic idea behind these neuroadaptive learning techniques is very simple. They provide a methodology for the fuzzy modeling procedure to learn information about a data set in order to compute the membership function parameters that best allow the associated FIS to track the given input–output data. ANFIS is based on the premise of mapping a FIS into a neural network structure so that the membership functions and consequent part parameters are optimized using a hybrid learning algorithm. In this algorithm, parameters of the membership functions are determined by a neural network back-propagation learning algorithm while the consequent parameters are by the least square method. Fig. 3 shows the structure of ANFIS including three inputs x (sintering time), y (sintering temperature), z (sintering pressure) and one output f (hardness or porosity) and three rules which were described

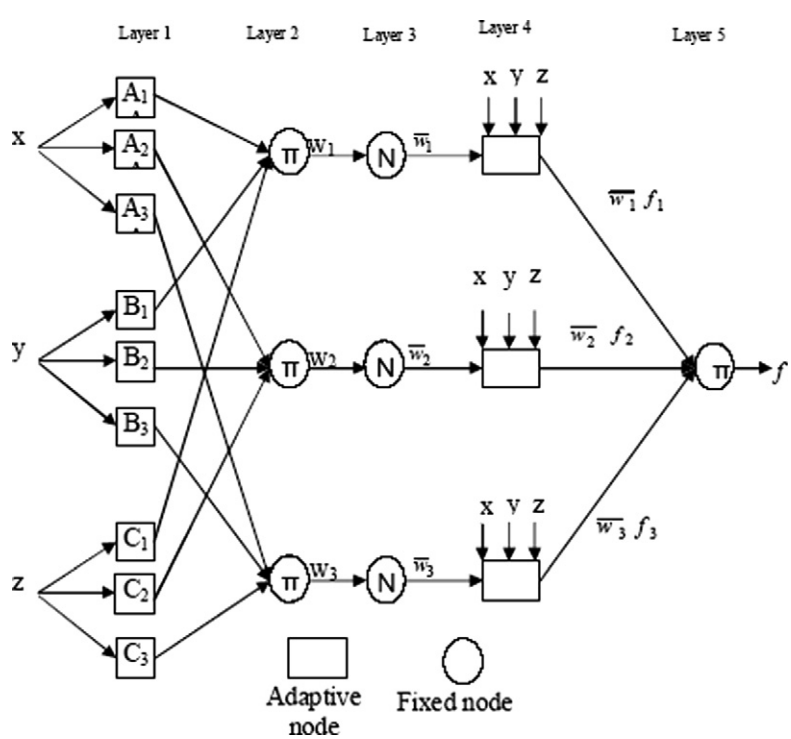

Fig. 3. ANFIS architecture.

in previous section. The first step is the fuzzifying layer in which A_i, B_i and C_i are the linguistic labels. The output of this layer is the membership functions of these linguistic labels. In other words, in this step, the premise parameters are calculated. The second step calculates the firing strength for each rule. The output of this step is the algebraic product of the input signals as can be seen in Eq. (3). The third step is the normalized layer. Every node in this layer calculates the ratio of the ith rule's firing strength to the sum of all rules' firing strength as

$$\bar{w}_i = \frac{w_i}{w_1 + w_2 + w_3}, \quad i = 1, 2, 3 \tag{5}$$

The output of every node in fourth layer is:

$$w_i f_i = w_i(p_i x + q_i y + r_i z + s_i) \tag{6}$$

The fifth layer computes the overall output as the summation of all incoming signals, which represents the results of hardness or porosity as can be seen in Eq. (4).

In this study, two ANFIS models were developed; the first one as hardness predictor and the second as a porosity content predictor. For ANFIS simulation, the data sets were divided into two groups. The first group was used for training, while the second for assessment. After development of the ANFIS models, the testing data was used for validation of the models. It was important that the ANFIS be kept as fast and efficient as possible. A subtractive clustering method [36,37] was hence used to estimate the number of clusters and cluster centers in the data set including sintering time and temperature, compaction pressure, hardness and porosity. This helped finding an initial FIS in which the number of fuzzy rules was manageable. Subsequently, ANFIS was used to tune this initial FIS.

Eighteen data points were used for training and six for testing purposes. All six data points were used for testing the model to verify the accuracy of the predicted hardness and porosity values. Figs. 4 and 5 show the observed values in comparison with those predicted. As can be seen, ANFIS has performed quite well in predicting hardness and porosity figures.

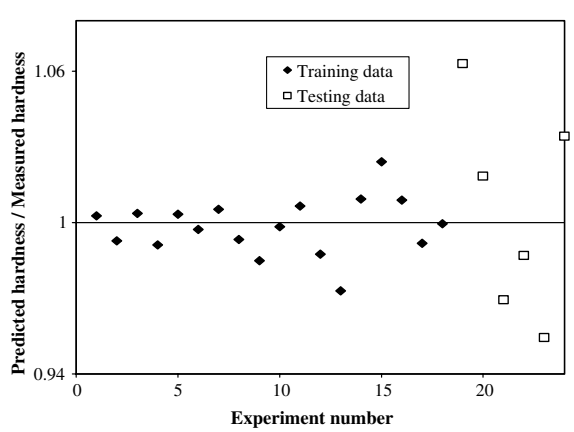

Fig. 4. Hardness predictions and relative errors by the ANFIS during sintering conditions.

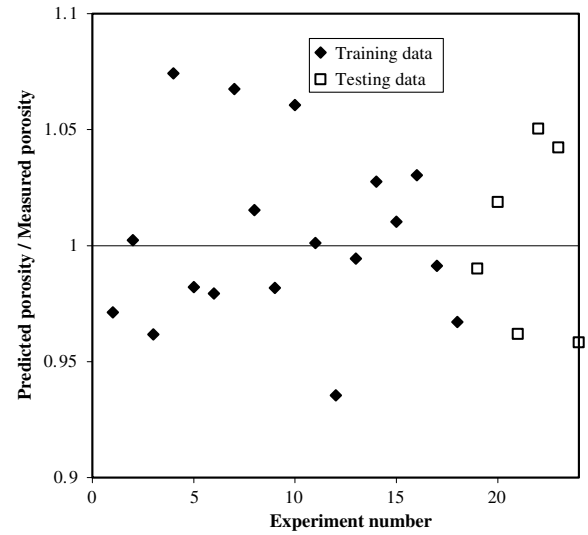

Fig. 5. Porosity content predictions and relative errors by the ANFIS during sintering conditions.

5. Results and discussion

Using a fixture has interesting effects such as stabilizing dimensions and strengthening of the sintered objects [25,26]. Fig. 2 demonstrates distribution and sizes of the pores of the specimen produced at 400 MPa and sintered for 4 h at 1000 °C with a fixture. A sintered equiatomic Ni–Ti binary alloy is generally more porous than most other powder metallurgical fabricated alloys. The reasons can be enumerated as follows:

1. Formation of new liquid-phase drops that accompany solidification shrinkages formation [38].
2. Initial free spaces between green grains of Ni and Ti powders mixed together [39].
3. Vacancy formation due to larger intrinsic diffusion coefficient of nickel in titanium than titanium in nickel in the interdiffusion zones of the specimens [40].

Different parameters, such as the sintering time and temperature, the amount of the internal porosity and the phase formation during the sintering influence the hardness of the NiTi sintered specimens [25]. Experimental results show that increasing sintering temperature and time reduces pore percentage and improves mechanical properties of the specimens. Sintering at 1050 °C results in a denser specimen compared to those sintered at lower temperatures. Free liquid movement at higher temperatures clearly shows a pronounced compaction effect. Less than 2 h of sintering may be sufficient, hence, for formation of the NiTi phase at 1000 and 1050 °C. Greater times can, however, help the formation of a denser specimen. In addition, increasing the compaction pressure decreases porosity, increases average hardness and enhances the strength of the specimens. It was seen that less than 3 h of sintering at 950 °C causes a decrease in the average hardness of the samples. This

Table 1
Statistics of predicted parameters by ANFIS model

Output parameters	RMSE	Mean relative error (%)	Max. relative error (%)	Bias
Hardness (V)	2.3	3.4	6.3	−0.1125
Porosity (%)	0.81	3.3	4.7	0.010667

decrease can simply be attributed to the porosity increase in the same time range. More than 3 h of sintering at 950 °C causes, however, a hardness increase in all compacted samples. The internal porosity is, of course, decreased and the amount of the hard precipitates may initially increase under these conditions [25].

For comparison of predicted and observed hardness and porosity values, statistical quantities of bias, root mean square (RMSE) and maximum relative error were determined:

$$\text{Bias} = \frac{1}{N}\sum_{i=1}^{N}(P_i - O_i) \tag{7}$$

$$\text{RMSE} = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(O_i - P_i)^2} \tag{8}$$

where O_i is an observed quantity, P_i is a predicted one and N is the number of observations.

Table 1 shows the statistical error of the predicted hardness and porosity values calculated by ANFIS model. It can be seen that ANFIS model over predicts the porosity (bias = 0.010667) and under predicts the hardness (bias = −0.1125) for the studied case. Hardness and porosity values predicted by the model are shown in Figs. 4 and 5. The maximum relative error for testing data was approximately 6.3% for hardness and 4.7% for porosity prediction. The mean relative error was 3.4% for the former and 3.3% for the latter. This level of error is smaller than that normally occurred due to the experimental variations and the accuracy of instrumentation. It is therefore considered satisfactory for the present predictions.

6. Sensitivity analysis

Sensitivity tests were carried out to determine the relative significance of each of the input parameters in NiTi shape memory alloy hardness and porosity predictions. A step-by-step method was carried on the trained ANFIS by varying each of the input parameters, one at a time, at a constant rate. Various constant rates were selected in this study. For every input parameter, the mean percentage change in the output, as a result of the change in the input parameter, was observed.

Results showed that the increasing of the values of input parameters affected outputs, linearly. Figs. 6 and 7 show the sensitivity of variations for hardness and porosity content at each input variable. As can be seen, sintering temperature and compaction pressure are the most significant parameters which influence the porosity content and hardness of the samples. Hardness increases with increasing of compaction pressure and decreases with increasing of sintering time and temperature. Porosity increases with increasing of the sintering temperature and decreases with increasing of the sintering time and compaction pressure. These results are very close to results of the pervious investigations, which reported that temperature is the most important parameter which considerably changes the hardness and the porosity content.

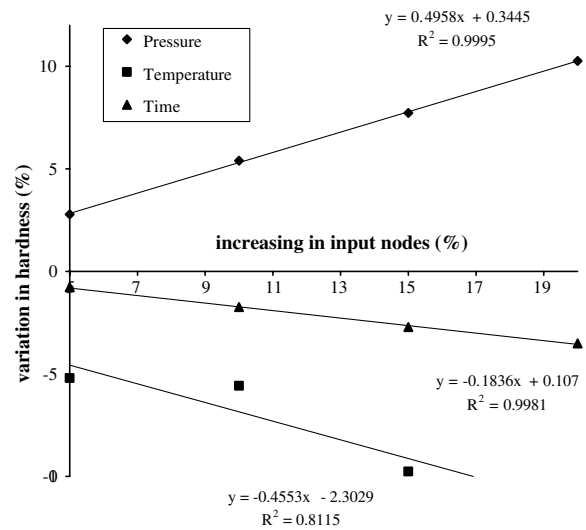

Fig. 6. Sensitivity of variations for hardness with increasing in input nodes.

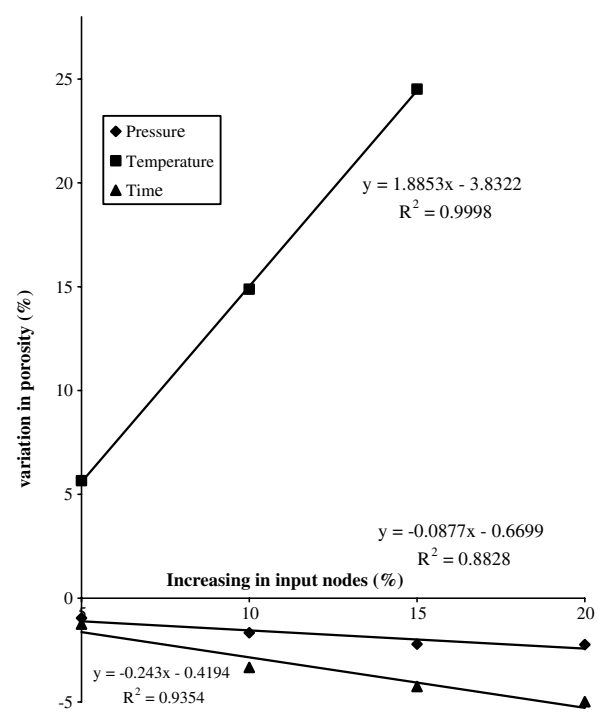

Fig. 7. Sensitivity of variations for porosity content with increasing in input nodes.

7. Summary and conclusions

Application of ANFIS for prediction of the effect of vacuum sintering parameters on properties of the NiTi SMA samples was investigated. Two ANFIS models were developed to predict hardness and porosity of NiTi shape memory alloy samples. Results of fixtured sintering experiments at 950–1050 °C for up to 5 h were successfully used to develop the models. Time, temperature and compaction pressure were used as input variables for training the models and hardness and porosity as output variables.

A FIS along with using a subtractive clustering method was developed for hardness and porosity prediction. This FIS was then used as an initial FIS for ANFIS modeling. ANFIS finds the most appropriate functioning maps of the input variables. For this purpose, 18 data points were used for training of the model. Six data points were then used to test the data evaluated by the ANFIS models. The maximum relative errors during the prediction of hardness and porosity content were approximately 6.3% and 4.7% with mean relative errors of 3.4% and 3.3%, respectively. This level of error is satisfactory and smaller than the errors that normally arise due to the experimental variations and the accuracy of the instrumentation.

ANFIS models appropriately predicted the variations of the porosity content and hardness of the synthesized NiTi shape memory samples. Increasing of the values of input parameters affected outputs, linearly. Sintering temperature and compaction pressure were the most significant parameters which affected porosity and hardness. It was concluded that suitable effects are achievable via appropriate selection of fixtured sintering conditions. New findings can help near-net-shape technology via powder metallurgy to overcome the traditional casting problems such as oxygen, nitrogen, hydrogen and carbon absorption and intermetallic compound precipitation, which lower the workability and the homogeneity of the final SMA objects.

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