Thermo-elastic response of a NiTi SMA at compression solicitation

I. CIMPOEŞU, S. STANCIU*, V. GOANŢĂ, D. C. ACHIŢEI, N. M. LOHAN, B. CONSTANTIN
"Gheorghe Asachi" Technical University of Iasi, Faculty of Materials Science and Engineering, 61A Mangeron Blvd., 700050, Iasi, Romania

Nitinol material exhibit properties as shape memory effect and super-elasticity with new important applications in many fields. Two different NiTi materials with shape memory effect (SME) and super-elasticity (SE) were subject of compression with a 4% degree using Instron equipment to simulate an external force. The initial, fully annealed materials and compressed samples were analyze by dilatation behavior point of view using Linseis L75H dilatometer. The purpose of the experiment is to establish the thermal behavior of two materials, with SME and SE, after a compression force action from linear point of view. The shape memory alloy exhibit a shape transformation domain in 80-120 ºC range. Materials, the one with SME as full cylinder and the SE one as tube were mechanically put together (no shrink forces applied) and compressed in similar conditions. After the compression test the "composite" material was heated from room temperature until 400 ºC on dilatometer. All samples (initial, compressed and combined) were heated for more cycles until a stabilization of the temperatures transformation domain was observed. The effect of super-elastic material on exhibiting of shape memory effect under temperature variation during the martensitic domain was also observed. Behavior of martensitic transformation domain was analyzed in order to establish the external effects, like temperature and stress, influence of shape memory effect.

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1. Introduction

At the micro (nano) scopic level, the distinct mechanical properties of shape memory alloys are produced by a reversible thermo-elastic martensitic transformation [1-5]. Of the dozen or more [6] intermetallic alloys that undergo a thermo-elastic martensitic transformation, equiatomic NiTi alloys have the greatest promise for large-scale applications, especially large force actuators, due to their exceptional physical and mechanical properties [6]. Most recently, NiTi have attracted increasing attention for possible application in medical implant devices and as high energy absorption structural material.

Owing to intense research efforts around the world, the mechanisms of deformation are well-characterized in polycrystalline NiTi shape memory alloys subjected to linear monotonic tensile loading conditions.

However, the characteristics of deformation in polycrystalline NiTi subjected to compression [7] and cyclic loading conditions [8] deviate significantly from the well-documented monotonic tensile response. In polycrystalline NiTi materials deformed under monotonic compression, the critical transformation stress level is higher, the transformation stress–strain slope is steeper, and the recoverable strain levels are smaller compared with tensile results [7]. During mechanical cycling, the stress–strain response of polycrystalline NiTi demonstrates a lowering of the critical transformation stress level, an increase in the transformation stress–strain slope, and a decrease in the transformation hysteresis compared with the first loading cycle [8]. More recent experimental efforts have confirmed the tension-compression asymmetry [9–11] and cyclic effects [12–17] in polycrystalline NiTi for a wide range of NiTi alloys and testing conditions.

Shape memory alloys are typically functional materials. Their unique properties like thermal and mechanical memory and superelastic properties lend themselves to a variety of applications. The shape memory effect involves the reversible thermo-elastic crystallographic phase transformation or martensitic transformation from a high temperature parent phase to a low temperature product phase [18-21]. In general, there are two types of martensitic transformations viz., a single stage A→M and a two-stage A→R→M where A is the high temperature austenitic phase, R is the intermediate rhombohedral phase and M is the low temperature martensitic phase.

Authors have studied the martensitic transformation in NiTi using DIL equipment before and after a compression force applied in elastic range. The uniaxial dilation with temperature is very sensitive to phase transformation in a material and can be used to study the transformation

behaviours of a shape memory alloy. Authors have shown that dilatometric measurements are equally reliable in characterizing phases of nitinol and martensitic transformation. The purpose of the present work is to investigate the nature of martensitic transformation in the NiTi using the dilatometric measurements. Temperature hysteresis is compared and the effect of early thermal cycles on transformation temperatures is discussed.

2. Experimental details

A nitinol shape memory alloy under bar form with 9.5 mm diameter and a super-elastic nitinol tube form with 9.55 mm inside diameter were analyze by linear thermal behavior before and after a compression test. The materials were acquisitioned from Saes Getters USA Company. Mechanically cut under water cooling flux two NiTi materials were subject of compression tests made on Instron equipment keeping the compression on elastic domain. After each compression test a dilatometer analyze was realized from room temperature to 400 °C paying respect for the martensitic transformation domain of nitinol material with shape memory effect. The linear-thermal analyses were carried out on a Linseis 75H dilatometer using a 10 K/min heating rate and a water cooling system to control the furnace temperature. For martensitic transformation information calorimetric results were analyzed on compressed sample using a DSC Maya Netzsch equipment with 10 K/min heating rate in Ar atmosphere.

3. Experimental results

Uniaxial dilation is measured after offsetting the initial compression brought about by the constant load of 100 g at room temperature. Fig. 1 shows a typical curve of dilatation response with temperature variation for analysed materials. Abrupt and large changes in uniaxial dimension are observed during heating at certain temperature in case of shape memory alloy, in a), and a classical metallic dilatation of the superelastic material in b). The modiffications in shape memory alloy linear behavior are attributed to phase transformations taking place in the material [10].

Martensite to austenite transformation during heating causes sudden contraction of the material with extreme large values. On heating, a normal expansion is observed till about 35 °C, with a contraction of 23.2 µm at 87.2 °C, and then it starts contracting. The contraction of the sample continues till it reaches 283 °C with a 428.5 µm contraction and again there appears normal expansion above this temperature. Concerning the superelastic sample dilatation a value of 16.32 µm is registered at 250 °C.

The materials as a comune structure of 19 mm length and 9.5 mm in diameter were subject of commpression with 4% degree. The compression curves present a very good recovery degree, results subject of a different article analyze, of the shape memory alloy and superelasticity of SE material. After the compression of composite structure the linear behavior of both in the same time materials were observed and presented in Fig. 1 c) for three heating cycles. Data obtained from linear behavior of shape memory alloy in initial state and as composite structure are presented in Table 1.

To set the transformation temperature tangents are drawn at the points where there are changes in the slope of dilatation curve. The point of intersection of tangents is taken as the appropriate transformation temperature.

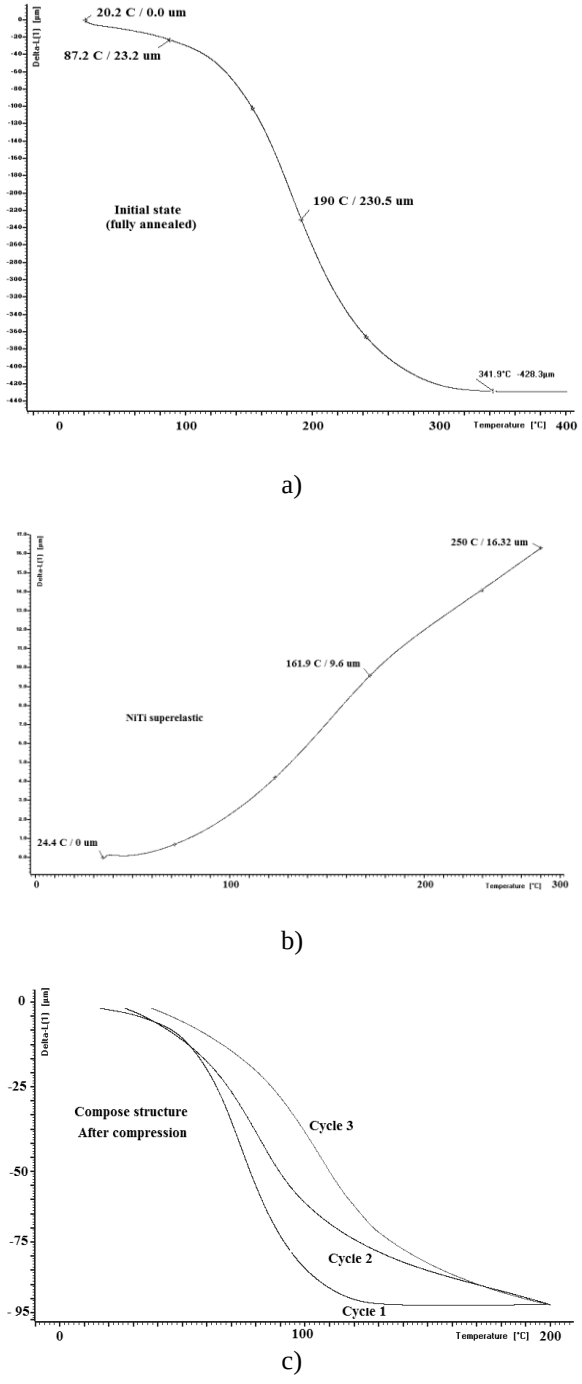

Fig. 1. Typical uniaxial dilation Vs temperature curve of NiTi a) SMA initial state sample (drawing cylinder), b) superelastic sample and c) composite structure (both elements).

The austenitic transformation points recorded on heating curve for initial state and composite state after compression are presented in Table 1. The martensitic transformation range, in degrees and µm as contraction, is also presented in Table 1 evidencing the material behavior change under compression, thermal solicitation and super-elastic element effect. In first case, initial state, the material behave a large transformation domain on almost 200 °C and a compression of 371 µm. Applying a compression to material and an extern constrain (SE material) the linear behave of material change by decreasing the transformation temperatures under 110 °C, sliming the transformation range to a value around 54 °C and decreasing by seven times the linear contraction of material from 370 to 55 µm.

Table 1. Dilatometric parameters and results for all shape memory NiTi sample tests and for composed structure NiTi SMA + NiTi super-elastic after a compression operation.

Nr. crt.	NiTi shape memory alloy	Sample diameter [mm]	A_s [°C]	A_f [°C]	ΔA [°C]	A_{50-1} [°C]	A_{50-2} [°C]	ΔA_{50} [°C]	ΔL_A [µm]	ΔL_{50-1} [µm]	ΔL_{50-2} [µm]
1	Initial producer state (fully annealed)	19.2	115	283	168	199	199	0	371	185.5	185.5
2	After compression Cycle 1	18.85	55.5	108.5	54	82.5	80	2.5	50	21	29
3	After compression Cycle 2	18.89	56	110	54	83	81	2	54	30	24
4	After compression Cycle 3	18.85	50	107	57	78.5	78.5	0	58	29	29

The compose structure were cycled three times until 200 °C and stabilization of transformation domain guilty of shape memory effect turn to stabilize to certain precise values. To follow the stability and extension or compression of the analyzed materials the half point of temperature transformation by temperature variation or dilatation variation were obtained as A50-1 and A50-2 in °C. These values present a good evolution and stability of martensitic to austenitic transformation with reduce variation in first and second heating cycle of compose structure by 2.5 respectively 2 °C with small influences on industrial application and maybe important for MEMS solutions.

The material behavior is at least curious first because the shape memory material suffer a mechanical 4% contraction in elastic domain (that represent 760 µm from 18000 µm the total height) and a material recovery of 2% that cover the compression with temperature variation in initial state, respective 371 µm, and in compressed state the material suffer a new thermal compression with energetic contribution of 50 to 58 µm.

Concerning the super-elastic element that cover the shape memory element his linear variation with temperature modification imply a dilatation of 9.6 µm at 150 °C but based on no firm contact between the compose structure two elements is difficult to appreciate if the shape memory effect is or not affected.

The compressed shape memory material was analyzed through differential scanning calorimetry (DSC) to establish few martensitic transformation parameters.

A 49 mg sample cut under water cooling system from the compressed SMA sample was analyzed in RT-400 °C temperature range.

The calorimetric result presented in Fig. 2 and based on tangent method determination shows the transformation points situated at 60 respectively 118 °C near to points determined through dilatation variation with temperature, position 4 in Table 1, with an endothermic character. The middle of transformation in this case A_{50}, showed in table 1 through linear dilatation characteristics results ΔA_{50}, is situated at the middle of A→M domain but with a small variation to the right with 4 °C that was not determined through linear calculation.

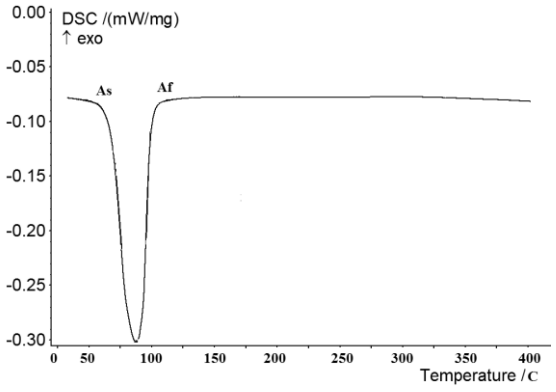

Fig. 2. Typical representative differential scanning calorimetry chart of shape memory alloy NiTi after compression.

Specific dissipated heat, usually noted with Δh, has a value of -0.3015kJ/kg characteristic for martensitic transformation and was determined based on a linear baseline. The specific dissipated heat represent the heat necessary for NiTi material to undergo martensitic transformation from A_S to A_f points. The martensitic transformation heat flow rate is -0.355 mW/mg/min which represent a high value comparing to copper based shape memory alloy [3] and increase the material response to external solicitations like temperature.

The SMA behavior is based on martensitic transformation evolution and the ability of NiTi shape memory material to store external forces and to still exhibit shape memory effect.

4. Conclusions

Two NiTi elements, one super-elastic under tube shape with 9.55 mm diameter and second with shape memory effect as solid cylinder with 9.5 mm diameter, were analyze through dilatometry separately and as composite structure in a RT-400 °C temperature range. The materials linear behavior Vs temperature was analyzed and comment to establish a 4 % elastic compression influence on martensitic transformation range of the shape memory alloy with interesting results concerning the transformation temperatures, domain limits and compression under thermal effect value. After compression the material present a stabilization of liner behavior, especially the martenstic transformation range, with nice characteristics for actuation applications of this material even in large force usage. Calorimetric investigations on compressed material present approximate values for austenitic transformation with the dilatation Vs temperature variation determinations. The transformation stability present a reduce variation of middle transformation point A_{50} with 4 °C to the right comparing to dilatation determination results. The thermal properties of martensitic transformation at heating like specific dissipated heat and heat flow rate were determined for further conclusions in compress shape memory alloy behavior at thermal modifications.

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*Corresponding author: sergiustanciu2003@yahoo.com