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A New Characterization Methodology For Shape Memory Alloys

A. Barcellona¹ and D. Palmeri

¹Università degli Studi di Palermo, Dipartimento di Ingegneria Chimica, Gestionale, Informatica, Meccanica, Palermo, barcellona@unipa.it.

Abstract

Shape Memory Alloys are metal materials that have capability to recover their original shape after being strained. The retention of the cyclic stability of the shape memory effect is a key topic for shape memory alloys used in shape memory conditions. Good shape memory properties frequently decrease during shape memory cycling. A method for improving the cyclic stability of TiNi shape memory alloys is the grain refinement by means of high-pressure torsion, equal-channel angular extrusion or friction stir processing. This topic requires a material characterization method able to detect all the influent factors on the functional properties of the grain refined material. Currently four techniques as the differential scanning calorimetry, the active A_f method, the strain measurement under constant load method and the resistivity method, are used for material characterization. In this research a quick and cheap dedicated method able to provide the whole stress-strain-temperature characterization in only one test of the shape memory material has been developed. The whole characterization of shape memory material was carried out using a properly designed experimental set up able to recording temperatures, loads and strains during the testing paths. The outputs of this analysis were summarized through three correlated values (stress–strain–temperature).

Keywords: Shape Memory Alloys, Functional Characterization, Transformation Temperatures.

1 INTRODUCTION AND SMA CHARACTERIZATION METHODS

Shape Memory Alloys (SMAs) are metal materials that have unique properties such as recovering their original shape after being strained, as a result of a reversible and diffusionless solid state phase transformation from the austenitic phase to the martensitic one. This capability of material to recover their original shape is named shape memory property, while the capability of material to undergo a large recoverable strain after unloading is named superelasticity.

The shape memory effect is attributable to a temperature and stress dependent change in the crystalline structure of material between two different phases named

martensitic and austenitic and respectively stable at “lower” and “higher” temperatures.

These temperatures, at which the phase transformations occur, are called transformation temperatures and are defined as following:

- A_s is the temperature at which the austenitic transformation starts during heating,
- A_f is the temperature at which the austenitic transformation finishes during heating,
- M_s is the temperature at which the martensitic transformation starts during cooling and
- M_f is the temperature at which the martensitic transformation finishes during cooling.

In the last decade the development of shape memory components has spread not only to sensor-actuator applications but also to the robotic, aerospace, micro-electronics, medical, dental and vibration control fields.

The extension of the application fields of SMAs is related to the possibility to manage the functional properties of material in an accurate way considering all the existing conditions of material as martensitic, austenitic and mixed austenitic/martensitic[1].

The retention of the cyclic stability of the shape memory effect (SME), as reported in the literature, is a key topic for SMAs used in shape memory conditions, in effect, shape memory properties frequently decrease during SME cycling.

The instability of transformation cycles, that appears in term of variations in transformation temperatures, makes difficult the accurate predictions of materials behavior and makes inaccurate designing of components for SME applications. In order to overcome the cyclic instability problem it is necessary to increase the critical shear stress for dislocation slip. This goal, as reported in literature, is achievable by means of coherent nano-precipitation, grain refinement and strain hardening by dislocation substructures. The material processing techniques as high-pressure torsion, equal-channel angular extrusion, and friction stir processing (FSP) are attractive in order to attain grain refinement [2-6] .

In particular, the high Stacking Fault Energy, an important parameter influencing deformation mechanism of TiNi alloys, suggests the occurrence of the continuous dynamic recrystallization (CDRX) during the FSP of material. In this way the FSP offers the possibility of grain refinement in only one processing phase [7, 8].

The aim of this work is the developing of a quick and cheap dedicated characterization method able to detect all the infuent factors on the functonal properties of the grain refined material in only one test. In order to consider different functional capability thin sheets (0.5 mm) of Ti-49.4Ni alloy flat memorized, which remains in the martensitic state at room temperature, where thermomechanically treated, by means of FSP, considering five thermo mechanical load pats. The whole functional characterization of SMA material was carried out using a properly designed experimental set up able to recording temperatures, loads and strains during the defined testing paths. The outputs of this analysis where summarized by three correlated values (stress-strain-temperature). In order to quantify the shape memory effect of material independently

of the material ductility variation originated from the grain refinement process, starting from the strain outputs of the designed characterization method, an index named Functional Index of material (FI) able to quantify the only SME has been defined.

Currently four techniques as the differential scanning calorimetry DSC, the resistivity method, the active A_f method and the strain measurement under constant load method are largely used for SMA characterization. Each specific thermomechanical test characterizes a specific remarkable property of SMAs; in effect, the stress-strain curve at constant temperatures illustrates superelasticity and the rubber-like behavior, whereas the strain-temperature diagram obtained under constant load usually describes the one-way shape memory effect. The whole characterization of SMAs currently requires the carrying out of a lot of tests; in particular the DSC test coupled to the mechanical loading/unloading test at the varying of the temperature give many information on the transformation temperatures and on the superelastic or shape memory effect. All the existing characterization methods are based on the enforcement of various loading paths while phenomena associated with the phase transformation are recorded.

The Constant Load method is based on the applying a load to the alloy and monitor its deformation and shape recovery simultaneously with temperature as the material is cooled and heated through the transformation temperatures range. The Differential Scanning Calorimeter method allows determining of the transformation temperatures under zero stress, but require an expensive instrument. It is a thermo analytical technique in which the difference in the amount of heat flow required to maintain the same temperature of a sample and of a reference chamber throughout the experiment is measured as a function of temperature. The Active A_f method is based on bending a sample of SMA in martensitic condition and then monitoring the shape recovery while it is heated above the A_f temperature.

The complete characterization of functional properties of material foresees the determination of material phase diagram in which the four transformation temperatures are plotted as function of the stress level of material. This diagram shows a linear trend of the transformation temperatures at the varying of the stress level and it allows to identify all the parameters connected to the functional properties of material as: the transformation temperatures under different stress level (included zero stress level), the material thermomechanical hysteresis values and the stress influence coefficients defined by the slope of transformation temperatures lines. At least two common characterization techniques must to be performed in order to attain a full functional characterization of a SMA component [9-13].

2 VARIABLE LOAD CHARACTERIZATION

The goal of improving the cyclic stability of SMA components used in SME condition, as highlighted in the previous section, requires the development of an appropriate characterization method able to obtain in addition to the complete material functional characterization, through the phase diagram determination, the assessment of the

effect of the grain refinement process on the functional properties of material. Together with these aims, it must be possible to have the possibility of a cyclical evaluation of the characterization parameters and it must also be possible to distinguish between the effects of the grain refinement process in terms of modifying the material mechanical properties and change the material functional properties. For the achievement of these purposes, a methodology for characterization, that brings together some aspects of the method Af active and some characteristics of the method at constant load, has been developed. The shape memory properties were measured by a variable load (VL) tests allowed by a properly designed equipment. Four different FSP grain refinement have been considered at the varying of the process parameter identified as the tool sinking depth and performing a post processing annealing of material (following indicated as TT). In particular the FSP was performed on the middle of two 100 mm × 70 mm thin SMA sheets (0.5 mm) for a length of 40 mm. The influence of the variation of the tool sinking depth was investigated by selecting two values of (p) parameter respectively equal to 0.2 mm and 0.3 mm (following indicated as 0.2 an 0.3). Three specimens (100 mm × 8 mm) were transversally cut, regard to the processed line, and tested on the VL equipment; moreover each processed specimen has been submitted to a post-processing TT following the typical thermal cycle used in order to confer the shape memory properties to the NiTi alloys. The processed specimens, therefore, have been heated up to 450 °C and maintained at this temperature for 300 seconds, than they have been quenched in water. Also the post-thermal treated specimens have been characterized by VL tests [14-16].

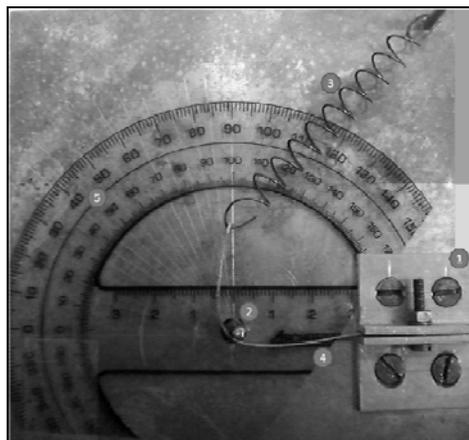


Figure 1. VL tester: equipment for functional characterization of material: 1 clamping, 2 bending , 3 contrast spring, 4 specimen, 5 goniometrical reference.

Figure 1 shows the employed equipment constituted by a clamping device for the specimen, a bending spike positioned to bend the processed line, a contrast spring in

order to macroscopically detect the phase transformations during the cooling and a goniometrical reference in order to measure the bending angle variations. The contrast spring applies a variable load on the material during the thermal scanning as function of the spring elongation.

The stiffness of the used spring is equal to 2.7 N/cm. For each bending angle it is possible to compute the corresponding load value, by recording the spring elongation, and therefore it is possible to define the load levels at which the transformation temperatures were measured. This equipment was put inside a metallic container filled with oil and the thermomechanical cycles were carried out as follows. The VL tester was heated up to the temperature A_f , to activate shape memory effect (SME), by a resistance heater; the temperature was measured using a K thermocouple and it was recorded together to the bending angle using a digital camcorder.

The cooling down to the temperature M_f was carried out using a water cooler. These investigations revealed the phase transformation temperatures (M_s , M_f , A_s and A_f) under different loads for both as-received (AR) and processed (Grain Refined) materials. The whole thermomechanical characterization of material required also the determination of transformation temperatures under zero load condition. These further tests, performed using the previous apparatus without using the contrast spring, allowed to visualize the stress-temperature characterization phase-diagram of material as reported in figures 2, 3 and 4.

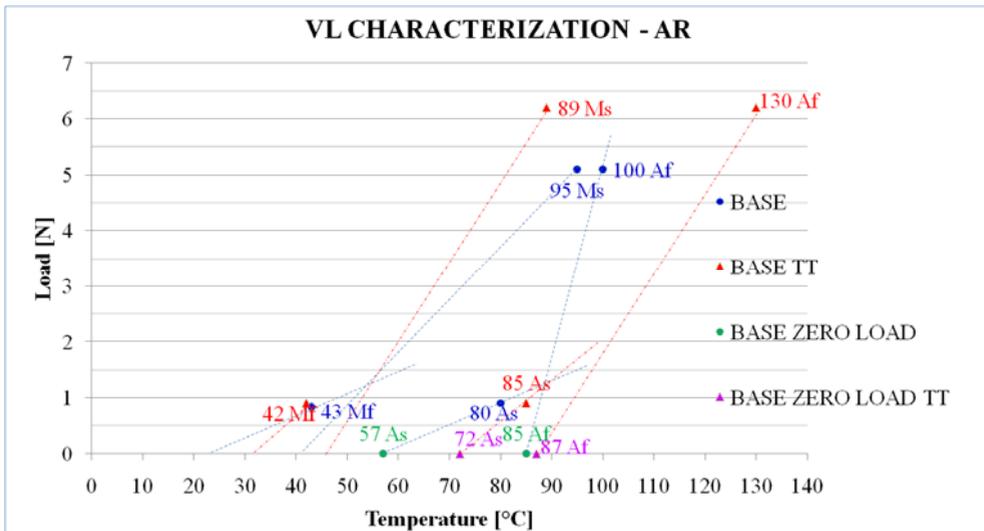


Figure 2. Phase – diagram for AR material (dashed line) and AR TT material (dashed dot line).

In particular these diagrams report the transformation temperature values at the varying of the load levels; the evaluation of the dL/dT slopes (stress influence

coefficients) respectively for the M_s , M_f , A_s and A_f temperatures may suggest the influence of the thermomechanical process on the shift sensitivity of the transformation temperatures under load.

The functional properties of material were identified taking into account two highlighted phenomena by the VL characterization tests, indeed, the used technique in order to determine the transformation temperature of material evidenced that the FSP gives both ductility and shape memory capability reduction. This occurrence is related to the ausforming of shape memory material produced by friction stirring. In order to quantify these two different effects and to compare them to the functional properties of the base material (AR) a Functional Index of material (FI) has been defined.

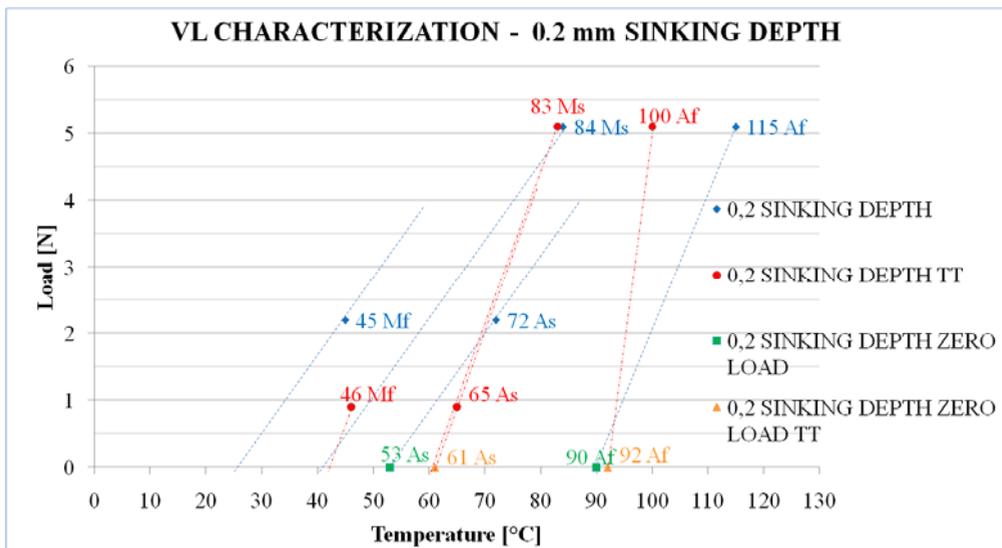


Figure 3. Phase – diagram for 0.2 material (dashed line) and 0.2 TT material (dashed dot line).

Considering the initial bending angle θ_i and the final bending angle after shape recovery θ_f the FI is given by the ratio (1):

$$FI = \frac{\left(\frac{\theta_i - \theta_f}{90} \right)}{0.8} \quad (1)$$

Where 90 is the initial bending angle of the base material and 0.8 is the effective shape memory capability of the AR material given by the ratio (2):

$$\left(\frac{90 - \theta_f}{90} \right) \quad (2)$$

Were θ_f of AR material is equal to 18° . The FI index therefore quantifies the material shape memory capability reduction compared to that one of the AR material considering also the decreasing of material ductility associated to the FSP. The indicator of the material ductility reduction is given by the θ_i value, instead the intrinsic total material shape memory capability is given by the $\theta_i - \theta_f$ value.

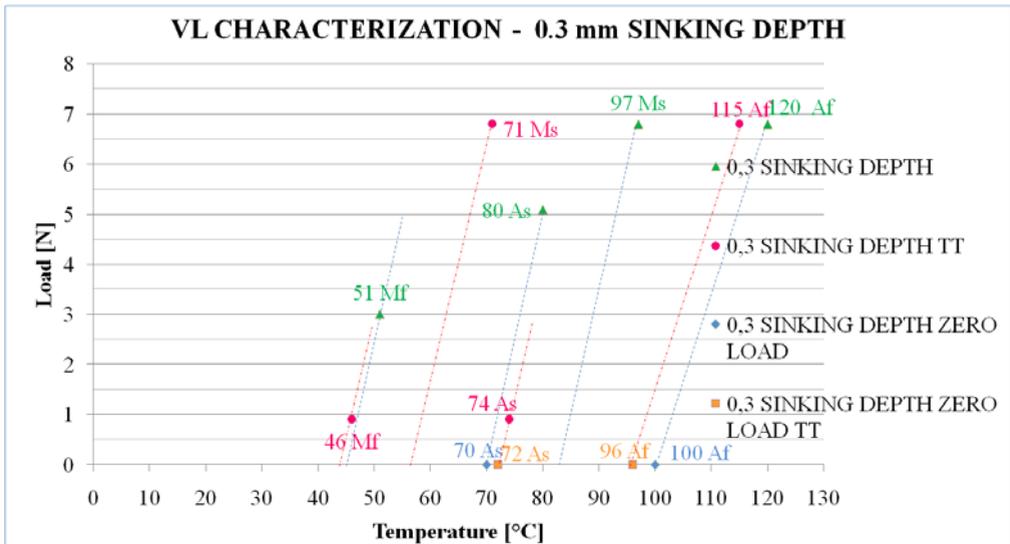


Figure 4. Phase – diagram for 0.3 material (dashed line) and 0.3 TT material (dashed dot line).

The key point of method consist in the occurrence that the generated variable load is strictly correlated with the functional material properties since the stress condition of material is generated from the material phase transformation.

There is therefore the possibility to give simultaneously information on the transformation temperatures of material and on the effect of the grain refining process on the functional material properties.

3 DISCUSSIONS AND RESULTS

The output of the VL testing consist in the evaluation of the material Functional Index and on the recording of the variable load curves by performing an heating-cooling thermal cycle and recording the angular displacement for each temperature. All the determined temperatures, related to a specific load value, and FI index have been respectively summarized in the tables 1 and 2. All the evaluated phase transformation temperatures are referred to different loads, therefore, in order to have a correct

reading of these results it is helpful to place these temperature values on the load-temperature characterization phase-diagrams.

Specimens					
0.2	2,2	2,2	5,1	5,1	Load [N]
	72	45	115	84	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	53	26	90	41	Transformation temperatures [°C]
0.2 TT (300 s at 450 °C)	0,9	0,9	5,1	5,1	Load [N]
	65	46	100	83	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	61	42	92	60,5	Transformation temperatures [°C]
0.3	5,1	3	6,8	6,8	Load [N]
	80	51	120	97	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	70	45	100	83	Transformation temperatures [°C]
0.3 TT (300 s at 450 °C)	0,9	0,9	6,8	6,8	Load [N]
	74	46	115	71	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	72	44	96	57	Transformation temperatures [°C]
AR (as-received)	0,9	0,83	5,1	5,1	Load [N]
	80	43	100	95	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	57	23	85	41	Transformation temperatures [°C]
AR (as-received) TT (300 s at 450 °C)	0,9	0,9	6,2	6,2	Load [N]
	85	42	130	89	Transformation temperatures [°C]
	As	Mf	Af	Ms	
	0	0	0	0	Load [N]
	72	32	87	46	Transformation temperatures [°C]

Table 1. Experimental results: transformation temperatures and VL values.

The zero load test results allowed to evaluate the slope $dLoad/dTemperature$ for the transformation temperatures. The determined phase diagrams are able to highlight the effect of the grain refinement process or post-thermal treatment on the functional properties of material. In effect, considering the case of FSP 0.2 material the TT annealing treatment generates an appreciable reduction of the temperatures variation under load; in effect a counterclockwise rotation of 15° of the A_s and A_f lines have been observed. In the case of FSP 0.3 material the TT generates only a rigid shift of the A_f and A_s lines that reduces their distance. The comparison of both the previous cases to the AR material highlights the advantage of FSP to reduce the transformation temperatures sensitivity to the load variation.

	AR	0.2 mm sinking depth	0.3 mm sinking depth
FI	1	0.275	0.187
Annealed (300 s at 450 °C)			
	AR	0.2 mm sinking depth	0.3 mm sinking depth
FI	1	0.412	0.625

Table 2. FI (Functional Index).

All these results underlined two distinct effects that allow a FI index value reduction following the grain refinement process. There is a ductility reduction measured by the decreasing of the initial bending angle θ_i , and a total shape memory capability reduction measured by the decreasing of the difference $\theta_f - \theta_i$. In effect, it is necessary to highlight that the total shape memory capability reduction is also affected by the reduction of the material ductility, but the value of the θ_f angle, compared to the same angle of the AR material give a measure of the only shape memory reduction because of the bending angle is strictly related to the material properties.

The ductility reduction resulted strongly affected by the chosen values of the grain refinement process parameters.

In particular, it has been observed that the FSP determines an increased ductility reduction at higher values of sinking depth parameter, furthermore, this ductility reduction is coupled with a reduction of the material functional properties. The VL results for the TT specimens shown the possibility to obtain an increasing ductility and a functional properties recovery when the sinking depth parameter increases.

This occurrence is attributable both to a reorientation of martensite variants inside the grains of processed region and to the formations of a free martensite area at the top of the processed zone. In effect, the plastic flow of detwinned martensite, due to the tool stirring action, brings the ductility reduction, while the shape memory capability reduction is attributable also to a lack of martensite plates at the top of the stir zone. This occurrence appears much high when the sinking depth value is lower.

The computation of the FI value for each processing condition shown that the FSP gives a reduction of 72.5% and 81.2% of the functional properties of material,

compared to the FI of base material, respectively for 0.2 and 0.3 mm sinking depth. The reduction of FI value takes into account both the above mentioned phenomena but the contribution of each of them varies depending on the occurring microstructural modifications.

The reading of the post processing thermal treatment effect on the material phase diagram highlighted the impossibility to recover the material functional properties when the FI reduction is attributable to a reduction of the martensite plate region. The FI reduction imputable to the ductility reduction may be partially recovered by performing a post processing thermal treatment able to activate a development of a multivariants martensite grow inside the new formed austenitic grains. The post processing thermal treatment effectiveness, in effect, has been underlined for the 0.3 TT specimens.

4 CONCLUSIONS

The key topic for SMAs used in shape memory conditions of retention of the cyclic stability of the shape memory effect (SME) need to develop of an appropriate characterization method able to obtain in addition to the complete material functional characterization, through the phase diagram determination, the assessment of the effect of the grain refinement process on the functional properties of material. Together with these aims it must be possible to have the possibility of a cyclical evaluation of the characterization parameters and it must also be possible to distinguish between the effects of the grain refinement process in terms of modifying the material mechanical properties and change the material functional properties.

For the achievement of these purposes a new methodology for the SMA characterization that brings together some aspects of the method Af active and some characteristics of the method at constant load has been developed. The shape memory properties were measured by a variable load (VL) tests allowed by a properly designed equipment.

The key point of method consist in the occurrence that the generated variable load is strictly correlated with the functional material properties since the stress condition of material is generated from the material phase transformation. There is therefore the possibility to obtain simultaneously information on the transformation temperatures of material and on the effect of the grain refining process on the functional material properties. Moreover the whole characterization of material is achievable using only one testing method.

The output parameters of the testing method are:

- The material phase diagram in which the four transformation temperatures are plotted as function of the stress level,
- The material thermomechanical hysteresis values,
- The stress influence coefficients,
- The FI index that quantifies the total shape memory capability of material compared to shape memory capability of the AR material.

The used characterization method allowed to identify the effect of both the grain refinement process and the post-thermal treatment on the functional properties of material and to evaluate all the characterization parameters of material. In effect the obtained results underlined the possibility to preserve up to 62.5% of functional properties of the base material by properly control the ratio between the martensite plate free region and martensite reoriented region, produced by the heating and stirring tool action, through a post processing annealing treatment.

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A New Characterization Methodology For Shape Memory Alloys

A. Barcellona¹ and D. Palmeri

¹Università degli Studi di Palermo, Dipartimento di Ingegneria Chimica, Gestionale, Informatica, Meccanica, barcellona@unipa.it, dinapalmeri@hotmail.com.

Keywords: Shape Memory Alloys, Functional Characterization, Transformation Temperatures.

Extended Abstract

Shape Memory Alloys (SMAs) are metal materials that have unique properties such as recovering their original shape after being strained, as a result of a reversible and diffusionless solid state phase transformation from the austenitic phase to the martensitic one. This capability of material to recover their original shape is named shape memory property, while, the capability of material to undergo a large recoverable strain after unloading is named superelasticity.

The development of shape memory components in the last decade has spread not only to sensor-actuator applications but also to the robotic, aerospace, micro-electronics, medical, dental and vibration control fields. The extension of the application fields of SMAs is related to the possibility to manage the functional properties of material in an accurate way considering all the existing conditions of material as martensitic, austenitic and mixed austenitic/martensitic. Thermomechanical treatment of SMAs allows the adjusting of a number of functional properties, like the phase transformation temperatures or the shape recover capability. Moreover, the annealing of the component or the semi-finished shape, usually is the last step of the fabrication process.

Currently four techniques as the differential scanning calorimetry DSC, the resistivity method, the active A₁ method and the strain measurement under constant load method are largely used for material characterization.

Each specific thermomechanical test characterizes a specific remarkable property of SMAs; in effect, the stress-strain curve at constant temperatures illustrates superelasticity and the rubber-like behavior, whereas the strain-temperature diagram obtained under constant load usually describes the one-way shape memory effect. The whole characterization of SMAs currently requires the carrying out of a lot of tests, in particular the DSC test coupled to the mechanical loading/unloading test at the varying of the temperature give many information on the transformation temperature and on the superelastic or shape memory effect [1].

The three important physical parameters that interact in the description of SMA are stress, strain and temperature. These variables define a general three-dimensional

physical space described [2]. The aim of this work is to consider the functional temperature of material to consider different memorized, with thermomechanical material was found to identify the existing. In this research using a proper and strains data summarized by micrographic independently process, starting index named F defined.

The key point is strictly correlated material is given possibility to of material and its properties. More one testing method

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physical space in which the complex mechanical behavior of the material may be described [2 - 3].

The aim of this work is the developing of a quick and cheap method able to identifies the functional properties, the transformation temperatures and the whole stress-strain-temperature characterization of the shape memory material in only one test. In order to consider different functional capability thin sheets (0.5 mm) of Ti-49.4Ni alloy flat memorized, which remains in the martensitic state at room temperature, where thermomechanically treated considering five thermomechanical load pats. The tested material was firstly characterized from the microstructural point of view with the aim to identify the existing conditions of material.

In this research the whole functional characterization of SMA material was carried out using a properly designed experimental set up able to recording temperatures, loads and strains during the defined testing paths. The outputs of this analysis where summarized by three correlated values (stress-strain-temperature) related with the micrographic observations. In order to quantify the shape memory effect of material independently of the material ductility variation originated from the grain refinement process, starting from the strain outputs of the designed characterization method, an index named Functional Index of material (FI) able to quantify the only SME has been defined.

The key point of method consist in the occurrence that the generated variable load is strictly correlated with the functional material properties since the stress condition of material is generated from the material phase transformation. There is therefore the possibility to obtain simultaneously information on the transformation temperatures of material and on the effect of the grain refining process on the functional material properties. Moreover the whole characterization of material is achievable using only one testing method.

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