INVESTIGATION OF SOME ELECTRICAL PROPERTIES OF NiTi WIRES PRESENTING THE SHAPE MEMORY EFFECT

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Lucrarea prezintă rezultatele unor experimente de măsurare a rezistivităţii electrice a două tipuri de sârme de aliaj de NiTi tratate pentru a prezenta efectul de memoria formei.Probele utilizate au fost analizate micrografic şi compoziţional, precum şi prin scanare calorimetrică diferenţială(DSC).Utilizarea tot mai frecventă a unor asemenea materiale în diverse domenii ale tehnogiilor de vârf impune cunoşterea cât mai în detaliu a proprietăţilor acestor materiale cu memoria formei în vederea unor utilizări cât mai variate, dar şi pentru a valorifica pe deplin potenţialul lor mai ales că NiTi păstrează multe dintre proprietăţile remarcabile ale Ti.

Experimental results of resistivity measurements for two kinds of NiTi wires previously treated to have memory shape effect are presented in this paper. The samples were analised using DSC method. The increasing number of applications of memory shape alloys is reflected connected to an increasing number of studies regarding their utility mainly in high technology field. Therefore a better knowledge of their properties increases the opportunities for more and more different fields. NiTi is one of the most popular shape memory alloys becouseit preserves many of Ti remarcable properties.

Keywords: Shape memory alloys (SMA), shape memory effect (SME), DSC (Differential Scanning Calorimetry), electrical resistivity

1. Introduction

Shape memory alloys (SMA) received a lot of interest and intensive research during the last few decades, due to their many applications in different domains of human activities: avionics, producing special medical instruments, robotics, actuators, antennas, etc. [6].

Shape Memory Effect consists in turning back to its initial shape a sample previously deformed in the plastic range at A_s by heating it at a temperature higher than A_f thanks to the reverse transformation. Strain may be a tensile stress, a

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compression or a bending stress. As long as the stress doesn't exceed a critical value, the reverse transformation takes place.

This effect can be observed on samples that were stressed at M_f or at temperatures between M_f and A_s, after that it starts to be unstable. Depending on temperature shape memory mechanism is slightly different. Many researches have described the case of a monochrystal initially at a temperature $T \leq M_f$. Martensite is formed in these conditions without shape modifications in a self-accommodating manner. If an external stress is applied, the twin boundaries move in order to accommodate to the applied stress. When the speciment is heated above A_f, the reverse transformation occurs and the original shape is regained if the transformation is crystallographically reversible [4].

The mechanical motion in shape memory alloys (SMA) is caused by the changes of their crystalline structure which is affected by several factors. There are known two types of crystalline structures: one is a highly symmetrical structure – austenite and the second is a structure with less symmetrical crystalline organization (orthorhombic, tetragonal) – martensite. Several types of martensite can arise from one austenite structure during martensite transformation. The reverse martensite transformation results only in one austenite type. Conversion inside the SMA materials isn't a thermodynamically reversible process. There is energy dissipation due to internal friction and creation of structural defects [2].

The most flexible and beneficial shape memory alloy (SMA) that has been discovered so far for engineering applications is TiNi. Its applications are based on its properties, as for instance the fact that it has a hard deformable state at high temperature (austenite) and a deformable state (martensite) at a lower temperature. The shape memory alloys (SMA) based on TiNi possess the ability to undergo shape change at low temperature and retain this deformation until they are heated, at which point they return to their original shape [3].

The most important in characterizing an SMA material is to determine the characteristic phase transformations temperatures. Actually, these SMAs are presenting a hysteretic behavior, and there are several transformation temperatures to speak about, including the austenite start temperature (A_s) and the austenite finish temperature (A_f) during heating and the martensite start temperature (M_s) and the martensite finish temperature (M_f) during cooling. Additionally, an intermediate phase (R phase) often appears during cooling, having its own start temperature (R_s) and finish temperature (R_f), before the transformation proceeds to martensite at lower temperatures [5]. All the three martensitic-type transformations exhibited by binary near-equiatomic NiTi alloys are hysteretic, as expected for first-order phase transformations [4].

Advantages and disadvantages of TiNi shape memory alloys compositions and their usual applications are to be analyzed. The price of producing such an alloy is still high and involves some difficulties dew to its special properties, but precisely those ones make TiNi one of the alloys of the future (roughness, flexibility, biocompatibility, excellent corrosion resistance, etc.). Inducing the shape memory effect (SME) through special treatments to TiNi alloys can really offer more and diverse perspective to their applications. Some of these particular properties of the TiNi SMA are: ability to be electrically heated for shape recovery, stable transformation temperature, more recoverable motion [1].

2. Materials and experimental procedures

The usual steps in producing TiNi based alloys presenting SME described in scientific references are:

1. starting with SMA sheet rod wire etc.;
2. forming the memory shape, clamping in this shape and heat for austenite forming, then unclamping and cooling back to martensite;
3. straining bend twist by (up to 10% for TiNi) to form strained martensite;
4. heating back to austenite and the alloy reverts to its memory shape. Cooling, deforming, etc, as often as it is neccesary;
5. The SMA process adapted to an actuator configuration by using the wire resistance to generate the heat necessary to recover the memory shape effect [2].

For this particular experiment, the steps of procedure used for obtaining two kind of TiNi wire samples having a near-equiatomic composition, in order to acquire shape memory properties are the following:

1. Thermal treatment first step: hartening from 750 °C for 25 minutes in water,
2. Thermal treatment second step: artificial aging according to table 1.

Table 1

Aging heat treatment matrix

Sample no.	Temperature [°C]	Diameter [mm]	Heating rate [°C/ s]	Time [min]
1.	450	0.48	5	45
2.	510	0.48	5	45
3.	450	1.08	5	45
4.	510	1.08	5	45
5.	450	0.48	25	45
6.	510	0.48	25	45
7.	450	1.08	25	45
8.	510	1.08	25	45
9.	450	0.48	5	75
10.	510	0.48	5	75
11.	450	1.08	5	75

12.	510	1.08	5	75
13.	450	0.48	25	75
14.	510	0.48	25	75
15.	450	1.08	25	75
16.	510	1.08	25	75

For these treated samples the electrical resistivity was measured using the four contacts method with Keithley instruments and thermal analysis measurements using a Netzsch 200 F3 Maia Differential Scanning Calorimeter. Keithley instruments have an data interface that allows saving datas measured every second while low electric currents are applied to the samples.

3. Results and discussions

After the thermal treatment all the 16 samples presented the shape memory effect (SME). The thermal properties analysis (DSC) was performed in both senses during increasing and afterwords decreasing of temperature, in order to show those important temperatures for this type of materials (SMA):
1. the austenite start temperature (As) and the austenite finish temperature (Af) during heating ;
2. the martensite start temperature (Ms) and the martensite finish temperature (Mf) during cooling [1].

Table II

Experimental results of the DSC and electrical resistivity measurements

Nr. crt.	T, [°C]	D, [mm]	v, [°C/s]	t, [min]	M_f [°C]	M_s [°C]	R_{frac} [°C]	R_{srac} [°C]	R_{sinc} [°C]	R_{frac} [°C]	A_s [°C]	A_f [°C]	ρ [$\cdot 10^{-7}$ Ωm]
1.	450	0.48	5	45	-43.3	-28.7	15	29.2			25.8	36.3	8.05
2.	510	0.48	5	45	-38	-30.5	-5.9	13.6			9.7	21.1	7.49
3.	450	1.08	5	45	6.4	33.1					18.4	41.1	9.91
4.	510	1.08	5	45	-44.9	-30.1	-10.9	11.7			11.1	29	8.45
5.	450	0.48	25	45	13.9	31.4					17.5	26.8	7.95
6.	510	0.48	25	45	-36.4	-30.4	-7.7	16.4			7.1	20.7	7.67
7.	450	1.08	25	45	-49.5	-35.6	10.8	34			19.4	32.2	8.56
8.	510	1.08	25	45	-9.2	43.8			26.1	0.3	29.5	48.3	8.19
9.	450	0.48	5	75	-44.9	-31.4	17.4	33			25.5	34.8	8.58
10.	510	0.48	5	75	-36.6	-23.7	-4.1	15.6			13.2	21.8	8.26
11.	450	1.08	5	75	14.6	40.4					22.5	47.8	8.13
12.	510	1.08	5	75	12.2	53.3					31.3	63.7	7.97
13.	450	0.48	25	75	10.7	32.5					18.5	29.7	8.21
14.	510	0.48	25	75	-35.3	-24.8	-3.9	15.8			11.2	22.6	7.78
15.	450	1.08	25	75	-32.7	-22.5	-6.5	14.1			14.3	26.4	8.69

16.	510	1.08	25	75	-15.9	42.6			27.2	-1.1	27.2	64.3	9.00

SMA have hysteretic behaviour, which is illustrated by the behaviour of the used samples shown in Fig. 2. For the memory shape alloys that present thermodynamically reversible transformations, those temperatures are stable and reveal important structural changes that happen in crystalline organization of the atoms. All those changes are reflected also in the physical properties of the samples. The first step in characterizing a material presenting SME is to determine the characteristic transformation temperature. An intermediate phase (R phase) sometimes appears during cooling, having its own start temperature (R_s) and finish temperature (R_f), before the transformation proceeds to martensite at lower temperatures. Under stress-free conditions, these are commonly measured by DSC thermograms [1].

DSC investigation of the samples revealed that the shape memory effect behavior for the samples is placed between 20 - 40˚C. So it means that the martensite – austenite reverse transformation that ensures the shape memory effect takes place in both senses within 20˚C and 40˚C (really easy to be achieved). Some samples present also the R phase as the DSC results showed.

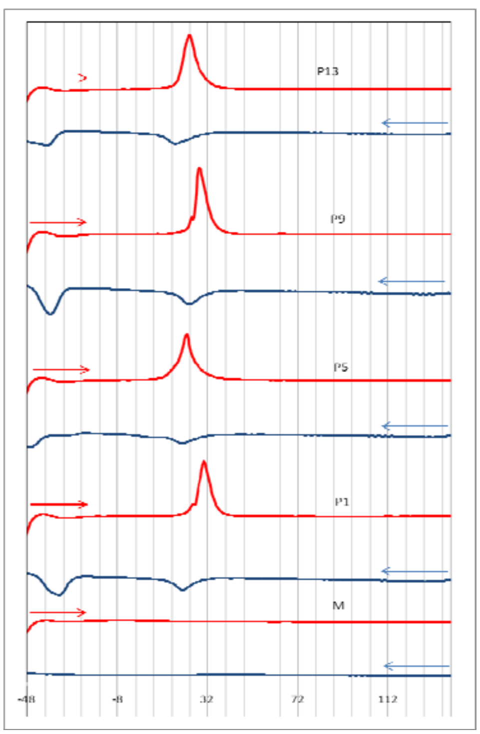

Fig.1. DSC curves compared for samples n° 1, 5, 9, 13

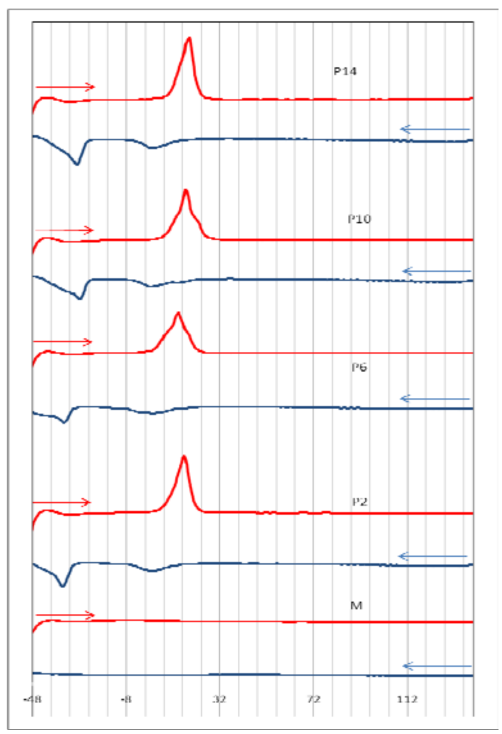

Fig.2. DSC curves compared for samples n° 2, 6, 10, 14

DSC is a recognized accurate method to determinate critical temperatures of shape memory alloys [1]. These temperatures for all the 16 investigated samples are presented in table 2 toghether with the electrical resistivities around 20°C. Figs. 1 and 2 allow the comparison of the evolution of the critical temperatures for the thin wire samples in order to reveal the aging treatment temperature and its duration influences upon them . Ageing temperature variations thus reveal to be a mean of readjusting the transformation temperatures to needed values, of course, in a reasonable temperature interval. However, we can see that R_s varies within a range of 15 degrees. This is due, most certainly, to sample cutting that induces local deformations, leading to a raise of R_s values. The raise of the ageing maintaining time (from 45 min to 75 min) leads to a slight growth of the R_s values.

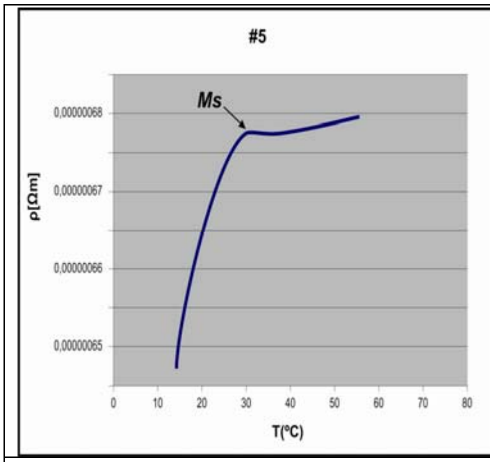

Fig.3. Electrical resistivity evolution at cooling for the sample no.5 reveals the M_s temperature

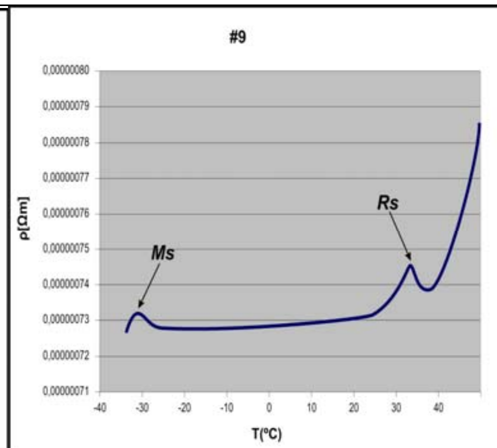

Fig.4. Electrical resistivity evolution at cooling for the sample no.9 reveals the M_s and R_s temperatures

The measurements of the electrical resistivity using the four contacts method (recommended in case of measurements of small values of electrical resistance and resistivity) revealed the following:

- the electrical resistivity of the samples increases when the temperature decreases (Fig. 3);
- the measured resistivities are between $7{,}49\cdot10^{-7}$ Ω·m measured for the sample no.2 and $9{,}91\cdot10^{-7}$ Ω·m measured for the sample no. 3 at a temperature of 28-30ºC;
- the electrical resistivity decreases slowly during the measurements, dew to the low electrical currents involved in the measurements (this influence is

strongly reduced when using the Keithley instruments that are very precise and per formant-Fig. 4).

- the electrical resistivity measured at zero degree Celsius of the samples present smaller values than the one for the untreated sample $8{,}49 \cdot 10^{-7}\ \Omega \cdot m$ (Fig. 5).

A good correspondence between the results of the DSC measurements and electrical resistivity ones can be noticed. The differences between the results of the two methods of measurement are not important and are due to the fact that samples analised through the DSC method must be small and cutting them induces strong stresses that can affect their microstructure and as a consequence of their critical temperatures too.

3. Conclusions

TiNi alloys present high resistivity, stable transformation temperatures that are easy to achieve, making them appropriate for different uses: actuators, sensors, microcontrollers, medical stends, etc. The basic rule for electrical actuation is that the temperature of complete transformation to martensite M_f , of the actuator, must be well above the maximum ambient temperature expected. This condition is respected by all the used samples in the present experiment, meaning that they can be heated using not very high electrical currents.

Measurements were made in order to enlarge the applications of these alloys in the electrothechnical field based on an accurate description of their behavior.

Shape memory actuators are considered to be low power actuators and such as compete with solenoids or bimetallic contacts. The use of shape memory alloy can sometimes simplify a mechanism or device, reducing the overall number of parts, increasing reliability and therefore reducing associated quality costs. TiNi is an expensive alloy, but because its high corrosion resistance, great biocompatibility, relative low shape memory transformation temperature, high melting point, and convenient resistivity it provides large perspective to applications in building electrical actuators or sensors, in substituting bimetallic contacts or building electrical connectors.

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