MECHANICAL BEHAVIORS OF TiNi BASE ALLOYS SUBMITTED TO DIVERSE THERMAL TREATMENTS

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Abstract. *This work aims to accomplish an analysis of the Mechanical behavior of TiNi base alloys with form memory effect. These alloys are TiNi wires with different diameters. The mechanical behavior will be analyzed by shipments, traction assays, and hardness. The used wires are commercial and had been submitted to different thermal treatments with the purpose of studying the thermo-mechanical process and its influences in the thermo elastics properties of the form memory effect. The wire samples had been cut in pieces, weighed and submitted to the thermal treatment at 300°C, 400°C, 500°C and 600°C quenched in water at 25°C. The time duration of thermal treatments had been: 1, 2, 4, 8, 12 and 24 hours. After this procedure, assays in a distinguishing calorimeter of scanning had been carried through, with the following parameters of scanning: two thermal cycles in the interval of temperature between - 40°C and 110°C, and of - 60°C and 90°C. There had also been carried through, traction assays for wires. The temperatures of phase transformation, hysteresis and enthalpies of transformation, as well as the influences of the re-crystallization in these properties will be analyzed.*

Keywords*: Differential scanning calorimetry, TiNi alloys, Shape memory effect.*

1. INTRODUCTION

Research involving intelligent materials has instigated some applications in the most diverse areas of knowledge (from medical area to aerospace industry). The most used materials in intelligent structures are the alloys with shape memory effect (Campista, 2005). The basic principle of the functioning of the effect form memory is in the fact of the occurrence of change of phase in the solid state without the occurrence of atomic diffusion. This change is known as martensitic transformation, in which two phases are able to coexist, martensite and austenite. The so called low-temperature martensitic phase possesses a conjugated monoclinical structure and a low modulus of elasticity and the austenitic phase is the high temperature matrix phase, which possesses a homogeneous crystalline cubical structure and a relatively high modulus of elasticity. Although many sources quote an additional phase of annealing and another intermediate phase R, austenitic and the martensite are the main components. The transformation of the phases in the Ti-Ni alloy had been extensively studied with great emphasis of the Ti-Ni alloys with compositions closer to the equiatomic. This work aims to analyze the behavior of the NiTi alloy with 1,27 mm of diameter with composition close to equiatomic under several times of aging.

2. METHODOLOGY

In order to accomplish these experiments commercial Ti-Ni wires with 1.27mm diameters and composition near equiatomic had been used, provided by the company Memory Metalle. These wires had been cut in small pieces, weighed in a scale of precision Mettler Toledo PL303 and submitted to the thermal treatment at 400°C and quenched in water at 25°C. After this procedure one had accomplished test in a differential calorimeter of scanning of the Mettler brand, model 823e, with the following parameters of scanning: thermal cycles in the interval of temperature between - 40°C and 110°C, using a 10°C/min rate (Paula, 2003). The transformation temperatures of phase, hysteresis and enthalpies of transformation, as well as the influences of the re-crystallization in these properties had been analyzed. Then new thermal treatments had been effected, in the same conditions of the previous one, and the samples had been analyzed in a diffractometer of x-rays of SHIMADZU model XRD 600 brand using Cu-Kα radiation with wave length of λ = 1,54060 Å. The diffractometry of x-rays were applied in the samples in order to verify the variations or modifications of the specters for the diverse used thermal treatments in this work.

3. RESULTS AND DISCUSSION

The peaks A, R and M indicate the transformations of parent B2 phase into rhombohedral premartensite R – phase, R – phase into maretensite B19’ phase and B19’ phase into B2 phase, respectively (Wu et al, 1996). These graphs express the results regarding temperature. The results of the DSC (figure 1), show that the alloy whit shape memory effect of Ti-Ni with composition near-equiatomic and diameter of 1,27 mm display direct and reverse transformations in some stages (B2↔R, B2↔B19', R↔B19'). The alloy used in the experiment when tested in the DSC in the form as received from factory presents two distinct peaks in the cooling (figure 1), indicating the presence of R - phase and the martensitic phase.

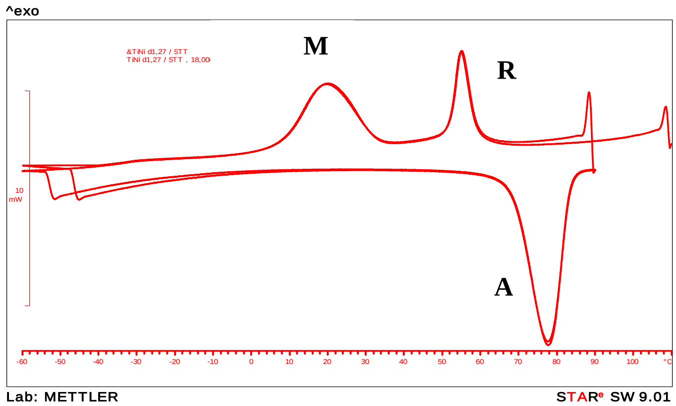

Figure 1 – Analysis of DSC in the sample of the Ti-Ni alloy without thermal treatment

How much it suffers to a thermal treatment during some hours (1 and 2 hours) it continues displaying two peaks in the cooling, but closer to the other (figures 2 and 3 respectively).

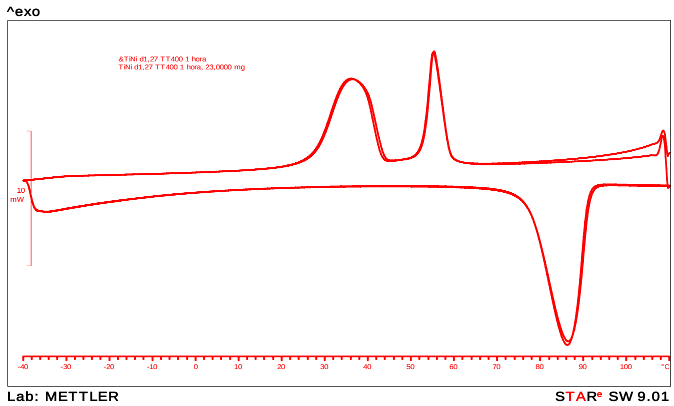

Figure 2 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 1 hour

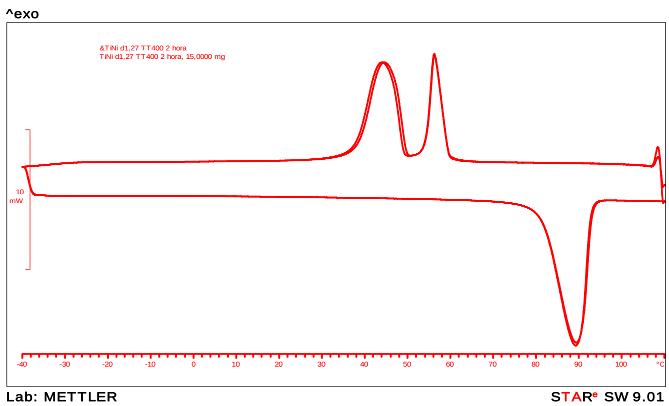

Figure 3 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 2 hours

For thermal treatment with a higher time, 4 and 8 hours, they still appear two peaks in the cooling, but there is a narrowing and greater approach of the peaks (figures 4 and 5 respectively).

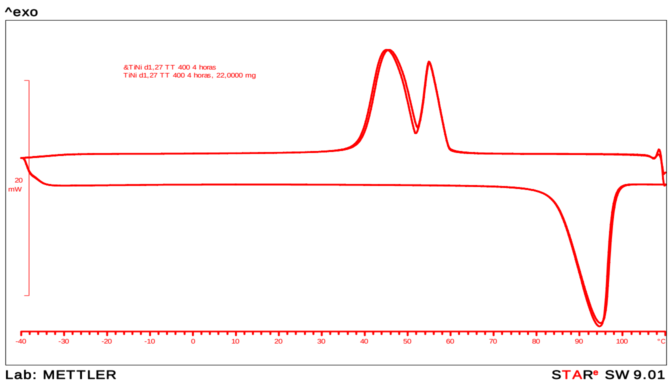

Figure 4 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 4 hours

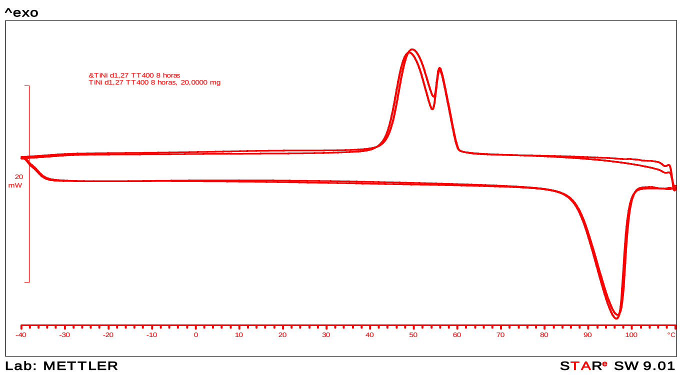

Figure 5 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 8 hours

For thermal treatment with a highly extended amount of time, 16 and 24 hours, only one peak is displayed in the cooling, there is an overlapping of the from previous treatments, that is the peaks that represent the R phase and the martensitic phase (figures 6 and 7 respectively).

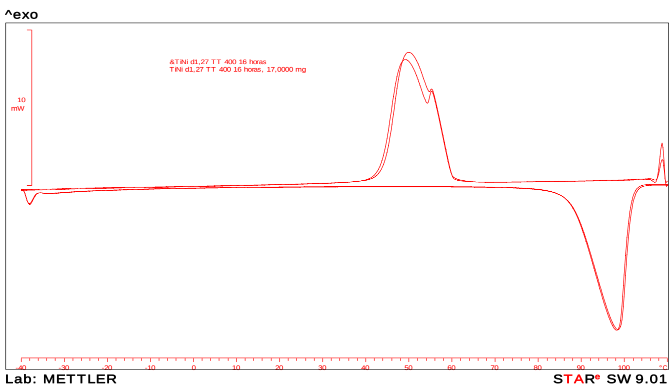

Figure 6 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 16 hours

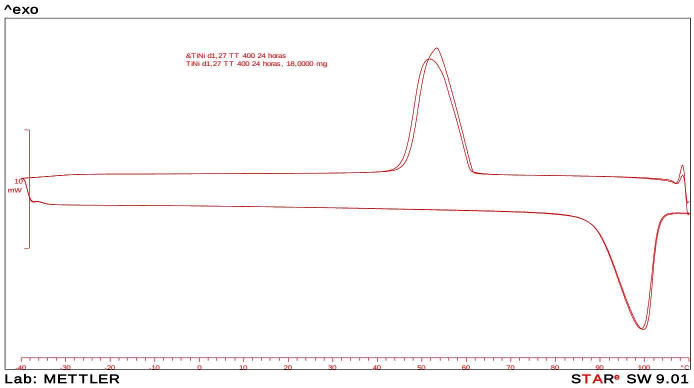

Figure 7 – Analysis of DSC in the sample of the Ti-Ni alloy treated at 400+25 during 24 hours

The table below represents the transformation temperatures (A_S, A_f, R_S, R_f, M_S and M_f) of the Ti-Ni wire of 1,27 mm of diameter treated at 400°C during 1, 2, 4, 8, 12, 16 and 24 hours.

Table 1 - Transformation temperatures of the Ti-Ni wire of 1,27 mm of diameter treated at 400°C +25

TEMPO	A_S	A_F	R_S	R_F	M_S	M_F
1 hora	79,9°C	93,8°C	59,0°C	51,7°C	45,8°C	30,0°C
2 horas	82,1°C	95,8°C	59,3°C	52,1°C	48,0°C	34,0°C
4 horas	84,1°C	98,1°C	59,3°C	51,8°C	50,3°C	36,8°C
8 horas	85,7°C	97,3°C	60,0°C	53,8°C	53,8°C	42,0°C
16 horas	90,0°C	103,6°C	61,0°C	55,0°C	55,0°C	44,4°C
24 horas	88,0°C	100,6°C	60,2°C	60,2°C	46,0°C	44,8°C

The diffractometry of X-rays was applied in the samples in order to verify the variations or modifications of the specters for the diverse thermal treatments used in this work. X-ray diffraction is developed in materials samples in two condition: as-receive and heat treated at 400°C. The analyses of the diffractographs had been carried through considering the following comments of the diffraction rays: displacement, intensity, narrowing or widening. Literature comments say that ageing at elevate temperatures may results in TiNi decomposition to obtain precipitates likes Ti_3Ni_4, Ti_2Ni_3 and $TiNi_3$. As the annealed time is raised the decomposition results in more simples elements like $TiNi_3$ and TiNi (Otsuka et al, 2005; Melton, 1990). For low temperatures and short times decomposition riches Ti_3Ni_4 and more elevated temperatures decomposition riches $TiNi_3$. Ti_3Ni_4 are important because it can improve shape memory properties and like $TiNi_3$ it is involved with R-Phase formation, acting as nucleation center for R-phase formation.

The diffraction of X-rays in figure 8 display the results for the Ti-Ni sample without thermal treatment. In this diffractograph there is a predominance of precipitated Ni_3Ti_2 and Ti_2Ni, which are responsible for the sprouting of R phase in the NiTi alloys with composition close to the equiatomic.

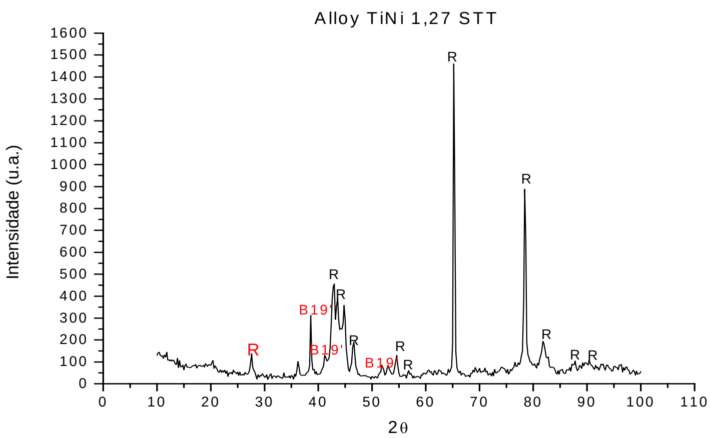

Figure 8 – Diffraction x-ray of the Ti-Ni alloy without thermal treatment

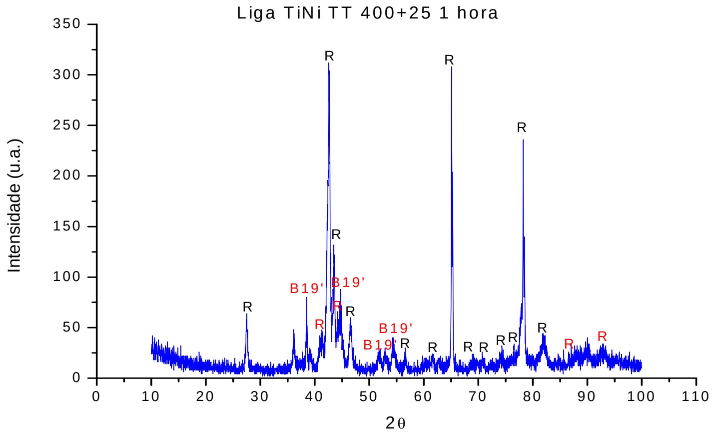

Figure 9 – Diffraction x-ray of the Ti-Ni alloy with thermal treatment 400+25 during 1 hour.

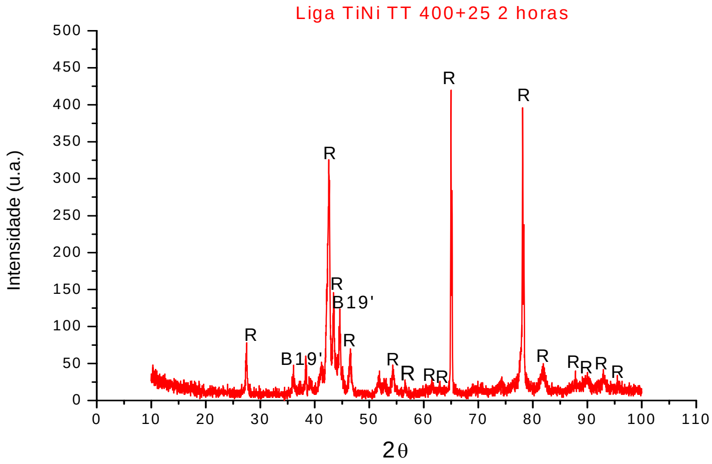

Figure 10 – Diffraction x-ray of the Ti-Ni alloy with thermal treatment 400+25 during 2 hours

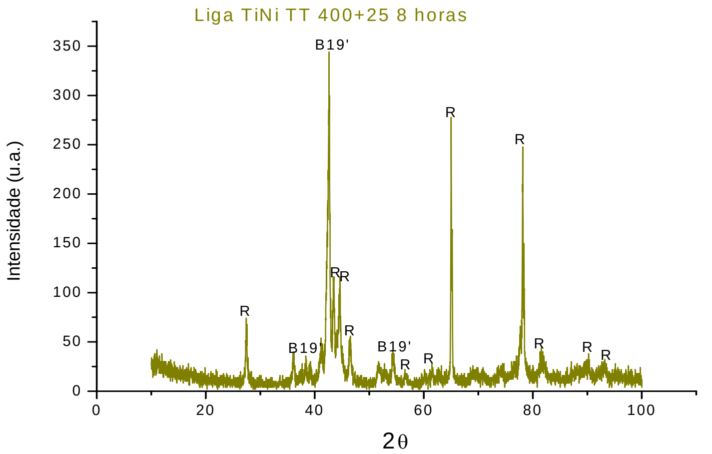

Figure 11 – Diffraction x-ray of the Ti-Ni alloy with thermal treatment 400+25 during 8 hours

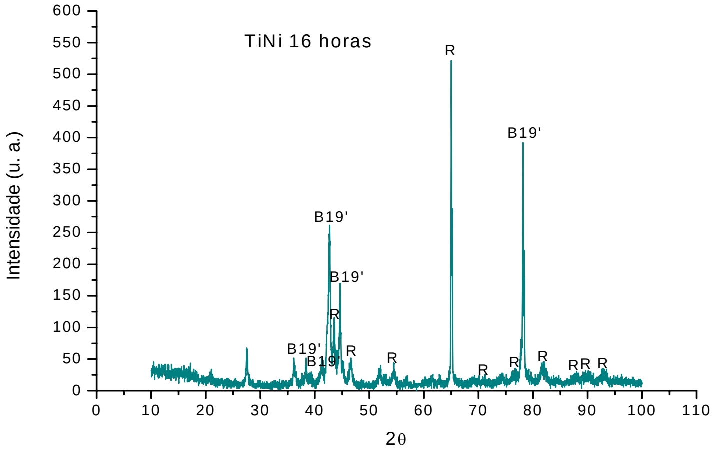

Figure 12 – Diffraction x-ray of the Ti-Ni alloy with thermal treatment 400+25 during 16 hours

The diffraction of x-ray in figure 13 display the results for the Ti-Ni sample with thermal treatment at 400+25 during 24 hours. In this diffraction there isn't any predominance of precipitated Ni_3Ti and Ni_4Ti_3, which are the responsible ones for the sprounting of R phase. There is a great predominance of the B19' phase which is the martensite transformation, this is the same as saying that the precipitated ones had been eliminated by the greatest time of thermal treatment the alloy suffered. Therefore in figure 7 there is only one peak of transformation in the cooling.

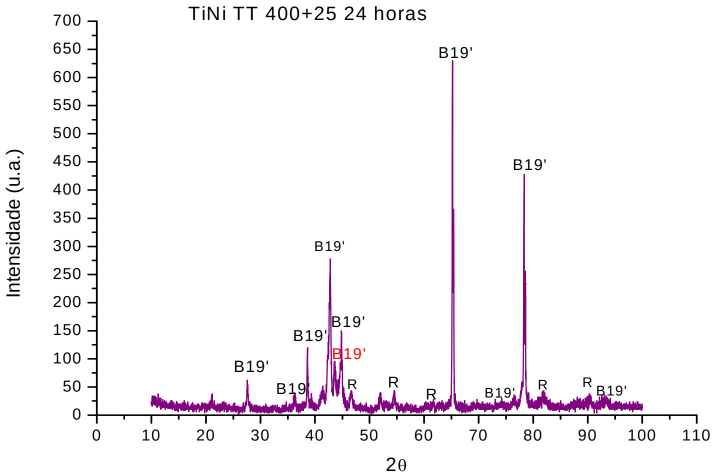

Figure 13 – Diffraction x-ray of the Ti-Ni alloy with thermal treatment 400+25 during 24 hours

4. CONCLUSIONS

Heat Treatments applied in the Ti-Ni commercial wire produce changes in the martensitic transformation thermo-elastic properties. This observation is due to the precipitates formed during heat treatment procedure.

The results of the DSC (fig. 1), show that the alloy whit shape memory effect of Ti-Ni with composition near-equiatomic display direct and reverse transformations in some stages (B2↔R, B2↔B19', R↔B19').

X-ray diffraction proves the existence of Ti-Ni precipitates in samples as-receive and heat treated. Heat treatment in elevated temperatures facilitates decomposition of precipitates as Ti_3Ni_4 and Ti_2Ni_3. These elements are very important in R-phase transformation because they modify the shape memory effect response to external stimulus.

Increasing the temperature and the time of annealing the behavior of the league Ti-Ni is altered.

5. AKNOWLEDGEMENTS

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