

Martensitic transformation in Ti-Ni-Cu shape memory wires broken by thermal cycling under constant load

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Abstract:

In this paper, the physical properties of a Ti-45.0Ni-5.0Cu (at) alloy are studied after failure (rupture) by thermal cycling under constant load between 75MPa and 350MPa. Transformation temperatures measured by electrical resistance (ER) change and DSC techniques show a good agreement in the virgin specimens, however this is not the case for the failed material. In addition, for wires broken under stresses until 125MPa, the DSC peaks becomes sharper than the ones measured with the virgin wires. In all cases, the relative change of ER associated to the martensitic transformation in the failed wires was higher than the value measured in the virgin material. These results are discussed essentially considering the role of the oriented defects and internal stresses created in the wires during thermal cycling under constant load.

1. INTRODUCTION

It is well established that the thermoelastic martensitic transformation is at the origin of the shape memory phenomena in many alloys [1, 2]. Among these systems, Ti-Ni based alloys are considered the best because of their excellent shape memory characteristics and biocompatibility which are favorable for many potential applications [2]. In general, the martensitic transformation of Ti-Ni based alloys were well studied as a function of some typical thermomechanical treatments including solution treatment, annealing after cold working and aging after solution treatment [3-6]. In some cases, the fabrication of shape memory elements is carried out by cold working in martensitic state. After this procedure, the plastically deformed martensite shows a complex structure including dislocations, vacancies and stabilized martensite variants [5-7]. These non-oriented defects are responsible for a total or partial stabilization of martensite variants causing a reduction of the transformed fraction during heating of the material. As a consequence a decrease of the heat of transformation is observed as well as a decrease of the transformation temperatures (in relation to recrystallized state) after the first heating corresponding to the reverse transformation [8,9]. Additionally, it has been confirmed that recrystallized Ti-Ni specimens submitted to thermal cycling without applied stress exhibit a decrease in heat of transformation [4, 10] accompanied with a decrease of the martensitic transformation start temperature (M_s) [3,4] as a result of the introduction of dislocations. However, it is still lacking in literature some experimental results concerning the transformation of Ti-Ni based alloys plastically deformed by a particular thermomechanical treatment as, for example, thermal cycling under mechanical load. Thus, the major aim of this work is to clarify the transformation behavior of Ti-45.0Ni-5.0Cu (at%) specimen wires failed after a large number of thermal cycles under constant load.

2. EXPERIMENTAL PROCEDURE

The material used in this study were commercial Ti-45.0Ni-5.0Cu (at%) wires with 0.55mm diameter after cold drawing of about 40%. As received materials were heat treated at 425°C for 1h followed by cooling at room temperature. Table 1 shows the transformation temperatures and thermal hysteresis (H_t) related to the martensitic transformation under stress free conditions obtained by Electrical Resistance (ER) change and DSC (Mettler TA3000) measurements. The stability of these thermal properties as a

function of the number of thermal cycles was also verified and the superposition of 20 cycles between 0 and 110°C has confirmed an excellent thermal stability of the transformation characteristics in absence of external stresses. In addition, a very weak increase of electric resistance in martensitic (measured at 10°C) and austenitic states (measured at 150°C) indicates that probably there is no introduction of defects into the material [11].

Table 1 – Thermal properties of the heat treated Ti-Ni-Cu wires (here called virgin material).

Technique	M_f (°C)	M_s (°C)	A_s (°C)	A_f (°C)	H_t (°C)
DSC	31.5	39.0	47.5	57.5	17.5
ER change	30.0	39.0	46.0	58.5	18.0

The Ti-Ni-Cu wires were tested during the thermal cycling under constant load (uniaxial tensile) mode with an apparatus presented in a previous paper [12] and originally designed for fatigue tests. Specimens having a gauge length between 25 and 30mm were loaded by weights in martensitic phase, then heated above A_f and the temperature cycling was started. Constant applied stresses were set between 75 and 350MPa. For all stress levels, temperature cycles were carried out between 10°C and 154°C measuring at the same time the deformation and ER change of the wire as function of the temperature. The wire specimens were first pre-cycled during 20 cycles and afterwards tested until failure by a special logarithmic law of cycling involving classical thermal cycles by forced convection in a silicone oil medium and electrical cycles by Joule effect in the same environment maintained at low temperature (~10°C) [12].

3. RESULTS

The specimens wires were thermally cycled under load (75-350MPa) until fracture. The evolution of the deformations in the martensitic ($\epsilon_{10^\circ\text{C}}$) and austenitic ($\epsilon_{154^\circ\text{C}}$) states for 175MPa as well as the fatigue life of the wires are shown in figure 1(a) and 1(b), respectively. The stress assisted two-way memory effect (SATWME) is calculated in figure 1(a) as the difference between $\epsilon_{10^\circ\text{C}}$ and $\epsilon_{154^\circ\text{C}}$. The progressive accumulation of these deformations with cycling is proportional to the applied stress and characterize a creep deformation of the wire. It is observed in figure 1(a) that failure occurs suddenly without degradation of the SATWME immediately before fracture. The Wohler curve of the wires is pointed out in figure 1(b).

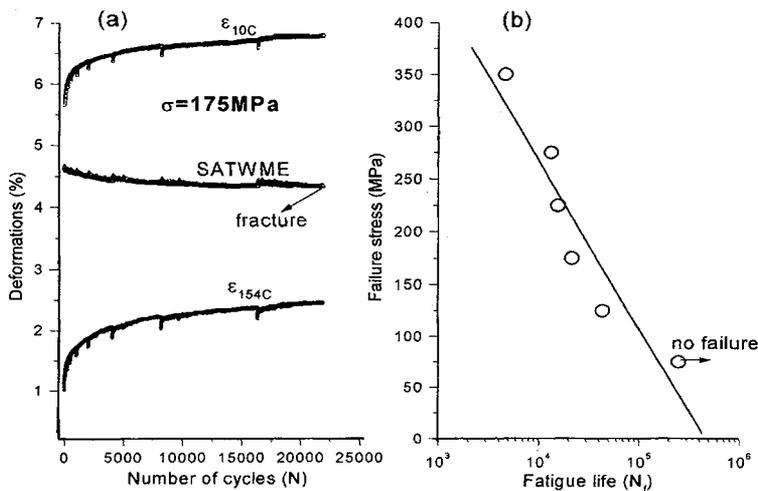


Figure 1 – Some thermomechanical properties of the Ti-Ni-Cu wires during cycling. (a) deformations as a function of the number of cycles for applied stress of 175MPa. (b) fatigue life as function of the applied stresses.

Specimens broken under various stress levels were analyzed by DSC and ER change measurements. Figure 2 shows the shape of the DSC peaks during cooling (fig.2a) and heating (fig.2b) between 0 and 120°C as a function of the applied stress. It is clear that there is a displacement of the peaks towards the higher temperatures when broken specimens are compared to the virgin material (0MPa). This effect exists for all applied stress levels, but this is more important for lower stresses like 125MPa. In addition, the peaks measured during cooling and heating for these lower stresses have intensities higher or close to the ones of the virgin material. Broadening of the peaks is observed for stresses higher than 125MPa.

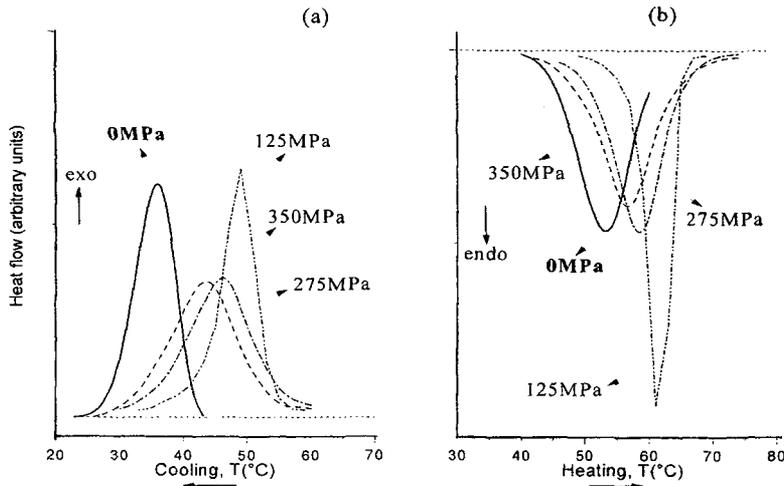


Figure 2 – DSC peaks of the specimens failed under different stresses. (a) forward transformation during cooling. (b) reverse transformation during heating.

Figure 3 shows the ER loops for two different failure stresses in comparison with the behavior measured with the virgin wire in absence of external stress. From this figure, the ER change ($\Delta R/R$) associated to the martensitic transformation is larger in the failed wires in relation to the unloaded virgin material. This phenomenon is followed by a displacement of the ER loops towards the higher temperatures similarly to the results obtained by DSC (figure 2). Additionally, a reduction of thermal hysteresis is also observed. During cycling ($N \sim 50$) of the failed wires in absence of external stress it has been observed a downwards displacement of the ER loops shown in figure 3 [11]. This behavior characterizes a progressive decreasing of the electrical resistivity of the two phases (martensite and austenite) during cycling. However, it was confirmed that thermal properties, like M_S and thermal hysteresis, are almost unaffected by cycling.

A comparison between the M_S temperature measured by DSC and ER change is realized in figure 4(a), while figure 4(b) shows the heats of transformation measured by DSC during the forward (ΔH_{A-M}) and reverse (ΔH_{M-A}) transformation. These figures confirm an increase of the M_S temperature and a gradual reduction of the heat flow for failure stresses higher than 125MPa.

4. DISCUSSION

According to figure 1(a), there is a progressive accumulation of plastic deformation under load during fatigue cycling. A fraction of this deformation remains after failure and therefore the wires are work-hardened during the repeated transformation cycles under load. The defects associated to this work-hardening

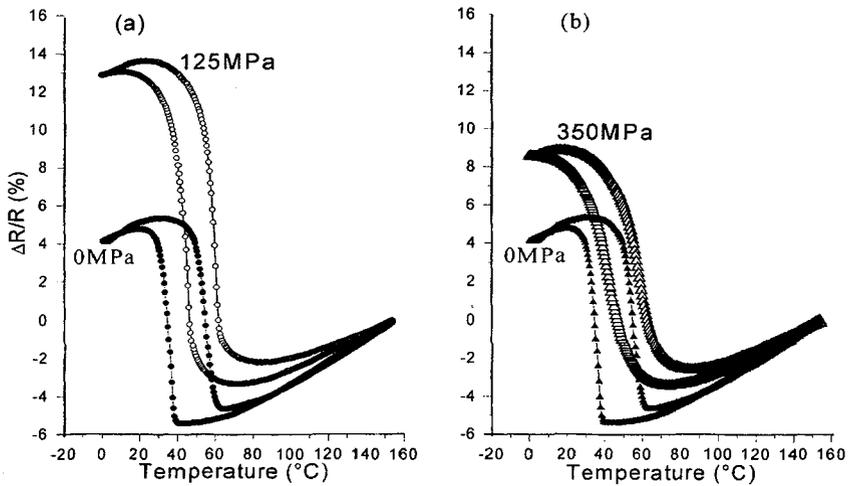


Figure 3 – ER loops after rupture for some applied stress levels. (a) 125MPa. (b) 350MPa.

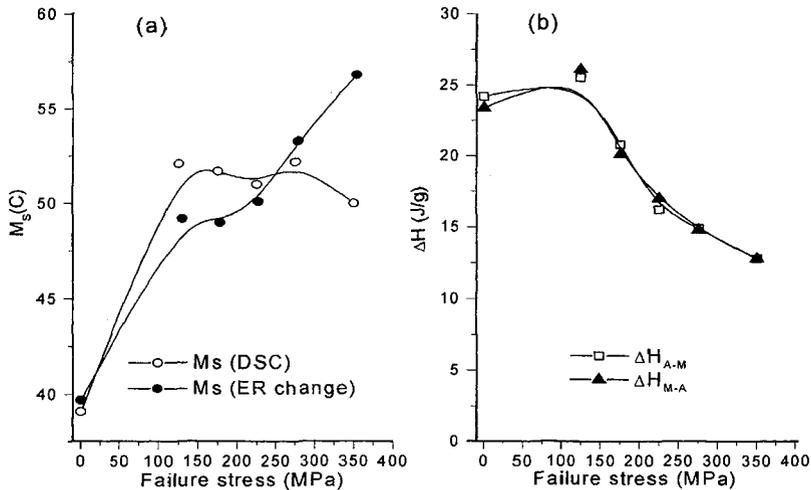


Figure 4 – Thermal properties of broken wires as a function of the applied stress. (a) M_s temperature measured by DSC and ER change. (b) enthalpies associated to the forward and reverse transformation.

ning process should be well oriented by the uniaxial tensile stress applied upon the wire. This phenomenon is responsible by the creation of an internal stress field into the material [13].

The broadening of DSC peaks (fig. 2) cause a decrease of the transformation enthalpies in failed wires (fig.4b). This behavior is similar to the one observed in Ti-Ni based alloys plastically deformed (until 40%) in martensitic state [6,8,9] and also in recrystallized Ti-Ni alloys submitted to thermal cycling in absence of applied stress [10]. On the other hand, figure 4(a) confirms that there is always an increase of M_s after rupture of the wires by thermal cycling under constant load. This augmentation of M_s indicates that the internal stress field created by the oriented defects assist the transformation during cooling. This is not the case when defects are introduced by plastic deformation of the martensitic and/or austenitic phase where a decrease of M_s is verified [8,9]. However, the production of defects by the martensitic

transformation is not the more important phenomenon. Apparently, the presence of the mechanical load during thermal cycling plays a major role because the M_s temperature of the virgin wires are unaffected by thermal cycles under stress free conditions [11]. In addition, it is important to notice that the M_s temperature of recrystallized Ti-Ni based alloys decreases during thermal cycling without applied stress [3,4]. Miyazaki *et al.* [4] has demonstrated that this diminution of M_s in recrystallized specimens is due to the introduction of a complex arrangement of dislocations created by the transformation cycles. It is clear that these non-oriented dislocations creates internal stresses that can suppress the martensitic transformation similarly to the effect of the plastic deformation of the stable phases during cold-work [6,8,9].

In addition to the observations above delineated, it is necessary to remark the transformation behavior obtained by ER measurements (fig.3). As pointed out in figure 3, broken wires show a variation of ER during transformation higher than the one verified in virgin wires (0MPa). Recently, De Araújo [11] has realized coupled measurements between SATWME and ER change. These measurements have demonstrated that the variation of electrical resistivity (after elimination of the strain contribution) associated to the transformation under load has been always higher than the one of the unloaded material. This is in agreement with others authors [14,15]. Consequently, electrical resistivity change due to formation of martensite variants oriented by the stress is larger than the one associated to the formation of self-accommodated variants. Thus, figure 3 seems to indicate that, in broken wires, there is formation and growth of martensite variants preferentially oriented by the internal stress field created during the fatigue process, contrary to the formation of self-accommodated variants in the virgin specimens. Figure 4(a) reveals a significant difference between M_s temperatures after rupture as measured by DSC and ER change. It must be precised that the length of the specimens used in ER measurements are about 5 times larger than the ones used in DSC, this behavior seems to indicate that there is a non-uniform distribution of defects along of the ER specimens, contrary to the fracture region used in DSC which given a M_s practically independent of the failure stress.

5. CONCLUSIONS

After fracture, the failed wires are trained by the thermal cycling under constant load process. Contrarily to the results found in classical processes of plastic deformation of SMA's, it was here confirmed that the reduction of the heat of transformation associated to the introduction of defects into the material is always accompanied by an increase of the M_s temperature. In addition, these defects being oriented by the applied stress, seem to assist the formation and growth of preferentially oriented martensite variants which are responsible for a higher variation of ER in comparison with the one due to the formation of self-accommodated variants in virgin wires. These highest values of M_s and variation of ER in failed material seem to confirm the existence of an internal stress field that assist the forward martensitic transformation.

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