ISSN 1318-0010
KZLTET 31(6)539(1997)

TWO-WAY SHAPE MEMORY EFFECT AND ITS DEGRADATION DURING THERMAL CYCLES IN Ni-Ti ALLOYS

DVOSMERNI SPOMINSKI EFEKT V Ni-Ti ZLITINAH IN NJEGOVA DEGRADACIJA MED TOPLOTNIMI CIKLI

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Prejem rokopisa - received: 1997-10-01; sprejem za objavo - accepted for publication: 1997-10-21

This work presents a study of the degradation of the Two-Way Shape Memory Effect (TWSME) due to working cycles. An intrinsic TWSME was induced in wire specimens of a near equiatomic Ni-Ti alloy by thermomechanical training. The development of the two-way strain was analysed and discussed with respect to the different training parameters and the preliminary heat treatment of the samples. Subsequent to the training, the stability of the obtained TWSME was examined by thermal cycling under stress-free conditions. Possible reasons and mechanisms for the degradation of the effect during thermal cycling are discussed.

Key words: shape memory, two-way-effect, Ni-Ti alloy, stability, martensitic transformation, fatigue

V delu se predstavlja raziskava degradacije dvosmernega spominskega efekta (TWSME) zaradi delovnih ciklov. Intrinsičen TWSME je bil induciran v žice iz equiatomske Ni-Ti zlitine s termomehanično obdelavo. Razvoj dvosmerne deformacije je bil raziskan in ocenjen glede na obremenilne in predhodno toplotno obdelavo vzorcev. Po obremenitvah je bila stabilnost dosežene TWSME preiskana s toplotnim cikliranjem brez napetosti. Razpravlja se o možnih vzrokih in mehanizmih degradacije efekta med termičnim cikliranjem.

Ključne besede: oblikovni spomin, dvosmerni efekt, zlitina Ni-Ti, stabilnost, martenzitna premena, utrujenost

1 INTRODUCTION

The term Shape Memory refers to the ability of certain alloys, to recover large strains perfectly either right after unloading (pseudoelasticity) or after heating (pseudoplasticity). Several alloys can show this phenomenon as a consequence of martensitic transformation[1]. In the case of the intermetallic phase Ni-Ti, such a transformation occurs between the high temperature modification (austenite) with a CsCl-type B2 superlattice and the low temperature phase (martensite) with a monoclinic B19' structure[2]. Furthermore, a TWSME may be obtained in these alloys after a suitable thermomechanical treatment, which is often termed training. This special behaviour refers to the ability to produce spontaneous shape changes on heating as well as on cooling, both without the application of external forces. Consequently, trained elements can directly be used as temperature-sensitive actuators. The actuators are usually electrically actuated and cooled by natural convection. One important criterion for the actual use of Shape Memory components in such multiple cycle applications is the stability of the functional parameters throughout application. A reliable component should exhibit constant transformation-temperatures and a two-way strain independent of number of cycles. In this work, the stability of an intrinsic TWSME during repeated thermal cycling has been investigated.

2 EXPERIMENTAL

The investigations have been carried out on a binary Ni-Ti alloy with a nominal composition of Ni-50,3 at% Ti. The material was melted from pure metals in an arc-furnace, hot extruded and cold-drawn with intermediate annealings. The as-received wire material was cold worked by 13,5% with a diameter of 3 mm. Wire specimens of 20 cm in length were prepared and some of them annealed at 550°C / 20 min. The transformation temperatures were deduced from resistivity measurements and are listed in **Table 1**. All transformation temperatures are above room temperature, with M_s, M_f indicating the start and finish temperature for the forward transformation, and A_s, A_f describing the reverse transformation. The mechanical properties of the alloy were investigated by tensile tests carried out at room temperature. The obtained stress-strain-curves of the martensitic phase are illustrated in **Figure 1**. The training procedure was simulated on a tensile testing machine. The specimens were fixed at room temperature, loaded with the constant training stress σ_{train} and repeatedly thermally cycled between M_f and a temperature above the highest temperature, at which martensite can be stress-induced (M_d). Heating was done by Joules-Effect, cooling by natural convection in air. According to the applied training stress σ_{train} and number of training cycles N_{train}, different magnitudes of the TWSME were obtained. Some of the values are given in **Table 2**. Subsequently to the training, the magnitude of the TWSME was measured

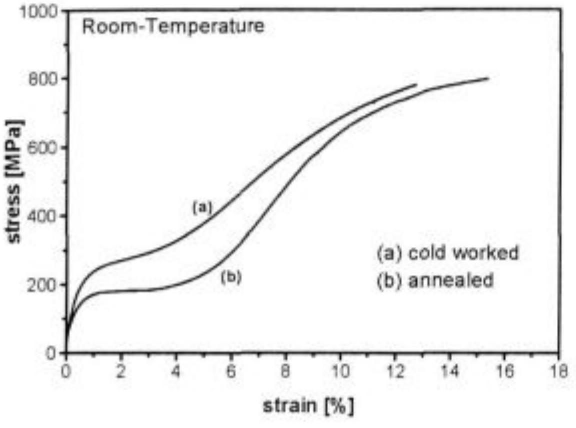

Figure 1: Stress strain curves of the martensitic modification

and its degradation during repeated stress-free thermal cycling studied.

Table 1: Transformation temperatures obtained by resistivity measurements

HT	M_s(°C)	M_f(°C)	A_s(°C)	A_f(°C)
none	52	38	82	141
550°C	55	42	84	127

Table 2: TWSME achieved with different numbers of training cycles and heat treatments

HT	σ_{train}	(MPa) N_{train}	ε_{2w} (%)
550°C	50	20	1,65
none	100	20	1,18
550°C	100	20	1,80
550°C	100	10	2,15

3 RESULTS AND DISCUSSION

It is very useful to study the strain temperature relationship in order to understand the shape memory behaviour. **Figure 2** illustrates the strain-temperature-curves as they have been recorded by microstrain measurement during the training cycles. On cooling the loaded wire specimen from a temperature above M_d, martensite is stress induced on reaching the M_s-temperature. Normally, the Ni-Ti martensite consists of 24 variants, that are crystallographically equivalent[3]. But the applied stress causes a preferred nucleation and growth of those variants, that have the highest compatibility with the strain-field. This orientation of the martensite results in a macroscopic strain in direction of the applied stress, as can be observed in **Figure 2**. On heating, the alloy transforms back to the B2 structure of the austenitic modification, forced to restore the associated original shape. A careful look on **Figure 2** shows, that the restoration does not succeed completely, but there is a small amount of irreversible strain. This irreversible strain consists as well of locally stabilised martensite as of true plastic strain[4]. During the growth of the martensite plates, stress concentrations may arise on the B2/B19' interfaces, locally exceeding the true plastic yield strength. Actually, this generation of dislocations during the thermomechanical training procedure is a precondition for the development of the two-way effect. The generated dislocation pattern is a characteristic feature of the deformed low-temperature shape, which is repeatedly formed during training. On subsequent stress-free thermal cycling, these dislocations are effective in guiding the martensite formation, causing the martensite to form at least partly oriented. The resulting two-way strain ε_{2w} has the same direction as the reversible strain during the training, but it is smaller in magnitude, as the efficiency of dislocations in the formation of a preferential martensite morphology is weaker compared to the stress field caused by an external force.

Furthermore, it can be observed in **Figure 2** that the temperature interval (A_f - M_f) increases throughout training. This is thought to be likewise due to the increase of the dislocation density. The increase of the internal stress that is connected with it reduces the mobility of the B2/B19' interfaces. As the martensitic transformation in near equiatomic NiTi occurs by the way that the martensite plates grow continuously on cooling and shrink on heating, the rise of frictional stress leads to an increase of A_f and a decrease of M_f.

The development of the TWSME can be observed best by studying the course of the different strains during training. **Figure 3** shows the labelling of the different strains describing a training cycle. If one follows the course of these strains throughout training, it becomes evident, that a high temperature and a low temperature shape is being established. The pseudoplastic strain ε_{pp} increases very fast during the first few cycles as a consequence of the stress-biased martensite formation. A certain σ_{train} is able to orient a certain amount of martensite variants. Consequently, ε_{pp} saturates after having reached a certain magnitude. To increase σ_{train} beyond a certain limit has no sense in the case of a thermomechanical training procedure as one obtains excessive irreversible strain, leading to a deterioration of the high temperature

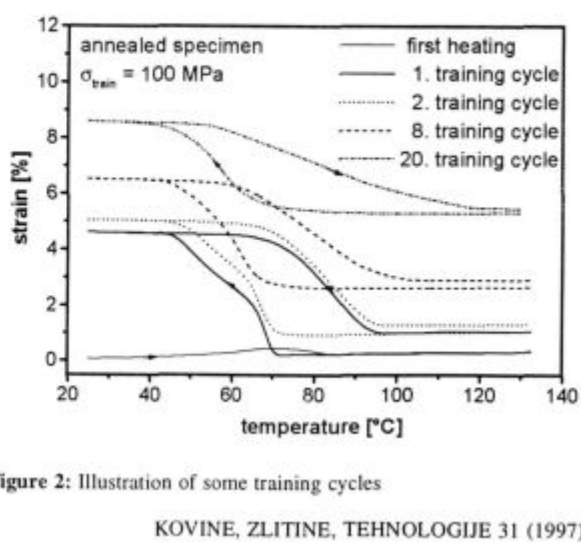

Figure 2: Illustration of some training cycles

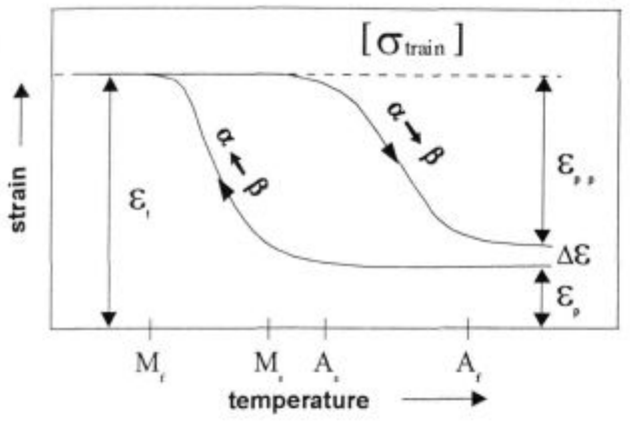

Figure 3: Schematic illustration of a training cycle indicating the labelling of the different strains: ε_t: total strain, ε_{pp}: pseudoplastic strain, ε_p: irreversible strain

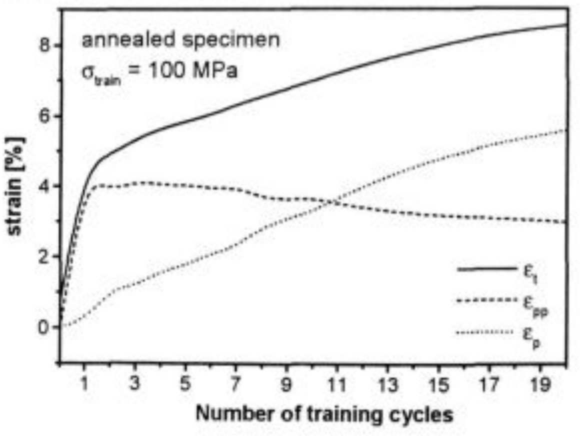

Figure 5: Development of the different strains during training, annealed specimen, 100 MPa

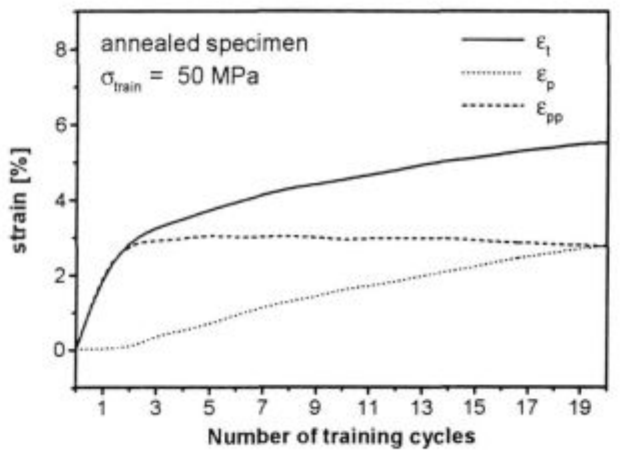

Figure 4: Development of the different strains during training, annealed specimen, 50 MPa

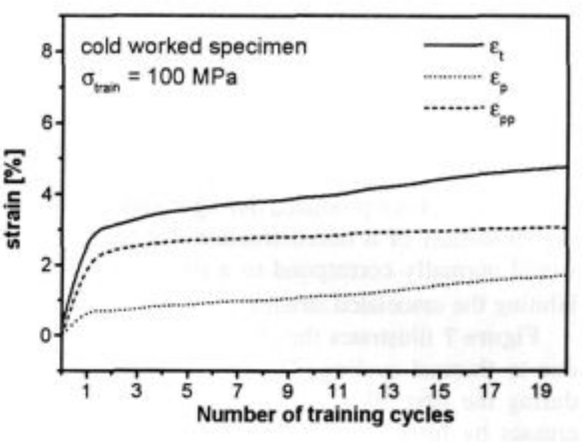

Figure 6: Development of the different strains during training, cold worked specimen, 100 MPa

shape. It has been shown by other authors that generally those training parameters (combination of training stress and number of training cycles) give the highest two-way memory, which result in the least permanent strain, while producing a full one-way strain[5]. The maximum transformation strain for a certain heat-treatment condition may be estimated from the extension of the martensite plateau in the stress-strain curve in **Figure 1**. As can be observed from **Figure 4**, 50 MPa is not enough to take advantage of the whole orientation capacity. The ε_{pp} in this plot is considerable smaller than the extension of the martensite plateau in **Figure 1**, which reaches about 4%. It is evident from **Figure 5** that an increase of σ_{train} to 100 MPa leads to a ε_{pp} of the aimed magnitude. It is interesting to note in this plot that the pseudoplastic strain decreases after having passed a maximum value after about 4 training cycles. Apparently, the irreversible strain rises too fast in the case of 100 MPa. This is quite important as it has been concluded elsewhere[5], that there exists a near proportionality between the pseudoplastic strain and the obtained two-way strain. This suggests that in order to maximise ε_{2w}, fewer than 20 training cycles should be applied. This is confirmed by the values listed in **Table 2**. In the case of σ_{train} = 100 MPa, the two-way strain obtained after 10 training cycles is larger than after 20 cycles. **Figure 6** shows the course of the strains for the work hardened specimen. Due to the high internal stresses, the σ_{train} is not as efficient in biasing the martensite formation as in the annealed specimens. A pseudoplastic strain of only 2,5% is reached and it has neither saturated nor passed a maximum after 20 training cycles. It may therefore be concluded that ε_{pp} would increase further, if training was continued.

In order to repeatedly achieve the same low temperature shape during stress-free thermal cycling it is essential, that the transformation follows the same transformation paths over and over again. The thermoelastic martensitic transformation occurs sequentially. Those martensite variants that have formed first, are the last to revert. A thermodynamic balance exists at all times on the B2/B19' interfaces between chemical and not chemical energy terms. The latter one is essentially the elastic

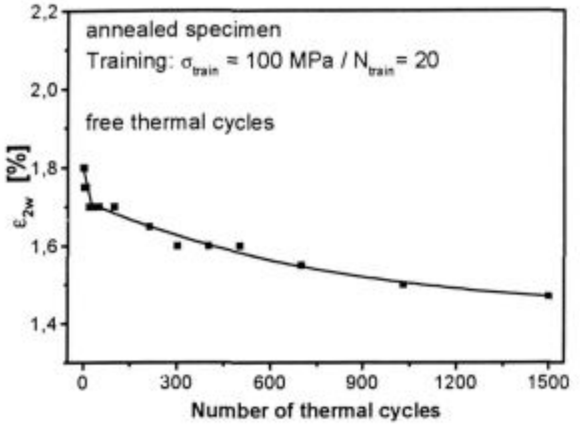

Figure 7: Degradation of the TWSME during thermal cycling, annealed specimen

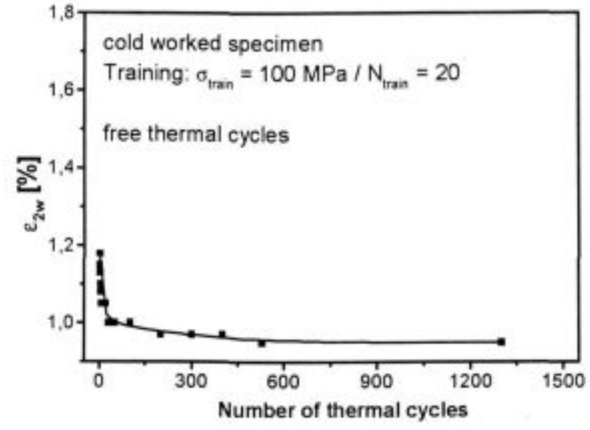

Figure 8: Degradation of the TWSME during thermal cycling, cold worked specimen

strain energy developed as the microstructural units of new phase nucleate and grow in the original phase[6]. By this energy term, the substructure can exercise some influence on the selection of variants, that are assisted in their nucleation and growth. In an asymmetric substructure, as it has been produced during training, this leads to the formation of a microstructure during cooling, that would normally correspond to a strained condition, exhibiting the associated strain ε_{2w}.

Figure 7 illustrates the change of the two-way strain due to thermal cycling. There is a rather strong decay during the first 50 to 100 cycles, which gradually decreases by further cycling. After about 500 cycles, the degradation rate becomes fairly small. Considering the importance of the asymmetric microstructure for the appearance of the TWSME, it appears very likely, that the decrease of ε_{2w} is the consequence of changes in the substructure. Investigations by Miyazaki et al.[7] showed, that thermal cycling causes a rearrangement and annihilation of existing dislocations as well as the introduction of additional ones. Both processes change considerably the dislocation pattern that has been created during the training cycles and will therefore lead to an increasing amount of unfavourably oriented martensite. Some hints about the actually controlling mechanism may be obtained by comparing the degradation of the annealed specimen in **Figure 7** to a cold worked one in **Figure 8**. While the initial decay cannot be avoided, the decrease in the latter stage shows a sensitivity from the initial dislocation density. In the annealed specimen, the two-way strain keeps degrading throughout the investigated range of 1500 thermal cycles whereas in the cold worked sample it stays almost stable. It may be suggested from this observation, that the degradation in this stage is primarily due to the introduction of dislocations. In the cold worked sample it is more difficult to generate additional dislocations as the dislocation density is already very high. This results in a more stable TWSME in the latter stage.

On the other hand, the sharp decay at the beginning of thermal cycling appears to be not affected by a higher dislocation density. Moreover, it seems to be a transient phenomenon which is limited to the first 50 to 100 cycles, reaching a saturation at the end of the initial period. It appears very likely that the decrease in this range corresponds to a continual change in the dislocation substructure - by rearrangement and annihilation - until a stable configuration, representative of the saturated state, is reached.

It should be further noted, that even on variation of the training parameters, the characteristic features of the degradation curve remain more or less the same. It becomes evident, that for the applied training procedure, the initial microstructure of the alloy influences the stability of the intrinsic TWSME far more than the training parameters.

4 CONCLUSIONS

With the chosen training procedure, a maximum two-way strain of 2,1% could be achieved. The induced intrinsic TWSME shows a sharp decay in the early stage of cyclic application. This is ascribed to the rearrangement and annihilation of dislocations during the first few thermal cycles until a new saturated state is reached. In the latter stage, the degradation rate is smaller and it shows a dependence from the preliminary heat-treatment. It is suggested that the controlling mechanism in this stage is the introduction of additional dislocations. For the practical point of view it may be concluded from this work, that trained shape memory elements should be cycled several times before application until the mechanism, responsible for the sharp decay in the initial stage, has been exhausted. The stability in the latter stage, especially in cold worked specimens, should be suitable for technical applications. The preliminary heat-treatment appears to be the crucial parameter to optimise the TWSME regarding magnitude and stability.

ACKNOWLEDGEMENT

The authors gratefully acknowledge financial support of this work by the "Jubiläumsfond der Österreichischen Nationalbank".

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