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Effects of different thermomechanical treatments on fatigue of NiTi shape memory alloys

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Abstract: Fully recrystallized NiTi sheets were deformed in four different temperature ranges: fully transformed martensite ($T < M_f$), transforming austenite ($M_s < T < M_d$), low temperature, stable austenite ($M_d < T < 1/3\ T_s$) and austenite at elevated temperatures ($T > 1/3\ T_m$). The types and distributions of defects produced in these materials depend on the deformation temperature. The characteristic defect structures resulting from the low temperature deformation lead to different microstructures even after annealing.
The thermomechanically treated as compared to the fully recrystallized NiTi alloys were subjected to thermal, mechanical and thermomechanical fatigue tests. Their microstructural origin was interpreted by TEM investigations.
In conclusion it is proposed that a low ratio of pseudo-yield stress to true yield stress is responsible for good fatigue resistance. Regarding fatigue it is better to increase the yield stress by a small grain size than by a high dislocation density (work hardening).

1. INTRODUCTION

For good SM-properties thermomechanical treatments are required. Defects like grain boundaries and dislocations increase the yield strength, which is decisive for high fatigue resistance.
The rolling process can be performed in four temperature ranges: range I: thermal martensite ($T < M_f$, martensite deformation = MF), range II: transforming austenite ($M_s < T < M_d$, mixed deformation = XF), range III: stable austenite ($M_d < T < 1/3\ T_m$, austenite deformation = AF) and range IV: warm austenite ($T > 1/3\ T_m$). The deformed microstructure depends on the deformed phase and the thermal activation. Therefore different types and distributions of defects are produced. In previous papers, the influence of rolling in specific temperature ranges on pseudo elastic and pseudo plastic behavior was reported:

- Range I (MF) and II (XF): Depending on the annealing conditions, favorable functional and structural properties were created [1, 2].
- Range III (AF): Only pseudo plasticity is improved. The criterion for good pseudo elasticity is a low ratio of pseudo yield stress to true yield stress. In case of strengthening due to rolling in range III, the pseudo yield stress is increased as well as the true yield stress [3].
- Range IV (hot rolling): Shape memory properties are not improved. The aim is to reach high thickness reduction. However, inclusions are oriented in rolling direction and the deformation texture is established. In case of particle stimulated recrystallization, the distribution of inclusions is very important for the results of the following thermomechanical treatments.

The motivation for fatigue experiments discussed in this paper is to reveal the best thermomechanical treatment for desired shape memory behavior. In case of pseudo elasticity, the difference between rolling in range I and II, followed by annealing is to be evaluated. For pseudo plasticity specimens deformed in range III are relevant also for fatigue tests. Soft recrystallized specimens were also cycled to compare them to treated samples. This shows the necessity of thermomechanical treatments and makes the microstructural analysis of incipient damage easier.

2. EXPERIMENTAL

Transmission electron microscopy (TEM) was used to characterize the microstructure before and after fatigue tests. Different microstructures were present before the fatigue tests due to the thermomechanical treatment (Fig. 1). The recrysrallized state shows a low dislocation density (Fig. 1 a). In case of ausforming (AF 350 °C / φ = 0,27), a high number of dislocations were produced (Fig. 1 b). The dislocations are oriented and form different types of deformation bands. By Marforming (MF), a homogeneously distributed defect structure was found. This microstructure recovers and form small grains after annealing (550 °C / 6 min). A similar microstructure was found if the sheets were rolled at room temperature (mixed deformation XF) and heat treated with the same parameters.

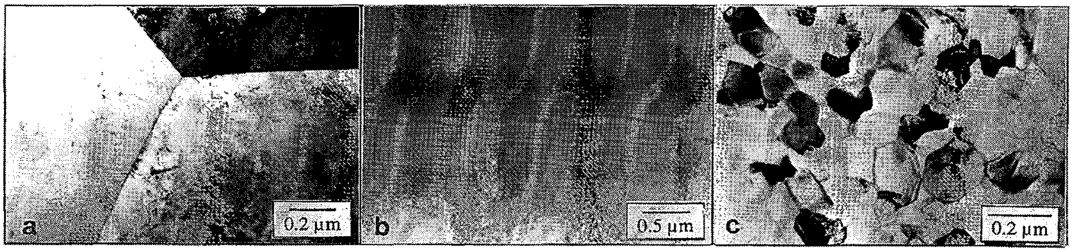

Fig. 1: Microstructures of the conditions before the fatigue tests: a) Fully recrystallized, b) AF 350 °C / φ = 0.27, c) MF φ = 0.44 + 550 °C / 6 min.

Thermal fatigue
The first 5 cycles were measured for thermal fatigue tests using differential scanning calorimetry (DSC). Higher numbers of cycles were reached by alternately dipping the sample into liquid nitrogen and boiling water. After a certain number of cycles, small pieces were cut off in order to measure the transformation temperatures and to analyze the microstructure.

Thermomechanical fatigue
The material was loaded with a constant force at room temperature (RT). The temperature was increased by applying direct electric current and cooled down to RT using an air fan. The strain was measured during all cycles. Data were collected using a computer based data acquisition system.

Mechanical fatigue (true plastic strain increment of φ = 0.06)
The transformation induced strain is the most important property for shape memory alloys. The mechanical fatigue tests were carried out only in tension. Under compression, different martensite variants are activated [4]. This had to be avoided, because different martensite variants in compression and tension make the microstructural analysis of damage more difficult. Fatigue tests with strain control show plastic strain after unloading down to 10 MPa. In the following cycle, the strain had to be calculated using the actual length.

3. RESULTS AND DISCUSSION

3.1 Thermal fatigue

All strengthened alloys show an almost independent behavior of transformation temperatures after thermal cycling (Fig. 2 b). In the first cycle, the R-phase transformation is observed. The reason is internal stress fields induced by dislocations or precipitates due to the hardening treatment [1].

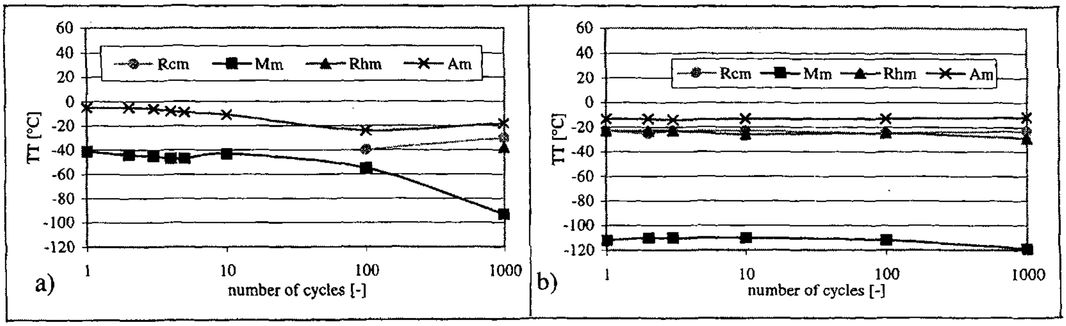

Fig. 2: Change of transformation temperatures (TT) during thermal cycles. a) In recrystallized specimens, the TT are not stable, but in b) MF φ = 0.44 + 550 °C / 6 min specimens, shown as an example for strengthened alloys, such as work hardened (AF) or precipitation hardened conditions.

The austenite peak- (A_m) and martensite peak temperature (M_m) of the recrystallized state decrease with increasing number of cycles (Fig. 2). The R-phase transformation is produced after 100 cycles during cooling and after 1000 cycles during heating. In this soft recrystallized alloy dislocations are easily produced during the phase transformation, which induce internal stress fields and promote R-phase transformation (Fig. 3).

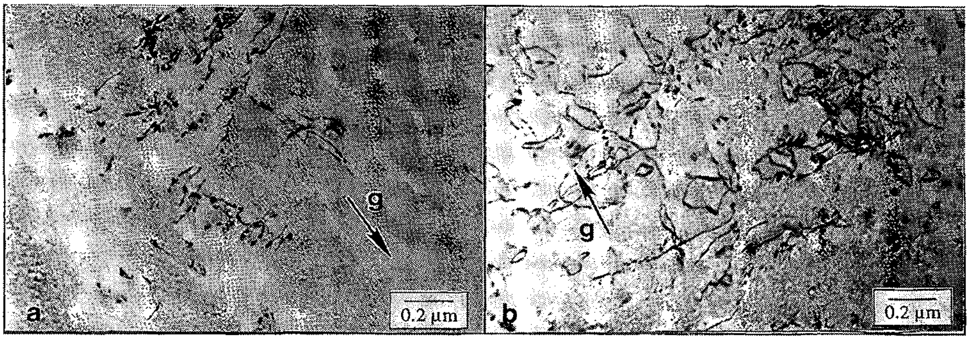

Fig. 3: The dislocation density increases during thermal cycling of the soft recrystallized state. a) n=10, b) n=100 cycles (g = 110).

The R-phase peak temperature (R_{cm}) during cooling increases with increasing number of cycles. More internal stress fields are produced by increasing number of dislocations, which promote the R-phase transformation. During heating, the R-phase transformation (R_{hm}) appears after 1000 cycles, and consequently A_m increases.
In contrast to Cu based shape memory alloys [5], no thermally induced cracks are found in any of the cycled specimens.

3.2 Thermomechanical fatigue

The AF-specimen is hardened by dislocations (Fig.1 b) and the MF + annealed primarily by small grains (Hall-Petch relation). Fig. 4 shows, that only MF + annealing provides a high stability of the extrinsic two way effect. The work hardened AF-specimen show high accumulating irreversible strain and, therefore, no desired fatigue resistance. Probably, the dislocations introduced by the AF-process glide during thermal cycling with constant load and are responsible for such plastic strains.

The dislocations impede the movement of interfaces, which results in a broad temperature range of the transformation.

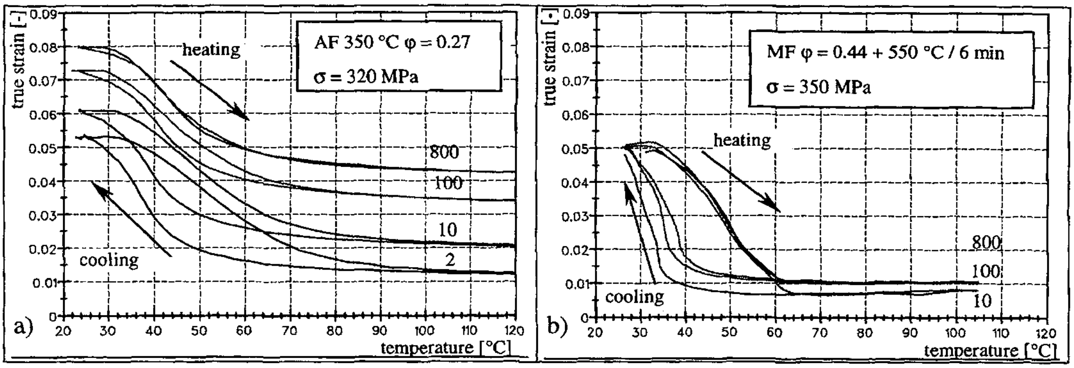

Fig. 4: Extrinsic two way effect of AF (a) and MF + annealed specimen (b). The dislocations in the AF state provide no stability against thermal cycling under constant load.

3.3 Mechanical fatigue

The recrystallized alloy endures only nine cycles, therefore strengthening during cyclic loading leads to no improvement [6]. Consequently, thermomechanical treatments are necessary in NiTi alloys to achieve the desired pseudo elastic behavior.

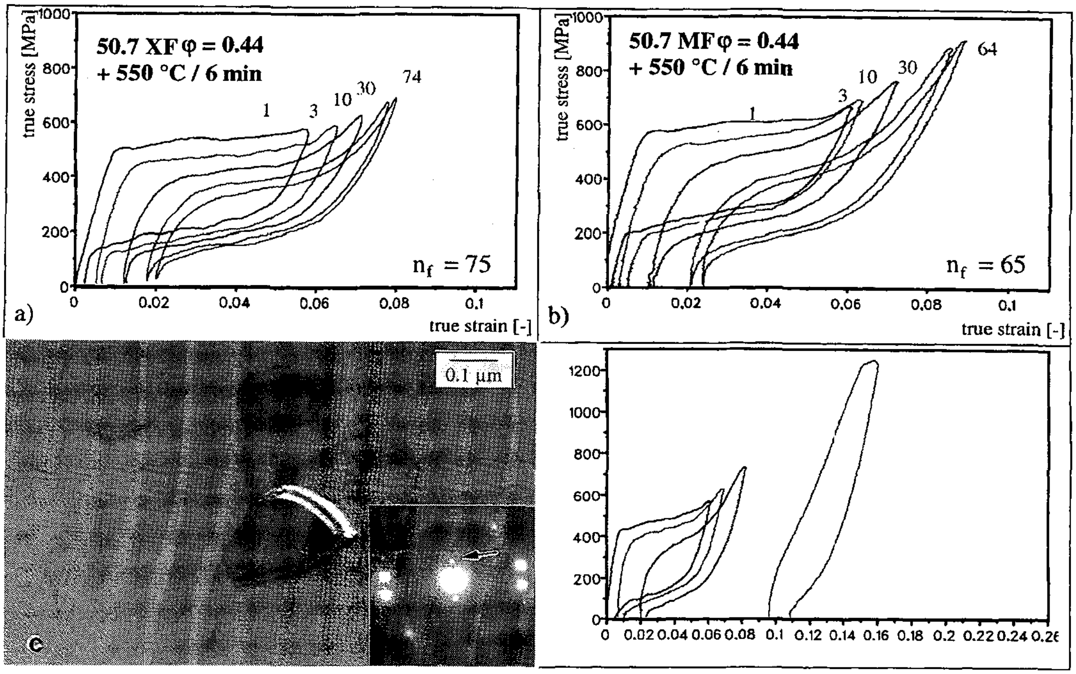

Fig. 5: High rolling reductions (φ = 0.44) lead to nearly the same fatigue life in specimen produced by marforming (a) or mixed deformation (b) followed by annealing. Longer annealing times lead to bigger precipitates on grain boundaries (c), which decreases the fatigue resistance (d).

Previous studies have shown that for one PE-loop, marformed or mixed deformed and annealed specimen provide a high reversibility [1, 2]. In case of high rolling reductions (φ = 0.44), both thermomechanical treatments lead to similar microstructures and, therefore, to comparable fatigue behavior (Fig. 5 a, b). After longer annealing times, precipitates (Ni_4Ti_3 and Ni_3Ti_2) are found on grain boundaries [Fig. 5 c]. They may act as a crack nucleus during cycling and reduce the fatigue life (Fig. 5 d).

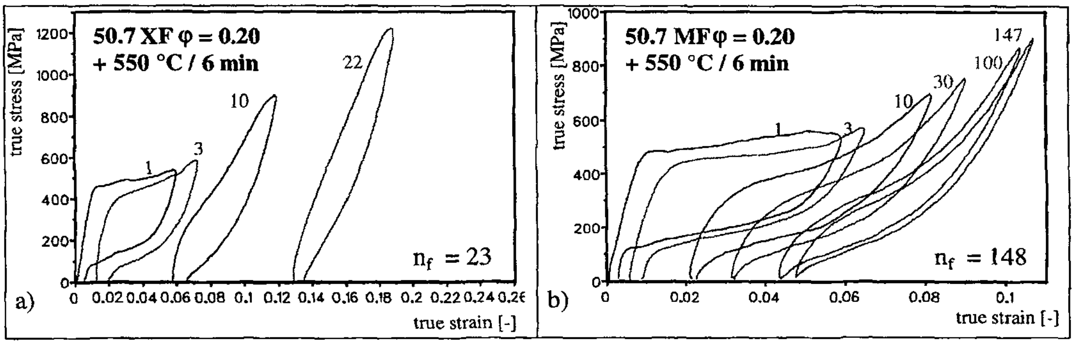

Fig. 6: Lower rolling reductions show the strong influence of the rolling temperature on the fatigue properties.

In case of lowering the amount of rolling reduction (φ = 0.2) by marforming, the microstructure show also small grains after annealing, but less inclusions are cracked during the rolling process. This provides a higher fatigue life (Fig. 6 b), because cracks must nucleate during fatigue.
The same rolling reduction by mixed deformation leads to an irregular microstructure after annealing. Larger areas with a high dislocation density (comparable to ausformed specimen) and areas with small grains similar to the MF + annealed specimen are found (Fig. 7). Compared to thermomechanical fatigue of the AF-specimen, the existing dislocations glide and produce high plastic strains during mechanical cycling (Fig. 6 a). In contrast to the same low rolling reduction as by marforming and annealing, the fatigue life (n_f) is strongly decreased.

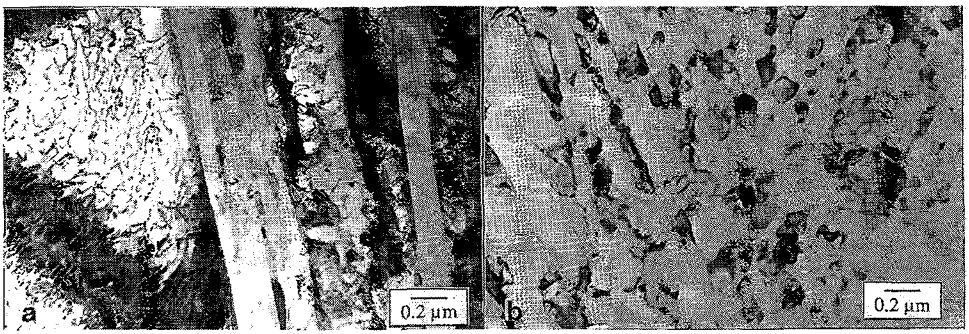

Fig. 7: An irregular microstructure is found in case of mixed deformation plus annealing with lower rolling reductions. Some areas look similar to annealed AF-specimen (a) and some similar to annealed MF specimen (b).

The transformation stress decreases with increasing number of cycles in all treated specimen. Probably defects produced during the repeated stress induced transformation, support the nucleation of martensite. Defects are oriented dislocations in austenite, dislocations of retransformed martensite and residual martensite (Fig. 8). On the other hand such defects are obstacles for the movement of the transforming interface. This results in a higher slope of the transformation plateau with increasing number of cycles and, therefore, higher defect densities.

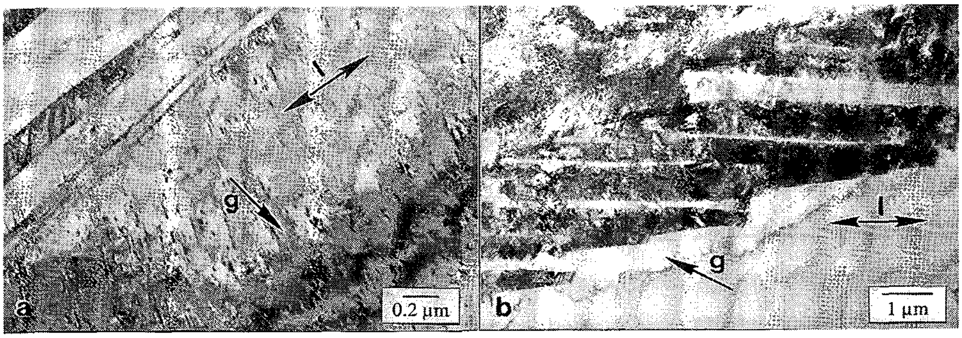

Fig. 8: Oriented dislocations, residual martensite (a) and dislocations of retransformed martensite (b) are produced during mechanical fatigue (g = 110, l = direction of load).

4. SUMMARY

In case of thermal fatigue any strengthening mechanism is successful to create a stability of transformation temperatures. Only in soft recrystallized conditions dislocations are produced. They are responsible for lowering the transformation temperatures and creating the R-phase transformation.

The thermomechanical fatigue tests reveal that work hardening by ausforming only slightly improve the extrinsic two way effect compared to recrystallized alloys. However, by treatments such as XF or MF plus annealing the yield strength is increased primarily by small grains, which is still more suitable during thermal fatigue at a constant load.

Mechanical fatigue experiments provide evidence that MF and annealing is the best thermomechanical treatment for good fatigue resistance. The results indicate the strong influence of rolling reduction and annealing condition.

Acknowledgements

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5. REFERENCES

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