



On the Importance of Structural and Functional Fatigue in Shape Memory Technology

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Abstract The present work provides a brief overview on structural and functional fatigue in shape memory alloys (SMAs). Both degenerative processes are of utmost technological importance because they limit service lives of shape memory components. While our fundamental understanding of these two phenomena has improved during the last two decades, there are still fields which require scientific attention. NiTi SMAs are prone to the formation of small cracks, which nucleate and grow in the early stages of structural fatigue. It is important to find out how these micro-cracks evolve into engineering macro-cracks, which can be accounted for by conventional crack growth laws. The present work provides examples for the complexity of short crack growth in pseudoelastic SMAs. The importance of functional fatigue has also been highlighted. Functional fatigue is related to the degeneration of specific functional characteristics, such as actuator stroke, recoverable strain, plateau stresses, hysteresis width, or transformation temperatures. It is caused by the accumulation of transformation-induced defects in the microstructure. The functional stability of SMAs can be improved by (1) making phase transformations processes smoother and (2) by improving the material's resistance to

irreversible processes like dislocation plasticity. Areas in need of further research are discussed.

Keywords Shape memory alloys · Fatigue · Microstructure · Short crack growth · High entropy alloys

Introduction

Shape memory technology has evolved into mature materials engineering field [1]. Two types of shape memory effects (SMEs), a thermal memory (one/two-way effect, 1/2 WE) and mechanical memory (pseudoelasticity, PE), are exploited for advanced applications in aerospace, automotive, construction and environmental engineering, and in the field of medical technology, e.g., [1–6]. Both types of SMEs rely on the martensitic transformation, a solid-state transformation where a high-temperature phase austenite transforms into a low-temperature phase martensite on cooling/mechanical loading [7, 8]. The reverse transformation occurs upon heating/unloading. Many fundamental aspects of the martensitic transformation are well understood, e.g., [7, 9–12]. For the field of shape memory technology, it is important that the formation of martensite is strongly governed by the chemical composition and the microstructure of an alloy [13–18]. This allows to control and to optimize properties and performance of shape memory alloys (SMAs) for specific applications. During the last decades, the evolution of shape memory technology has significantly benefitted from fundamental and application-related research [19, 20]. The main trends in SMA research were driven by the need for new alloys with high transformation temperatures [21–24] and good fatigue resistance [25–30]. Today, a better understanding of mechanical, functional, and microstructural

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aspects [16–18, 31–36], the possibility of 3D printing [37–40], the medical application of SMAs [41–43], and their use in niche applications such as solid-state refrigeration [44–48], advance the field. However, there are specific issues which still hamper the breakthrough of SMA technology. The two probably most important generic issues are structural and functional fatigue. They both limit the performance and the exploitable service lives of shape memory components.

In 2003, Eggeler et al. [49] highlighted the overriding importance of both, structural and functional fatigue of SMAs, and this is today well appreciated [30, 50–52]. Structural fatigue refers to the nucleation and growth of cracks during cyclic loading, which leads to fatigue failure. In contrast, functional fatigue is related to the degeneration of specific functional characteristics, such as actuator stroke, recoverable strain, plateau stresses, hysteresis width, or transformation temperatures. Functional fatigue is caused by irreversible microstructural changes. In the present work, a short overview on the importance of these two types of degenerative processes is provided and fields in need of further work are identified.

Structural Fatigue

Research on structural fatigue in SMAs has often been motivated by the requirement for medical implants to withstand a high number of loading/unloading cycles [27, 53, 54]. For example, stents which are implanted in blood vessels in the human body are exposed to pulsatile fatigue. A stent is expected to maintain its integrity for at least 10 years, which, at an average pulse of 70 min^{-1} , corresponds to 3.7×10^8 load cycles. Stents, therefore, have to withstand high-cycle fatigue conditions, with cycle numbers in excess of 10^8 . Today, a good understanding of factors and processes affecting fatigue lives has been established, e.g., [25–30, 49, 55, 56]. Depending on loading conditions, one can differentiate between high-cycle and low-cycle fatigue regimes (HCF and LCF), e.g., [25–30]. In the HCF regime, fatigue behavior is governed by microstructural processes which lead to the nucleation and growth of cracks. In contrast, cracks quickly form in the LCF regime, and therefore fatigue lives are controlled by crack propagation. For the nucleation of cracks, the surface quality is of utmost importance. Small surface defects like scratches, pores, notches, wire drawing die marks, etc., act as local stress raisers and thus promote the formation of fatigue cracks. Attempts are made to improve surface quality, for example, by electropolishing [57, 58], or to introduce compressive stresses in surface regions [53, 59]. In the absence of surface defects, the formation of fatigue cracks mainly occurs at small oxide and carbide inclusions

[25] which are related to small amounts of impurities, as documented in [14, 60–62]. The propagation of cracks in SMAs differs from what is known for conventional structural engineering materials. Due to high local stresses in front of crack tips, a stress-induced transformation of martensite occurs [29, 63, 64], such that cracks in SMAs grow into martensitic regions.

In general, structural fatigue follows three different stages: (1) crack nucleation, (2) short crack growth, and (3) macroscopic growth of engineering cracks [65, 66]. While crack formation, macroscopic crack growth, and fatigue lives were addressed in numerous studies, e.g., [25, 27, 67], the behavior of short cracks in SMAs has not received sufficient attention so far. This stage is significantly important. It has been documented in the literature [25] that NiTi SMAs are prone to the formation of micro-cracks. Rahim et al. [25] have demonstrated that close to 300 micro-cracks per square millimeter surface area form during cyclic loading at a strain amplitude of only 1.9% within ≈ 2000 fatigue cycles [25]. It is, therefore, important to understand how short cracks in NiTi SMAs evolve into larger cracks which grow at material-specific rates [68].

In the following, the results of a study [69] are reviewed, where the growth of short cracks in pseudoelastic NiTi wires was investigated using interrupted bending rotation fatigue (BRF) experiments. Details on alloy preparation/processing and fatigue experiments are documented elsewhere [14, 25, 57, 69, 70]. The NiTi wire was characterized by a slightly increased oxygen concentration (500 ppm), which allowed to investigate the effects of oxygen-rich inclusions on fatigue. Detailed information on the formation of these inclusions, their crystallographic nature, effects on transformation behavior, etc., are available in the literature, e.g., [14, 60–62]. The wire was subjected to interrupted BRF testing at human body temperature, which results in specific stress/strain states [71, 72]. The nominal imposed surface strain was 1.9%. After specific numbers of mechanical cycles, the surface of the wire was iteratively investigated by scanning electron microscopy (SEM) to detect and to evaluate the formation and growth of micro-cracks. A special SEM holder, Fig. 1, was used to monitor a specific surface region of the NiTi wire after increasing load cycles. This allows to follow the growth of one crack during different stages of BRF testing. The SEM holder is presented in Fig. 1a, and the region of interest on the wire sample is shown in Fig. 1b.

Figure 2 shows parts of the wire surface after 0 (Fig. 2a, initial state), 400 (Fig. 2b), and after 2100 BRF cycles (Fig. 2c). The dark particles represent oxides [14, 25]. The black dot on the left side of the images serves as a reference point. The loading direction is from the left to the right. Cracks which have formed during BRF testing are indicated by arrows, it is difficult to identify them in the

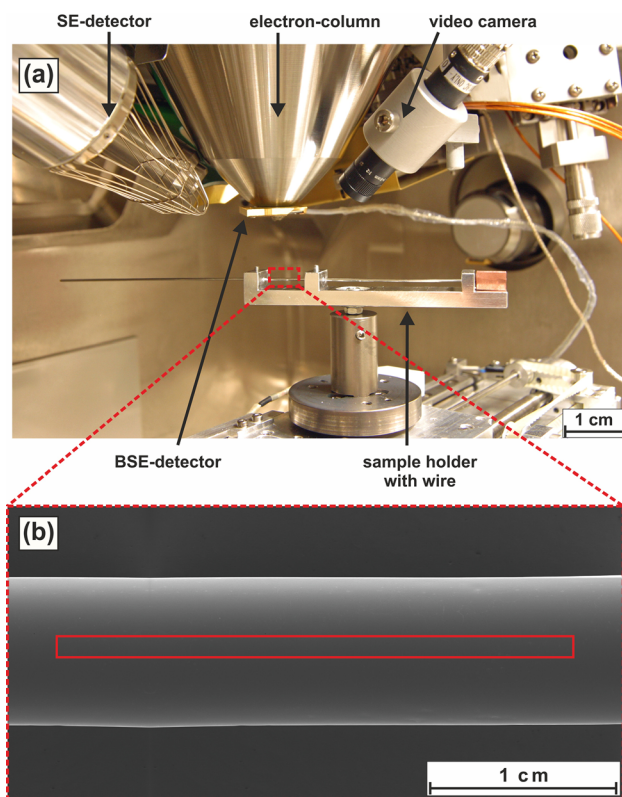


Fig. 1 SEM surface characterization of an electropolished NiTi wire during interrupted BRF experiments. **a** SEM chamber with sample holder which enables to monitor the same region during fatigue testing. **b** SEM image of region of interest (red rectangle)

overview SEM micrographs of Fig. 2. After 400 cycles, 4 micro-cracks were observed (Fig. 2b). The number of cracks increased to 9 within 2100 cycles (Fig. 2c).

Micro-crack nucleation and growth are presented in Fig. 3 at a higher magnification. Figure 3 shows two oxide particles which are associated with small voids. These types of defects were referred to as particle/void assemblies (PVAs) in the literature [25]. Voids form when larger inclusions fracture during thermo-mechanical processing [73], e.g., during wire drawing. These PVAs represent dominant crack initiation sites for electropolished NiTi SMAs, where no more severe surface defects are present [25]. Figure 3 shows that cracks form from interface regions between oxide particles, adjacent voids, and the SMA matrix. Figure 3a–c document how the two cracks shown in Fig. 3 grow during the different stages of the fatigue experiment, i.e., during 2100 cycles. As a striking result, it was found, that only one crack, which emanated from the right particle, underwent significant growth. In contrast, the left crack in Fig. 3 showed no increase in length. This is probably due to a stress-shielding effect, caused by the propagation of the larger micro-crack. The observed behavior is not uncommon. In general, the growth

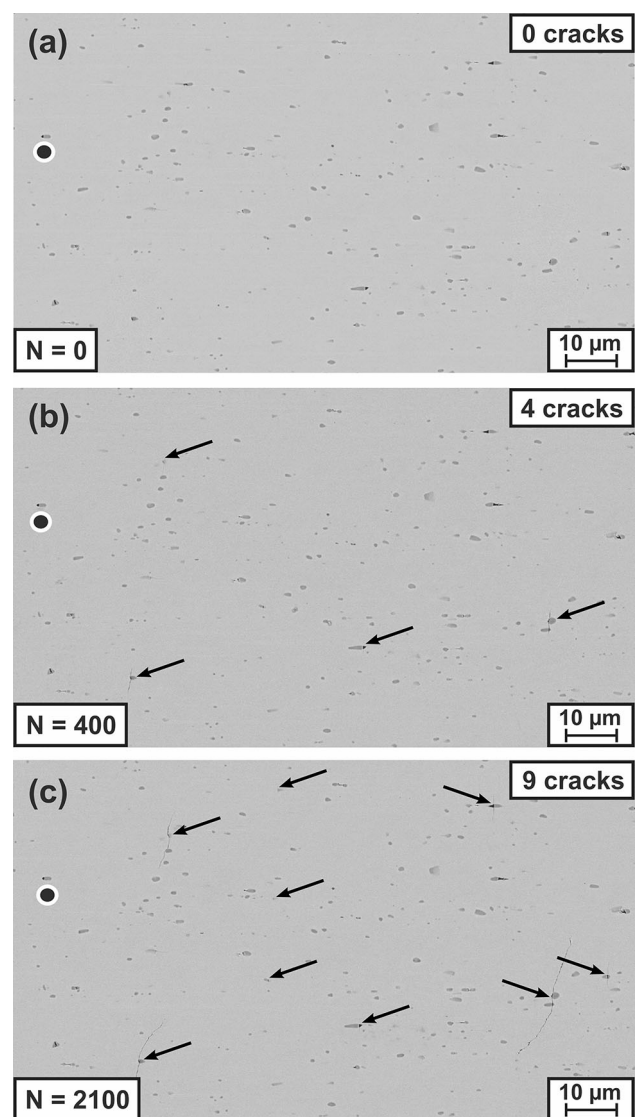


Fig. 2 Formation and growth of short cracks emanating from oxide inclusions during BRF testing. **a** Initial state. **b** After 400 cycles. **c** After 2100 cycles. The black dot indicates the same reference point in all three images

of micro-cracks cannot be rationalized by macroscopic growth models [74–76]. Their behavior is mainly governed by local stresses in different microstructural regions. Furthermore, shielding effects, as exemplarily shown in Fig. 3, play important roles. Not all micro-cracks are able to evolve into macroscopic cracks [74–76].

Figure 4 presents information on how the lengths of different short surface cracks in the NiTi wire evolve during BRF testing. The data sets in Fig. 4 rely on the evaluation of surface crack lengths by quantitative image analysis of 69 cracks during interrupted BRF experiments. The data in Fig. 4 suggest that only a small number of micro-cracks exhibits a significant growth. Fifty cracks did not reach lengths exceeding 25 μm . Only one single crack

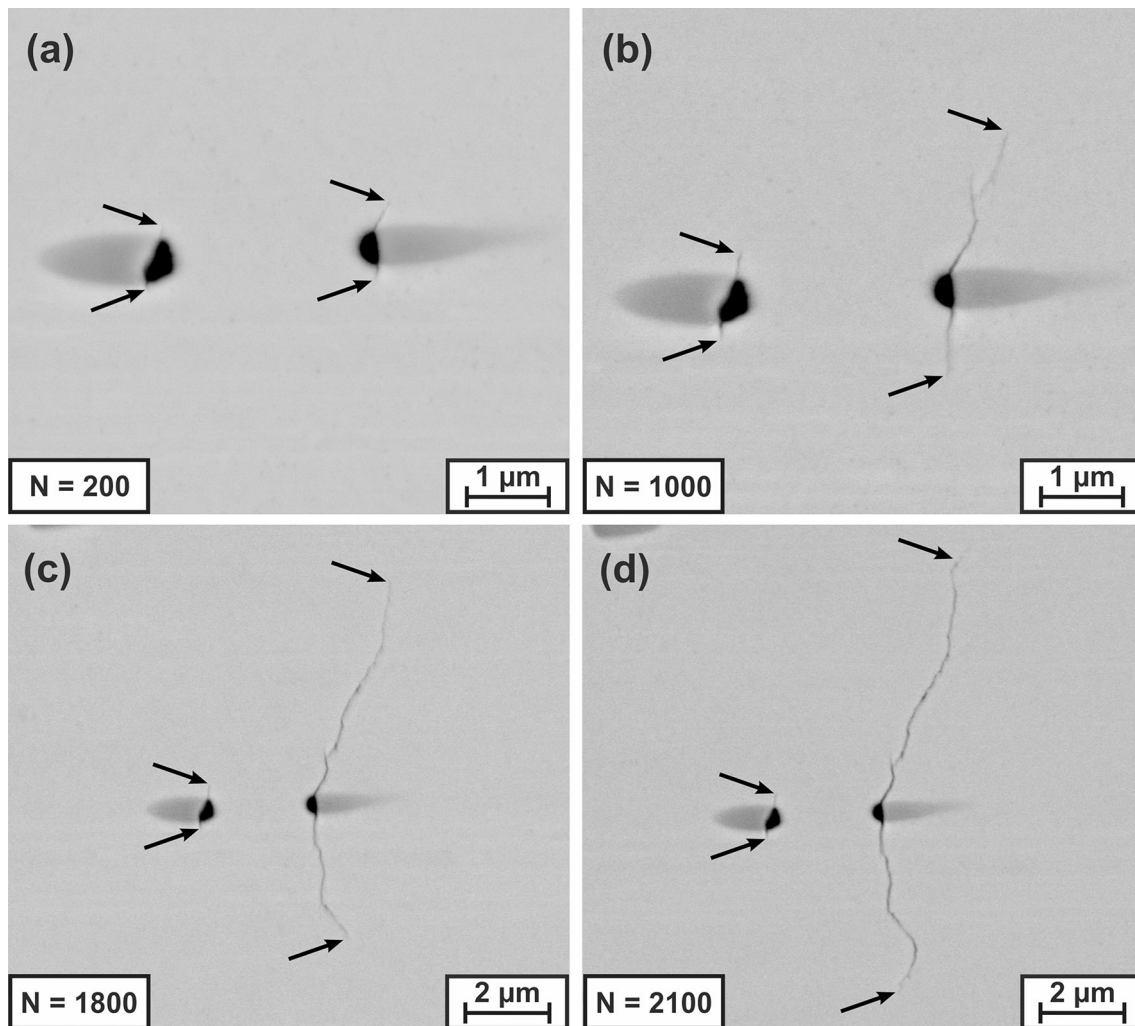


Fig. 3 Examples from short crack growth in pseudoelastic NiTi. Two cracks grow from particle void assemblies (PVAs). **a** 200 cycles. **b** 1000 cycles. **c** 1800 cycles. **d** 2100 cycles

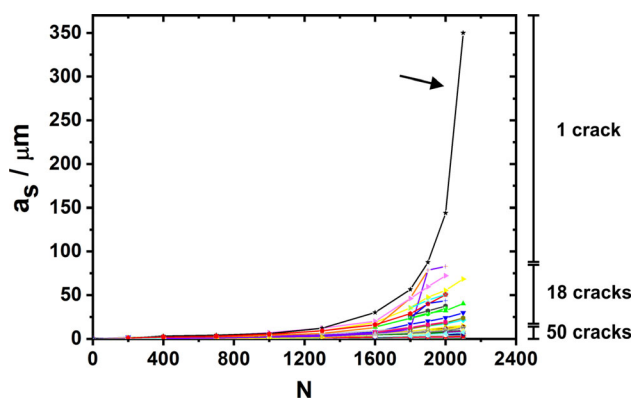


Fig. 4 Evolution of micro-crack lengths during structural fatigue testing of a pseudoelastic NiTi wire. For details see text

managed to grow by more than 100 μm (Fig. 4). In fact, the long crack highlighted in Fig. 4 by an arrow finally caused the fatigue failure of the wire specimen. The

behavior presented in Figs. 2, 3, and 4 merits further analysis. It is interesting to clarify which processes govern the growth of short cracks with sizes which match typical length scales involved in the formation of martensitic microstructures.

Functional Fatigue

In most SMAs, functional fatigue is directly caused by the accumulation of transformation-induced defects in the microstructure. Only a few exceptions exist, for example in β -Ti alloys, where ω -phase and α -Ti, which form during aging at elevated temperatures, affect martensitic transformations, e.g., [77–80]. In general, transformation-induced degradations can occur during (1) thermal cycling [81, 82], (2) thermal cycling under stresses [83–85], and (3) during mechanical (e.g., pull–pull) cycling of pseudoelastic

SMA s [86, 87]. In the early 1970s, Perkins [88] provided microstructural evidence for irreversible transformation-related microstructural changes, which result in functional degeneration. Using transmission electron microscopy, it could be demonstrated that dislocations accumulate during thermal cycling in NiTi SMA s. It was suggested that these dislocations may provide back-stresses which support shape recovery processes. While this can be true for situations where a two-way effect is exploited after a thermo-mechanical training, a different view has been established today. In general, dislocations form during martensitic transformations to compensate for the crystallographic misfit between austenite and martensite [81, 82, 84, 85, 89]. Dislocations directly interact with austenite/martensite transformation fronts [90] and affect phase transformation behavior [81, 91, 92]. They even can stabilize martensite [93, 94]. Recently, a symmetry-dictated non-phase-transformation pathway during phase transformation cycling was proposed, which also could play a role as a potential mechanism leading to functional fatigue [30].

Figure 5 presents a few results on functional fatigue of binary Ni₅₀Ti₅₀ and ternary Ni₄₀Ti₅₀Cu₁₀ SMA s. Two types of experiments were performed. First, both alloys were subjected to stress-free thermal cycling using differential scanning calorimetry (DSC). Second, spring actuators were prepared, and the functional performance of these springs was evaluated using a special test rig. Details on thermal cycling, alloy preparation/processing, and spring actuator fatigue testing are documented in the literature [70, 85, 95, 96]. Figure 5a, b present results from DSC experiments where 20 heating/cooling cycles were imposed. In the case of binary NiTi, the peaks associated with the forward and reverse transformation shift towards lower temperatures during cycling (Fig. 5a). The NiTiCu SMA, Fig. 5b, shows two-step transformations, which are related to the formation of B19 and B19' martensites on cooling and to the reverse transformations on heating [97]. The data presented in Fig. 5a, b show that the NiTiCu SMA exhibits a significantly better functional stability as compared to binary NiTi, where transformation peaks shift to lower temperatures during cycling. Similar trends were

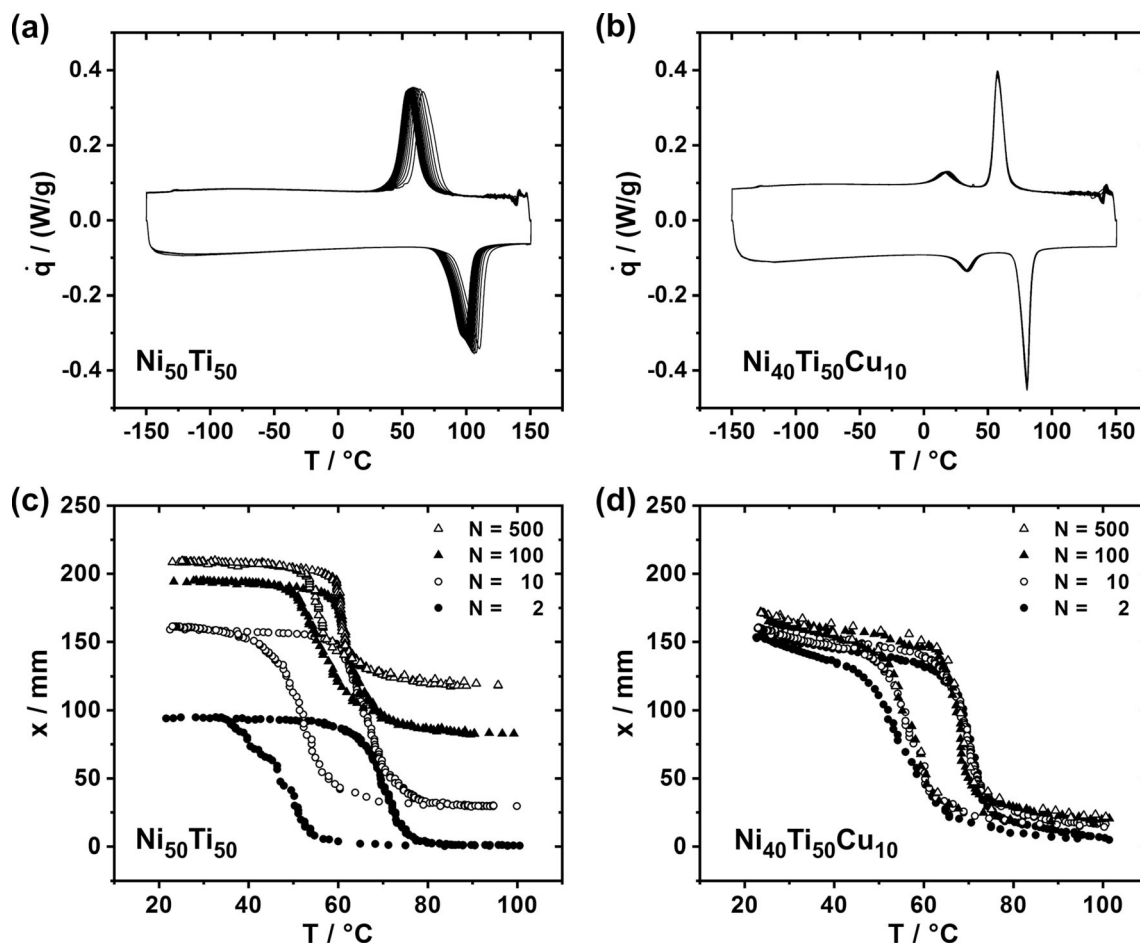


Fig. 5 Functional fatigue of NiTi and NiTiCu SMA s. **a, b** Stress-free thermal cycling. **c, d** Thermo-mechanical cycling of spring actuators (reprints from [85])

found for the performance of NiTi and NiTiCu spring actuators (Fig. 5c, d). Displacement/temperature hystereses of NiTi and NiTiCu spring actuators are presented in Fig. 5c, d [85], respectively. During heating, the actuators contract, which translates into a decrease of the displacement (x). A total number of 500 thermo-mechanical actuation cycles (N) was performed for both actuators. The data in Fig. 5c, d show that functional fatigue during cycling results in a shift of the hysteresis curves to higher x values. This means that the spring actuators permanently elongate through functional fatigue. Furthermore, a decrease of the hysteresis widths occurs during cycling. A detailed analysis of this behavior is presented in [85]. Most importantly, the functional stability of the NiTiCu actuator, Fig. 5d, is significantly higher as that of binary NiTi (Fig. 5c). This is in line with what was previously observed for stress-free thermal cycling in the DSC (Fig. 5a, b).

The reason for the better functional stability of NiTiCu alloys is that these SMAs exhibit a better compatibility between the crystal lattices of austenite and martensite [98–100]. Therefore, fewer defects accumulate in the microstructure during phase transformation events. Grossmann et al. [85] were the first to document a direct evidence for the correlation between crystallographic compatibility and functional fatigue. This effect was later on confirmed in other studies, e.g., [84, 101–103]. One has to mention that crystallographic compatibility not only plays a role for thermal cycling with/without stresses (Fig. 5). Jaeger et al. [102] demonstrated that different degrees of crystallographic compatibility, which can be adjusted in binary NiTi by variations of the Ni concentration [14], lead to different mechanical stability during pseudoelastic cycling. Early studies on the relation between crystallographic compatibility and functional fatigue invoked the λ_2 -misfit-parameter, which was introduced in [98, 99]. Since a few years, the effects of additional parameters, referred to as cofactor conditions, receive increasing scientific attention, e.g., [24, 104–110]. It has been discussed by Song et al. [104] that these cofactor conditions are highly relevant for functional fatigue. Shape memory materials have been identified which closely satisfy these supercompatibility conditions and which show a good functional stability, e.g., $\text{Zn}_{45}\text{Au}_{30}\text{Cu}_{25}$ and $(\text{Ti}_{54}\text{Ni}_{34}\text{Cu}_{12})_{90}\text{Nb}_{10}$ [104, 106–110]. The compatibility between the lattices of austenite and martensite can be improved by compositional variations.

Tuning the crystallographic compatibility is not the only option available to reduce functional fatigue. The present state of knowledge shows that two different strategies can be applied: first, one can make phase transformation processes smoother to reduce irreversibility effects. Second, one can make the SMA stronger to increase fatigue resistance. The first strategy addresses crystallographic effects

as has been discussed previously. The second strategy involves different measures which increase the resistance of the SMA with respect to plastic deformation. Grain size refinement has been shown to significantly reduce dislocation plasticity and thus to promote a stable functional performance, e.g., [95, 111, 112]. A second option is particle strengthening. It has been demonstrated for both binary and multi-component NiTi-based SMAs that precipitation hardening improves functional stability [103, 113–115]. A third option exists which may reduce functional fatigue: A few years ago, Firstov et al. [116, 117] introduced high-entropy SMAs. These materials receive increased scientific attention at present, e.g., [118–121]. Similar to conventional high-entropy alloys (HEAs), e.g., [122, 123], these materials are characterized by a large number of alloy components with equimolar compositions. The chemical nature of these SMAs may provide solid-solution-like strengthening effects. Further work is required to clarify how chemical complexity affects reversible and irreversible elemental processes of martensitic transformations.

Coupling Between Structural and Functional Fatigue

A strict differentiation between functional and structural fatigue is not always possible. Bigeon and Morin [124] studied the functional performance of NiTiCu and CuZnAl SMAs during thermal cycling under *constant* load. As a striking result, they observed that their samples broke after a certain number of cycles. The number of cycles to failure was depending on the loading stress, and increasing stress levels resulted in shorter fatigue lives. Bigeon and Morin [124] also showed that NiTiCu SMAs were less prone to this coupled functional/structural fatigue (CFSF) than CuZnAl alloys. Figure 6 presents SEM micrographs of NiTi wires after CFSF testing. The sample was loaded at a constant stress of 450 MPa and subjected to heating/cooling cycles until fracture occurred after 10,328 cycles. Details on experiments are given elsewhere [125]. The fracture surface, Fig. 6a, shows that a large number of small cracks (highlighted by arrows) have formed in surface-near regions from where they grew inwards into the wire prior to rupture. Figure 6b shows the skin surface of the wire. A high density of short cracks is visible, which grow perpendicular to the loading direction. The mechanism for the formation and growth of these cracks are not well understood at present. It has been suggested that cycling results in an increase of the surface roughness [126] which facilitates crack formation. Experimental studies are scarce at present, [115, 127–131]. Further work is required to analyze if and how incompatibilities between

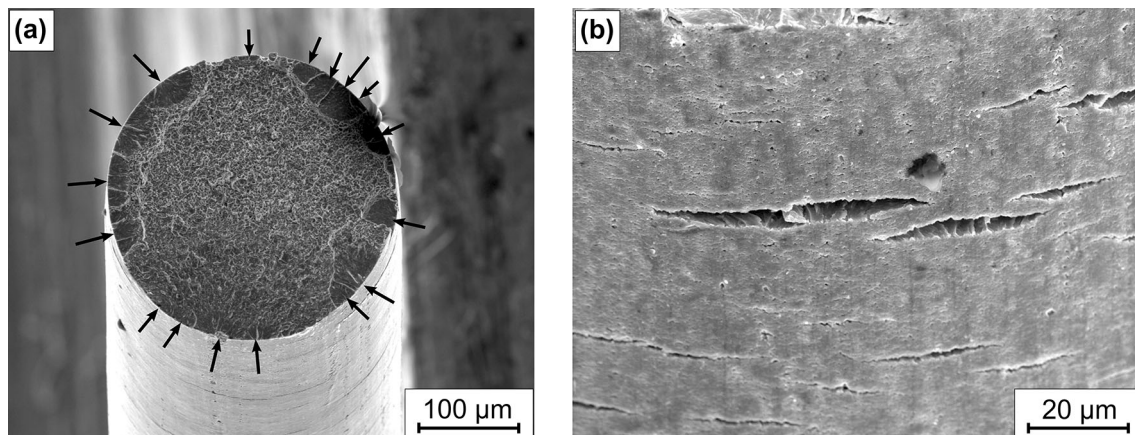


Fig. 6 Coupled functional and structural fatigue of NiTi SMA wires. **a** Fracture surface with crack initiation sites indicated by arrows. **b** Skin surface of the fractured wire showing high density of small micro-cracks

the lattices of martensite and austenite and related irreversible processes contribute to a coupling between functional and structural fatigue.

Summary

The present work stresses the importance of structural and functional fatigue in shape memory technology. Both limit the exploitable service lives of shape memory components. While a good state of knowledge has been established in both fields during the last decades, there are still aspects which require scientific attention. NiTi SMAs are prone to the formation of small cracks. There is a need for a better understanding of how micro-cracks can grow in a material which undergoes a stress-induced martensitic transformation, which redistributes stresses in the microstructure. The importance of functional fatigue has been highlighted. The functional stability of SMAs can be improved by (1) making phase transformations smoother and (2) by improving material strength to increase fatigue resistance. Further work is required to understand interactions between functional and structural fatigue and to clarify potential effects of solid-solution strengthening.

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