



Phase transformation volume amplitude as a low-cycle fatigue indicator in nickel–titanium shape memory alloys

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ABSTRACT

Transformation-induced plasticity due to the motion of martensite fronts is a key mechanism for damage accumulation during cyclic loading of NiTi shape memory alloys (SMAs). Intuitively, a larger motion of martensite fronts should result in a larger accumulation of defects, and lead to a shorter low-cycle fatigue life. We validate this hypothesis through fatigue tests and surface strain-field measurements. This result is agnostic of the tensile or bending loading mode. We also demonstrate that the fatigue lifetime intrinsically depends on a combination of two factors: The transformation volume amplitude and the number of inclusions interacting with the phase transformation domains.

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Superelastic Nickel–Titanium shape memory alloys (NiTi SMAs) are widely used to manufacture medical implants. The appeal of superelastic NiTi for this application comes from its ability to recover large cyclic deformation. This ability itself arises from a reversible martensitic phase transformation that can be induced by stress. While medical implants are expected to offer a long service life, the reversibility of NiTi deformation has certain limits. Thus, durability and fatigue remain topics of continued research in the field of NiTi SMAs. Inelastic deformation phenomena and metallurgical properties of NiTi are two key factors influencing its durability.

Transformation-induced plasticity – an inelastic deformation phenomenon where the motion of phase transformation fronts results in localized slip – is known to be a mechanism of structural and functional fatigue in NiTi [1]. While the mechanism of transformation-induced plasticity operates at the length scale of the local stress-fields at the interface between austenite and twinned martensite correspondence variants [2], it is reasonable to expect that accumulation of damage due to the same mechanism would occur at the macroscopic transformation fronts (i.e., at Lüders bands). Additionally, Sedmák et al. [3] demonstrated that a stress-concentration zone exists on the austenite side of a transformation front, potentially contributing to the damage accumulation. Results in the literature indicate that incremental damage accumulation due to transformation front motion may be con-

tributing to fatigue in NiTi. Various studies have revealed that low-cycle fatigue failures consistently occur at the transformation front or inside the martensitic region during fatigue testing of NiTi strip specimens [4–7]. Past efforts have also shown a correlation between cycles-to-failure (CTF) and various metrics indicative of the extent of phase transformation such as the hysteresis area, the local strain amplitude, and the size of transformed zone [4–6,8]. Zhang and He empirically demonstrated that branched transformation fronts in thicker fatigue-loaded NiTi strips correspond to lower CTF compared to thinner strips without branching fronts, indicating the deleterious effect of a larger transformation front area [9].

Non-metallic inclusion content is a key metallurgical property of NiTi alloys influencing durability. Commercial NiTi is offered in various purity levels. Standard-purity alloys consist of a certain distribution of carbide and oxide inclusions and high-purity alloys (sometimes referred to as extra-low interstitial or ELI) typically consist of smaller or fewer inclusions per unit volume [10]. The high-purity alloys tend to show superior fatigue performance to the standard-purity variants [11,12]. Micromechanically, Kafka et al. analytically demonstrated that the crack incubation period is inversely proportional to the inclusion diameter [13]. Rahim et al. empirically showed that crack propagation rate does not depend on alloy purity, but crack initiation does [14]. Thus, inclusion content appears to play a central role in determining the damage initiation in NiTi. This is intuitive since inclusions can act as stress raisers and lead to localized phase transformation during fatigue loading [15]. Interestingly, Alarcon et al. have shown that the fatigue life decreases before the maximum nominal stress during superelastic fatigue loading reaches the martensitic transformation onset stress value [16].

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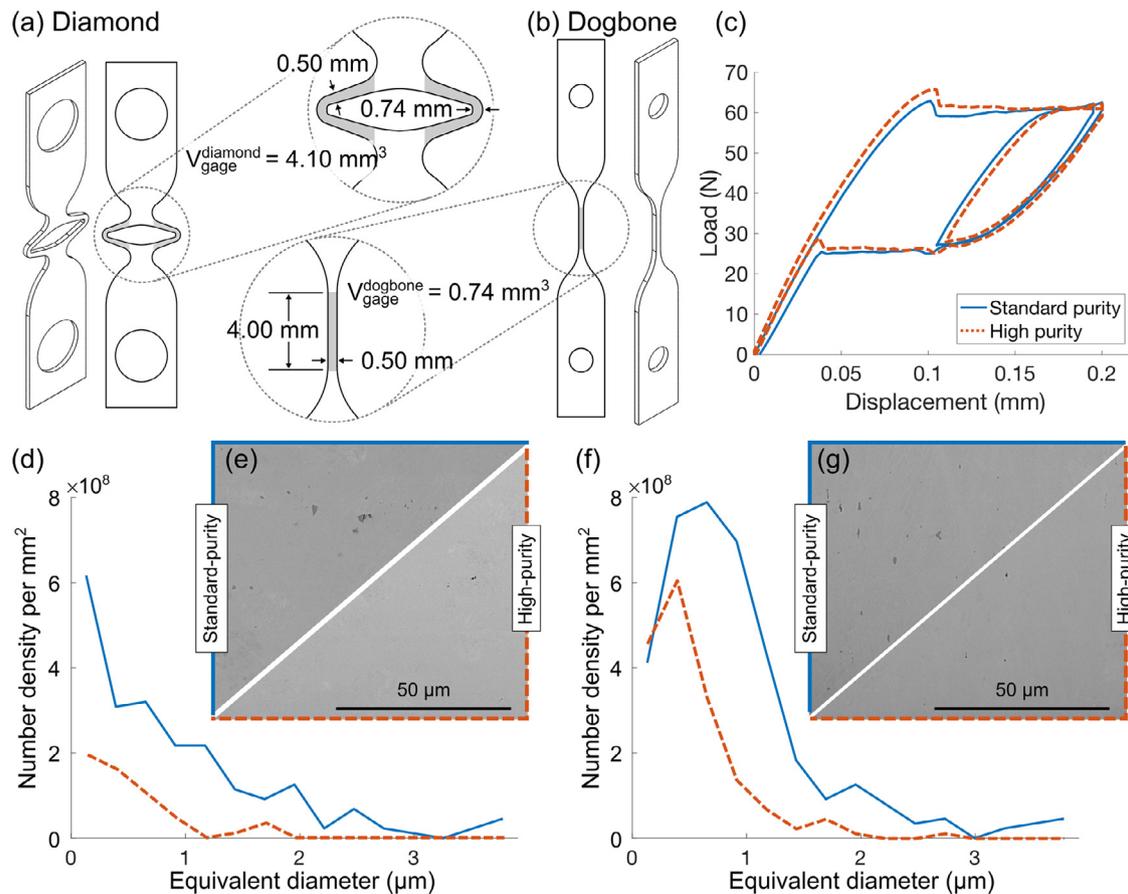


Fig. 1. Specimen description. (a, b) Diamond and dogbone specimen geometries respectively. The gage section is shaded gray and the volume of the gage is annotated. (c) The load-displacement curves at room temperature for a standard-purity dogbone and a high-purity dogbone showing equivalency. (d) Inclusion number density distribution in the transverse cross-section of a standard-purity and a high-purity dogbone. (e) Abridged SEM micrograph showing the inclusion content (dark gray features) in the two materials. Note that the high-purity material has fewer inclusions. (f, g) Corresponding histogram and micrographs for the longitudinal cross-section of the two materials. The legend in (c) applies to (c, d, f). See Supplementary Material for details.

Intuition and the above-mentioned evidence in the literature connecting transformation front motion and inclusion content to the fatigue lifetimes in NiTi led us to pose the following hypotheses. (1) We hypothesize that a larger motion of martensite fronts should result in a larger accumulation of defects, and lead to a shorter low-cycle fatigue life in superelastically loaded NiTi. (2) When the transformation fronts interact with larger or more numerous inclusions, the fatigue lifetimes are worse. We tested these two hypotheses through tensile and fatigue testing of superelastic NiTi.

We manufactured superelastic tensile test specimens with two geometries – dogbone and diamond – as shown in Fig. 1(a, b). When axially stretched, the deformation in the dogbones is primarily uniaxial and in the diamonds, it is bending-dominated. The dogbone specimens were manufactured from NiTi with two different purities: standard-purity and high-purity. These two materials are identical in terms of their thermo-mechanical performance as demonstrated by the equivalent load-displacement curves in Fig. 1(c). However, they differ in terms of the inclusion content. Overall, the high-purity material contains fewer and smaller inclusions as shown by the metallographic cross-sections and inclusion size histograms in Fig. 1(d–g). See Supplementary Material for further details on metallographic inclusion analysis. Thus, three specimen types were prepared: (1) Standard-purity dogbones, (2) high-purity dogbones, and (3) standard-purity diamonds. All specimens were manufactured from superelastic Ti50.8at.%Ni strips using laser-cutting. The specimens were electropolished to remove approximately 30 μ m material from the surface so as to eliminate

any large surface irregularities and the heat affected zone due to the laser cutting process.

We performed tensile tests on the specimens as shown in Fig. 2(a). For the dogbones of each purity, we first applied a pre-extension of 0.2 mm and then applied six subcycle amplitudes of 0.03, 0.06, 0.075, 0.09, 0.105, and 0.12 mm. For the standard-purity diamonds, a pre-extension of 0.5 mm was applied and seven amplitudes of 0.05, 0.1, 0.15, 0.2, 0.25, 0.3, 0.35 mm were applied in the subcycle. We repeated each test on at least two and generally three specimens to obtain a statistics on the deformation. Thus, we performed a total of 54 tensile test cycles. We performed the tests in displacement control at room temperature using a nominal displacement rate of 3.3×10^{-3} mm s $^{-1}$.

We characterized the 2D surface strains in the specimens at the extrema of the tensile subcycle (i.e., points ① and ② in Fig. 2(a)) using digital image correlation (DIC), which is a non-contact and optical technique. An example of the measured strain field at points ① and ② is shown in 2(b, d). We used a Matlab-based computer program to segment the gage area into a low-strain region and a high-strain region. For segmentation, we used a principal strain ($\epsilon_{\text{principal}}$) threshold of 0.015. We defined principal strain as the larger magnitude among $\frac{\epsilon_{xx} + \epsilon_{yy}}{2} \pm \sqrt{(\frac{\epsilon_{xx} - \epsilon_{yy}}{2})^2 + (\epsilon_{xy})^2}$. Here ϵ_{xx} , ϵ_{yy} , and ϵ_{xy} are the axial and shear Green-Lagrangian strains. The high-strain regions furnish the gage area that has undergone austenite to martensite (A \rightarrow M) phase transformation as shown in Fig. 2(c, e). The transformed area is multiplied by the mean specimen width to obtain the transformed volume. The

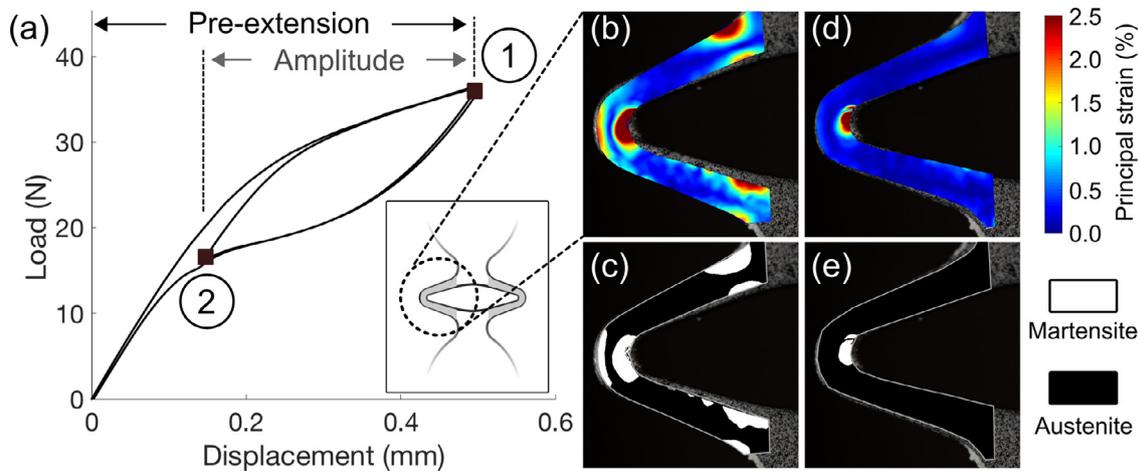


Fig. 2. Segmentation method. (a) The load-displacement curve for a diamond specimen showing the imposed pre-extension and the subcycle amplitude. Points ① and ② denote the extrema of the loading subcycle. (b, d) Gage-section surface principal strain plots at ① and ② respectively obtained using digital image correlation. (c, e) Segmented gage section into an austenitic region and a martensitic region at ① and ② respectively using a strain-based criterion.

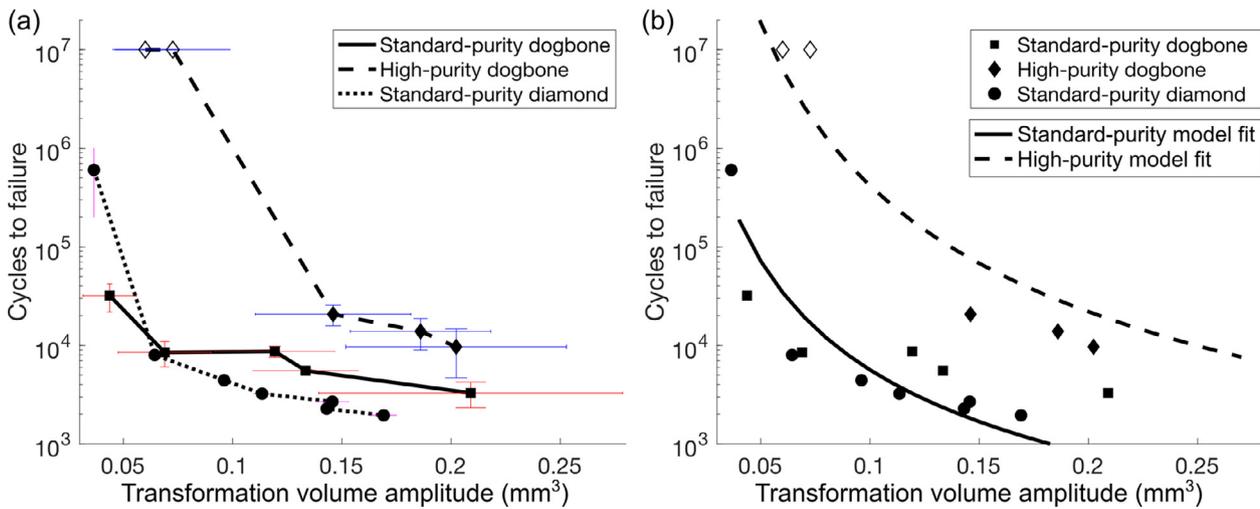


Fig. 3. Results. (a) Experimentally measured cycles-to-failure vs. phase transformation volume amplitude for all specimen types showing an inverse correlation. The filled markers indicate fractures and the empty markers indicate run-out to ten million cycles. The error bars indicate the standard deviation. (b) The same data as in (a) with the fit of a phenomenological model described by Eq. (1).

difference in the transformed volume between the extrema of the subcycle or between points ① and ② furnishes the *phase transformation volume amplitude*. We determined the transformation volume amplitude for all 54 tensile test cycles and then determined the mean and standard deviation of the transformation amplitude for each imposed subcycle displacement amplitude.

We performed fatigue tests on the three specimen types with the identical displacement pre-extension and subcycle amplitude as listed above for the tensile tests. The testing was performed at room temperature in a water bath. At least one and generally three specimens were tested for each amplitude condition. We recorded the CTF for each test and from that determined the mean CTF and the standard deviation CTF for each imposed displacement amplitude. Survival at ten million cycles was considered a run-out. See Supplementary Material for details on the testing methods.

The variation of CTF vs. phase transformation volume amplitude for all specimens is shown in Fig. 3(a). All curves in the figure indicate that the CTF decrease with increasing transformation amplitude. Thus, transformation amplitude is a reasonable low-cycle fatigue indicator. Comparison of the curves for standard-purity dogbone and standard-purity diamond indicates that the results hold for both geometries. That is, the results are agnostic of the axial-

dominant vs. bending-dominant nature of the deformation. Comparison of the curves for standard-purity dogbone and high-purity dogbone indicates that the CTF are larger for high-purity material compared to the standard-purity material. These results validate both hypotheses posed above. Larger motion of martensite fronts, quantified in terms of the phase transformation volume amplitude, leads to a shorter low-cycle fatigue life. The fatigue lifetimes are worse when the transformation fronts interact with larger or more numerous inclusions as in the standard-purity material vs. the high-purity material.

We propose a mechanism, schematically shown in Fig. 4, to rationalize these results. Consider a dogbone specimen loaded in tension. In such a specimen, a localized transformation band would form and propagate along the gage length during loading. A localized stress concentration would be present in austenite in the vicinity of such a transformation front as shown in Fig. 4(a) [3]. The inclusions in the NiTi specimen would host their own local stress field. As the transformation front moves, the stress field of the front would superimpose with the stress field of the inclusions that are in the transformation front path. Thus, it is plausible that the superimposed stress field could lead to elevated levels of damage accumulation near the inclusions. As

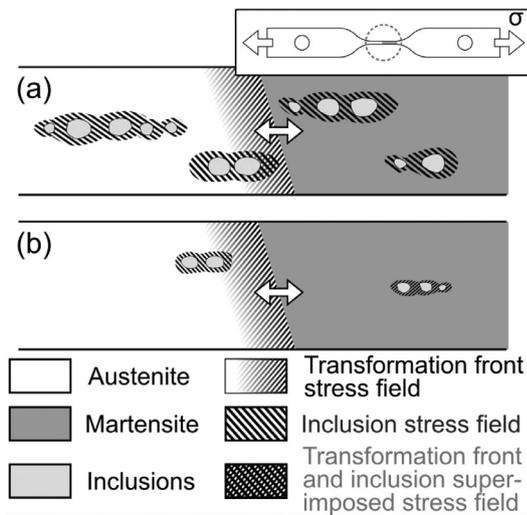


Fig. 4. An illustration of a proposed mechanism relating fatigue life to transformation volume amplitude and inclusion distribution. (a) A transformation front and inclusions in a standard-purity NiTi specimen. (b) A transformation front and inclusions in a high-purity specimen. The inclusions in (b) are fewer and smaller compared to (a).

the transformation front passes through the same collection of inclusions during cyclic loading and unloading, such damage could accumulate and eventually lead to the incubation of a crack and catastrophic fracture. Naturally, larger front motion would lead to a higher fracture probability.

In the case of a similar specimen manufactured from the high-purity NiTi alloy, fewer and smaller inclusions would be present in the gage as shown in Fig. 4(b) and as corroborated by the micrographs in Fig. 1(e, g). Thus, overall, fewer interaction events between a moving transformation front and inclusions would occur, leading to a smaller probability of damage accumulation to a critical level that would cause crack incubation. This rationalizes the relatively longer fatigue life observed in the high-purity dogbones in Fig. 3(a) vs. the standard-purity dogbones.

While explicit Lüders-like bands are not observed in the diamond specimens, under load, the volume of such specimens can still be partitioned into an austenite region and a martensite region. Thus, a similar damage accumulation mechanism as described above would operate in the diamond specimens.

Per the above-mentioned mechanism, the fatigue lifetime (N_F) depends on two factors related to inclusions. CTF inversely correlates to the extreme inclusion size or diameter (D_i) since larger inclusions would produce a stronger stress concentration. CTF inversely correlates to the number density of inclusions (ρ_i). Fatigue lifetime also inversely correlates with the transformation volume amplitude (Δv_{PT}). Thus, a phenomenological expression relating the low-cycle fatigue lifetime to the magnitude of the three factors can be written as

$$\log(N_F) = c(D_i\rho_i)^p(\Delta v_{PT})^q. \quad (1)$$

Here c , p , and q are unit-less constants.

We calculated the extreme inclusion diameter by fitting a generalized extreme value distribution [17,18] to the metallographic inclusion number density data shown in Fig. 1(d, f). The extreme diameter was found to be 5.4 μm for the standard-purity material and 3.8 μm for high-purity. We calculated the cumulative inclusion density from the frequency vs. size plots in Fig. 1(d, f). The inclusion density was obtained as $2.231 \times 10^9 \text{ mm}^{-2}$ for the standard-purity material and $5.549 \times 10^8 \text{ mm}^{-2}$ for high-purity. Based on these values, we obtained the fitting parameters in Eq. (1) as $c = 822.02$, $p = -0.23316$, and $q = -0.37221$. See Supplementary Ma-

terial for details of these calculations. A fitted model per the parameters determined above is shown in Fig. 3(b). The model reasonably captures the fatigue life of both the standard-purity and high-purity NiTi specimens.

While this work focussed on low-cycle fatigue, cyclic bio-mechanical processes such as the cardiac rhythm typically subject NiTi implants to fatigue-type loading of relatively small amplitude. Thus, high-cycle fatigue is more relevant in the context of implants. Intuitively, this mechanism would also be applicable in the high-cycle fatigue regime. During high-cycle fatigue, there is essentially no change in the macroscopic transformed volume. However, inclusions still harbor local stress fields that can cause localized transformation and lead to local damage accumulation. This aids in rationalizing the observations of Alarcon et al. [16], who reported that the fatigue life decreases before the maximum nominal stress during superelastic fatigue loading reaches the martensitic transformation onset stress value.

These results could be consequential to the NiTi material purity selection and design of medical implants in various ways. Minimization of phase transformation volume amplitude may be used as an implant design optimization strategy. A higher-purity NiTi alloy with smaller but more numerous inclusions may perform poorer compared to a standard-purity alloy. While this work did not focus on the effects of surface flaws, it is possible that surface flaws larger than a critical size may be detrimental to the fatigue life of a NiTi implant as much as non-metallic inclusions.

This work motivates future efforts directed at the following aspects. Micro-scale characterization studies may elucidate the precise mechanism for damage accumulation and crack incubation around individual inclusions. The dependence of crack incubation and fatigue life on the inclusion-matrix bonding, presence of any voids, and the inclusion aspect ratio may be quantified. The accuracy of the principal-strain-based segmentation process illustrated in Fig. 2 may be verified using a direct transformation volume quantification method such as X-ray diffraction. The mean transformation volume was not controlled in this study and remains a variable worth investigating.

In summary, we introduced a metric – transformation volume amplitude – to quantify the extent of reversible phase transformation during a fatigue cycle. We demonstrated that the fatigue lifetimes in the low-cycle fatigue regime in superelastic NiTi SMA inversely correlate to a combination of two factors: the transformation volume amplitude during a cycle and the number of inclusions interacting with the phase transformation domains. A transformation front + inclusion stress field based mechanism is proposed to rationalize these results.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Supplementary material

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2019.12.014.

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