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EFFECTS OF *IN SITU* PHASE TRANSFORMATION ON
FATIGUE-CRACK PROPAGATION IN TITANIUM-NICKEL SHAPE-MEMORY ALLOYS

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ABSTRACT

The fatigue-crack propagation behavior of near-equiatomic Ti-Ni shape-memory alloys has been investigated over a wide spectrum of growth rates from 10^{-11} to 10^{-6} m/cycle. Studies have been performed at room temperature in both non-transforming microstructures (stable austenite and stable martensite) and transforming austenitic microstructures (selected to undergo an *in situ* reversible or non-reversible stress-induced shear transformation to martensite). Crack-growth rates are found to be faster, and fatigue threshold ΔK_{TH} values to be lower, than in other metallic engineering alloys of comparable strength; values of ΔK_{TH} vary from 5.4 to 1.6 MPa \sqrt{m} in the stable and unstable (reversible) austenitic microstructures, respectively. Fatigue-crack growth rates are found to be much slower in the non-transforming microstructures; the occurrence of the *in situ* transformation, whether reversible or irreversible, leads to a significant increase in growth rates and a 50 to 70% decrease in the threshold ΔK_{TH} . Reasons for such behavior are briefly discussed in terms of the inherent properties of the parent and product phases, the energy of transformation, and dilatant and largely shear components of the phase change, and the role of these factors in suppressing shear localization and inducing crack-tip shielding.

INTRODUCTION

Near-equiatomic Ti-Ni alloys have become well known for their shape-memory properties, where, following deformation at one temperature, they can completely recover their original shape when heated to a higher temperature. Such properties are the result of a thermoelastic martensitic phase transformation, wherein apparent plastic deformation of the low-temperature martensitic phase is recovered on heating and reverting to the higher-temperature austenitic phase [1-3]. Such unique properties have led to considerations of shape-memory alloys for a wide range of applications, such as solid-state heat engines, electrical connectors, fasteners, couplings, and numerous bio-engineering and medical products.

Although many of the potential applications of shape-memory alloys involve alternating loading, there is a paucity of basic engineering fatigue-crack propagation data for these materials in the literature [4,5]. Early work on Ti-Ni alloys by Melton and Mercier [4,6], however, found that although the 10^7 -cycle fatigue limit (which essentially characterizes crack initiation) decreased with increasing martensite-start temperature, M_s , crack-growth rates were unaffected by the value of M_s ; in fact, over the range 10^{-10} to 10^{-6} m/cycle, they reported that growth rates were identical for Ti-Ni in the stable martensitic ($M_s = 47^\circ\text{C}$) and unstable austenitic ($M_s = 20^\circ\text{C}$) conditions. This may be regarded as somewhat surprising as analogous studies on the fatigue of unstable austenitic stainless steels [7] and on the toughness of partially-stabilized zirconia ceramics [8] have shown that in the presence of an *in situ* phase transformation, resistance to crack advance can be significantly enhanced. However, in both the latter examples, the transformation involves a significant and positive dilatational component, which due to the constraint of surrounding elastic

(untransformed) material, results in crack extension into a zone of compressed material [8,9]; the transformation in Ti-Ni alloys conversely involves largely pure shear with only a small, negative volume change [2].

In light of the limited data and uncertainty over the role of the transformation on the crack-growth properties of Ti-Ni alloys, the current work was undertaken specifically to compare the fatigue-crack propagation behavior of stable (non-transforming) and unstable (transforming) microstructures in these materials. The intent was to isolate the influence of the predominantly shear transformation on the development of crack-tip shielding (i.e., reductions in the local "crack driving force"), and to define how this shielding in turn affects the resulting crack-growth rate behavior.

EXPERIMENTAL PROCEDURES

A series of near-equiatomic Ti-Ni alloys was cast by Raychem Corporation to give at room temperature both stable (non-transforming) microstructures, namely a stable austenite (B12) and a stable martensite (B19'), and unstable (transforming) austenitic microstructures, which undergo reversible and irreversible stress-induced transformations to martensite; the difference in transformation properties was achieved by minor compositional changes and heat treatment. Optical micrographs of the resulting microstructures (prior to testing) are shown in Fig. 1. It is apparent that the stable and unstable (reversible) austenitic structures are similar (Figs. 1a,c), the latter showing evidence of twins (presumably from polishing) within the austenite grains. The stable martensite and unstable (irreversible) austenite structures conversely are both martensitic (Figs. 1b,d), the latter structure having undergone transformation during polishing. Schematic illustrations of the constitutive behavior of these microstructures are shown in Fig. 2; corresponding critical temperatures and uniaxial tensile properties are listed, respectively, in Tables I and II.

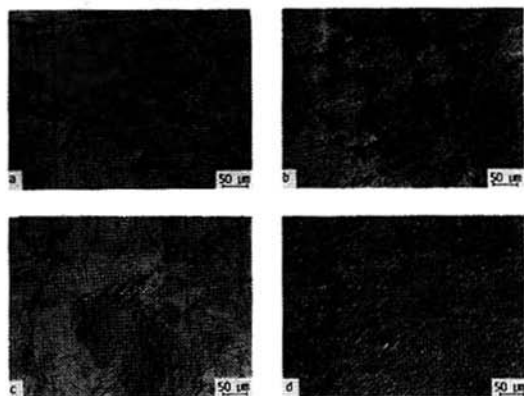


Fig. 1. Optical micrographs of the microstructures of near-equiatomic Ti-Ni shape-memory alloys, showing a) stable austenitic microstructure (B12), b) stable martensitic microstructure (B19'), and unstable austenitic microstructures which undergo c) reversible and d) irreversible stress-induced transformations to martensite.

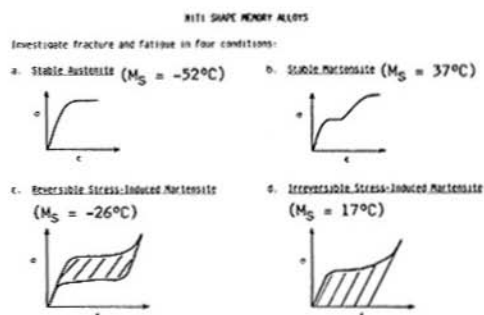


Fig. 2. Schematic illustrations of the room-temperature constitutive (σ/ϵ) behavior of a) stable austenitic, b) stable martensitic, and c,d) unstable austenitic microstructures in Ti-Ni shape-memory alloys.

Table I. Deformation Characteristics of Ti-Ni Alloys

Condition	M_S	M_f	A_S	A_f
		($^\circ\text{C}$ at a stress of 69 MPa)		
stable austenite	-52	-64	-15	-13
stable martensite	37	22	79	96
reversible stress-induced martensite	-26	-76	15	37
irreversible stress-induced martensite	17	-1	47	50

Table II. Room-Temperature Uniaxial Tensile Properties of Ti-Ni Alloys

Condition	Young's Modulus, E^+ (GPa)	First "Yield" Strength, σ_y (MPa)	Tensile Strength, σ_u (MPa)
stable austenite	85	605	800
stable martensite	45	96*	807
reversible stress-induced martensite	75	183	-
irreversible stress-induced martensite	75	262	931

*apparent yielding due to martensite twin arrangement.

+approximate value for loading only (significant variation with temperature and unloading).

Fatigue-crack propagation studies were performed on 10-mm-thick compact tension C(T) specimens, containing long (> 17 mm) through-thickness cracks, which were cyclically stressed at a load ratio R (ratio of minimum to maximum load) of 0.1 and frequency of 50 Hz (sine wave) in computer-controlled electro-servo-hydraulic testing machines; tests were conducted in an environment of controlled room air (22°C , 45% relative humidity). Electrical-potential measurements across ~ 5 - μm -thick NiCr foils (Krac[®]

gauges), bonded onto the specimen surface, were used to monitor crack lengths to a resolution better than $\pm 5 \mu\text{m}$; unloading compliance measurements using back-face strain gauges were similarly used to assess the extent of fatigue crack closure in terms of the stress intensity K_{cl} at first contact of the fracture surfaces during the unloading cycle [10]. Crack-growth rates, da/dN , were determined over the range 10^{-11} to 10^{-6} m/cycle under computed-controlled K-decreasing and K-increasing conditions, with a normalized K-gradient of 0.80 mm^{-1} [11]; data are presented in terms of the applied stress-intensity range ($\Delta K = K_{\text{max}} - K_{\text{min}}$, where K_{max} and K_{min} are, respectively, the maximum and minimum stress intensities in the fatigue cycle).

RESULTS

The variation in fatigue-crack growth rates, da/dN , as a function of stress-intensity range, ΔK , for the four Ti-Ni alloys is plotted in Fig. 3; this represents the widest spectrum of growth rates measured on a shape-memory alloy to date. Each microstructure shows a well-defined fatigue threshold stress intensity, ΔK_{TH} , below which fatigue-crack growth appears dormant. Compared to other engineering alloys (e.g., steels, aluminum and titanium alloys [10,12]) of similar strength levels, however, values of ΔK_{TH} are low, ranging from $5.4 \text{ MPa}\sqrt{\text{m}}$ in the stable austenitic structure to $1.6 \text{ MPa}\sqrt{\text{m}}$ in the unstable austenitic structure (with reversible transformation). Except above $\sim 10^{-8}$ m/cycle where growth rates in the former structure become accelerated, fatigue-crack growth rates are slowest in the stable martensitic and particularly the stable austenitic structures; somewhat surprisingly, growth-rates are fastest, and values of ΔK_{TH} lowest, in the austenitic structures that undergo an *in situ* stress-induced transformation, particularly when the transformation is reversible.

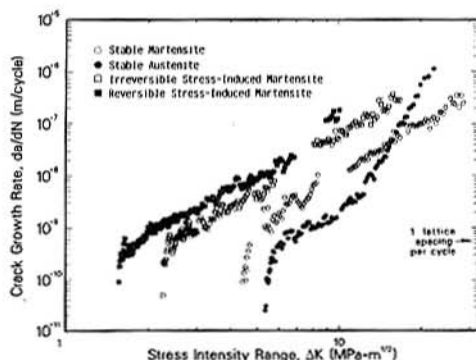


Fig. 3. Variation in fatigue-crack growth rates (da/dN) as a function of the stress-intensity range (ΔK) in near-equiatomic Ti-Ni shape-memory alloys at room temperature ($R = 0.1$). Note the faster growth rates and lower fatigue threshold ΔK_{TH} values in the transforming microstructures, compared to the stable austenitic and stable martensitic microstructures.

Corresponding data on crack-tip shielding, specifically on the role of crack closure, could not be deduced from the back-face strain compliance measurements in these alloys owing to their already nonlinear "elastic" load-displacement response on unloading. Alternative experimental methods to detect the crack-face contact associated with crack closure, and in fact to quantify other salient shielding mechanisms in shape-memory materials, are currently under investigation [13].

Fatigue fracture surfaces in the stable microstructures were relatively featureless, except for marked evidence of the underlying martensitic lath structure in the stable martensite failures. Fracture surfaces in the unstable austenites resembled that of the stable austenite or stable

martensite depending, respectively, whether the transformation was reversible or irreversible; fracture surfaces where the transformed martensite was stable also showed evidence of the B19' lath structure.

DISCUSSION

There are several somewhat surprising features about the results described above. Firstly, as noted above, fatigue-crack growth rates in Ti-Ni shape-memory alloys are relatively fast, and fatigue threshold ΔK_{TH} values relatively low, compared to other metallic alloys of similar strength levels. For example, steels of comparable yield strength have thresholds in the range 8 to 10 MPa \sqrt{m} and of comparable tensile strength in the range 5 to 8 MPa \sqrt{m} [12]. Secondly, in contrast to previous studies [4], growth rates are sensitive to the value of M_S , although the variation in crack-growth resistance (characterized by ΔK_{TH}) is not a linear function of M_S (Fig. 4). Thirdly and most importantly, *in situ* crack-tip phase transformations in these alloys do not result in improved crack-growth properties; in fact conversely, faster growth rates are observed in the unstable alloys.

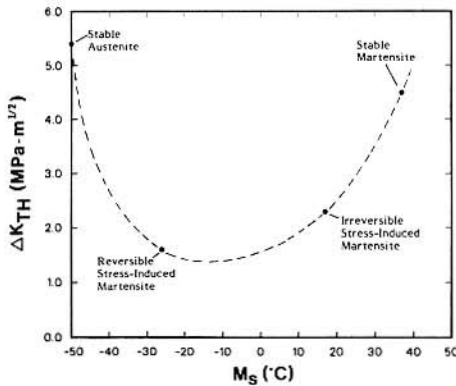


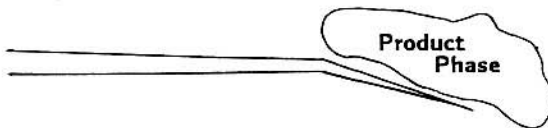
Fig. 4. Variation of the fatigue threshold (ΔK_{TH}) for crack propagation in Ti-Ni shape-memory alloys as a function of the martensite-start temperature, M_S . Threshold data are taken from the room-temperature results plotted in Fig. 3.

To attempt to rationalize the latter phenomenon, it should be noted that the role of an *in situ* transformation on crack-growth behavior may be extremely complex, as it involves such factors as the intrinsic properties of the parent and product phase (where, for example, significant differences in the Young's modulus and fracture toughness are expected), the energy expended by the transformation (including adiabatic heating effects near the crack tip) and the effect of the phase change in suppressing strain localization and inducing crack-tip shielding. To consider briefly each point in turn, for the present case there is clearly a difference in the intrinsic fatigue-crack growth resistance of the parent and product phases; if the stable martensite is representative of that formed on irreversible transformation, then with reference to Fig. 3, crack-growth rates are enhanced in the transformed martensite phase, at least below 10^{-7} m/cycle. Conversely, the transformation provides a steady source of hardening, which stabilizes plastic flow and acts to suppress strain localization [14]; this presumably favors resistance to crack advance, although since the mechanism of fatigue-crack growth in these alloys is not known, the specific role of strain localization is uncertain. Similarly, consideration of the energy expended in the transformation would tend to suggest slower growth rates in the transforming microstructures, simply because of the increased work of fracture and the effect of the transformation in enlarging the inelastic zone surrounding the crack [8,9]. Finally, in contrast to the predominantly dilatant phase transformations in steels [7] and zirconia ceramics [8],

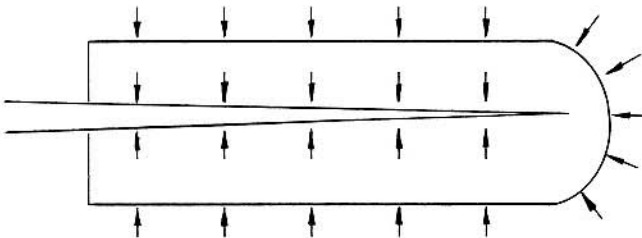
which are the principal source of toughening in these materials, the volume change in Ti-Ni is small and negative (-0.54%) [2]. Considering the development of crack-tip shielding due to the constraint of surrounding elastic material on the transformed zone [8], this negative dilation predicts an *increase* in the local stress intensity ("anti-shielding"), inferring conversely faster growth rates in the transforming microstructures.

Clearly, the fatigue behavior observed in the current shape-memory alloys is a result of these, and perhaps other, mechanisms operating in concert (Fig. 5). A precise understanding of the relative fatigue-crack growth resistance of the stable and unstable Ti-Ni microstructures, however, must await quantification of these mechanisms; such work is currently in progress.

* Crack Deflection



* Transformation Toughening (principally a "wake effect")



* Transformation Plasticity (near tip effect)

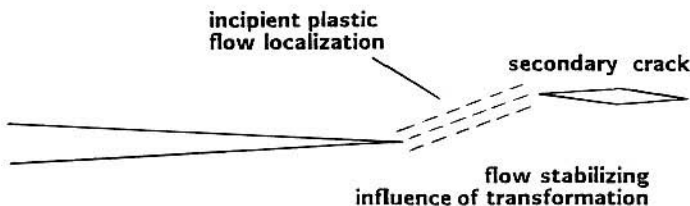


Fig. 5. Possible mechanisms for the role of an *in situ* stress-induced phase transformation in inducing improved resistance to fatigue-crack propagation in shape-memory alloys.

CONCLUSIONS

Based on a study of the growth of fatigue cracks in unstable (transforming) and stable (non-transforming) microstructures in near equiatomic Ti-Ni shape-memory alloys, the following conclusions can be made:

1. Fatigue-crack growth rates in four near-equiatomic Ti-Ni shape-memory alloys have been characterized over the range 10^{-11} to 10^{-6} m/cycle; the alloys represent a stable austenitic, a stable martensitic, and two unstable austenitic microstructures which undergo either reversible or irreversible *in situ* stress-induced phase transformations.

2. Fatigue-crack growth rates in Ti-Ni are significantly faster, and fatigue threshold values (ΔK_{TH}) significantly lower, compared to other metallic engineering alloys of similar strength. Values of ΔK_{TH} in Ti-Ni vary from 5.4 MPa \sqrt{m} in the stable austenitic structure to 1.6 MPa \sqrt{m} in the unstable austenitic structure undergoing a reversible transformation to martensite. Growth rates and ΔK_{TH} values, however, are not a simple function of the martensite-start temperature (M_s).

3. Contrary to first-order expectations, fatigue-crack growth rates are slowest in the stable (non-transforming) microstructures, particularly the stable austenite, and fastest in the unstable (transforming) microstructures, particularly involving a reversible transformation to martensite. Although the reasons for such behavior are uncertain, the lack of a significant crack-tip shielding effect is considered to result in part from the small, negative dilation associated with the transformation.

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References

1. K. Otsuka and K. Shimizu, *Int. Metals Rev.* **31**, 93 (1986).
2. C. M. Jackson, H. J. Wagner, and R. J. Wasilewski, NASA Report SP 5110 (1972).
3. H. A. Mohamed and J. Washburn, *Metall. Trans. A* **7A**, 10 (1976).
4. K. N. Melton and O. Mercier, *Acta Metall.* **27**, 137 (1979).
5. S. Miyazaki, M. Suizu, K. Otsuka, and T. Takashima, this volume (1988).
6. K. N. Melton and O. Mercier, *Mater. Sci. Eng.* **40**, 81 (1979).
7. E. Hornbogen, *Acta Metall.* **26**, 147 (1978).
8. R. M. McMeeking and A. G. Evans, *J. Am. Ceram. Soc.* **65**, 242 (1982).
9. B. Budiansky, J. W. Hutchinson, and J. C. Lambropoulos, *Int. J. Solid Struct.* **19**, 337 (1983).
10. R. O. Ritchie and W. Yu, in Small Fatigue Cracks, edited by R. O. Ritchie and J. Lankford (TMS-AIME, Warrendale, PA, 1986), p. 167.
11. ASTM Standard E 647-86A, in ASTM Annual Book of Standards (American Soc. Test. & Mats., Philadelphia, PA, 1987), vol. 3.01, p. 899.
12. R. O. Ritchie, *Int. Metals Rev.* **20**, 205 (1979).
13. R. H. Dauskardt and R. O. Ritchie, unpublished work, LBL (1988).
14. G. B. Olson and M. Cohen, in Mechanical Properties and Phase Transformations in Engineering Materials, edited by S. D. Antolovich, R. O. Ritchie and W. W. Gerberich (TMS-AIME, Warrendale, PA, 1986), p. 367.