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**Unified Theory and Experimentation for Fatigue and Fracture of High
Temperature Shape Memory Alloys**

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14. ABSTRACT
n this project, the team has - proposed a new analysis method crack growth under mechanical and actuation loading that provides a unified description for fatigue crack growth in shape memory alloys; - reported the observation of stable crack growth under thermal cycling in shape memory alloys for the first time; - investigated thermal stability of two-way shape memory effect in nano-precipitation hardened NiTiHf high temperature shape memory alloy tubes; - investigated the effects of training on the thermomechanical behavior of nano-precipitation strengthened NiTiHf and NiTiZr high temperature shape memory alloys; -measured fatigue crack growth rates in NiTiHf high temperature shape memory alloys under thermomechanical loading for the first time; - experimentally investigated the thickness dependence of the fracture toughness and fracture micromechanisms in NiTi shape memory alloys; - investigated the stress-induced transformation toughening by measuring the fracture toughness of NiTiHf high temperature shape memory alloys at different temperatures; -studied the effect of microstructure on the fracture toughness of NiTiHf high temperature shape memory alloys by comparing precipitated and solutionized microstructures; -experimentally demonstrated that the toughness enhancement due to crack advance in hysteretic materials may be "reversed" by partial unloading; - numerically investigated void growth and coalescence in shape memory alloys by unit cell simulations and quantified the importance of fracture mechanisms; - simulated crack growth under different thermomechanical loadings using a shape memory alloy constitutive model and validated the results against experimental data; - proposed a phenomenological phase-field model for the formation and growth of fatigue macro-cracks that can reproduce both total life and defect tolerant approaches.

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Unified Theory and Experimentation for Fatigue and Fracture of High-Temperature
Shape Memory Alloys
For the Period of June 15, 2018 – June 14, 2021
Ibrahim Karaman, Theocharis Baxevanis, and Dimitris C. Lagoudas

The following provides a summary of studies that have been supported through the AFOSR project Grant FA9550-18-1-0276.

In this project, the team has

- investigated thermal stability of two-way shape memory effect in nano-precipitation hardened NiTiHf high temperature shape memory alloy tubes;
- investigated the effects of training on the thermomechanical behavior of nano-precipitation strengthened NiTiHf and NiTiZr high temperature shape memory alloys;
- reported the observation of stable crack growth under thermal cycling in shape memory alloys for the first time;
- proposed a new analysis method crack growth under mechanical and actuation loading that provides a unified description for fatigue crack growth in shape memory alloys;
- measured fatigue crack growth rates in NiTiHf high temperature shape memory alloys under thermomechanical loading for the first time;
- experimentally investigated the thickness dependence of the fracture toughness and fracture micromechanisms in NiTi shape memory alloys;
- investigated the stress-induced transformation toughening by measuring the fracture toughness of NiTiHf high temperature shape memory alloys at different temperatures;
- studied the effect of microstructure on the fracture toughness of NiTiHf high temperature shape memory alloys by comparing precipitated and solutionized microstructures;
- experimentally demonstrated that the toughness enhancement due to crack advance in hysteretic materials may be “reversed” by partial unloading;
- numerically investigated void growth and coalescence in shape memory alloys by unit cell simulations and quantified the importance of fracture mechanisms;
- simulated crack growth under different thermomechanical loadings using a shape memory alloy constitutive model and validated the results against experimental data;
- proposed a phenomenological phase-field model for the formation and growth of fatigue macro-cracks that can reproduce both total life and defect tolerant approaches.

Publications

1. Young, B., Orrostieta R., Haghgouyan, B., Lagoudas, D.C., Karaman, I., On the effect of microstructure on crack growth resistance of NiTiHf under thermomechanical loading. *In preparation*.
2. Makkar, J., Young, B., Karaman, I., Baxevanis, T., Fracture resistance of shape memory alloys under thermomechanical loading. *Submitted to Engineering Fracture Mechanics*.
3. Haghgouyan, B., Young, B., Picak, S., Baxevanis, T., Karaman, I. and Lagoudas, D.C., 2021. A unified description of mechanical and actuation fatigue crack growth in shape memory alloys, *Acta Materialia*, p.117155.
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Conference Presentations

1. B. Haghgouyan, “Fatigue crack growth in Ni-rich NiTiHf high temperature shape memory alloys under thermomechanical loading”, TMS, March 2021, Virtual.
2. M.M. Hasan, “A phase-field model for fatigue of brittle materials”, WCCM XIV and ECCOMAS 2020, January 2021, Virtual.
3. B. Haghgouyan, “Fatigue crack growth in shape memory alloys under mechanical and actuation loading”, ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems, September 2020, Virtual.
4. B. Haghgouyan, “Mechanical and actuation fatigue in Ni-Rich NiTiHf high temperature shape memory alloys” TMS, February 2020, San Diego, CA.
5. D. C. Lagoudas, “Crack growth behavior of NiTiHf high temperature shape memory alloys under thermo-mechanical loading”, International Conference on Plasticity, Damage, and Fracture, January 2020, Rivera Maya, Mexico.
6. T. Baxevanis, “On the mechanics of overload and fatigue failure in shape memory alloys”, ASME SMASIS, November 2019, Louisville, KY.
7. D. C. Lagoudas, “Fatigue crack growth and fracture behavior in NiTiHf high-temperature shape memory alloys”, EUROMAT, September 2019, Stockholm, Sweden.
8. B. Young, “Temperature effect on fracture toughness in NiTiHf high temperature SMA”, Shape Memory and Superelastic Technologies, May 2019, Konstanz, Germany.
9. T. Baxevanis, “On the mechanics of overload and fatigue failure in shape memory alloys”, IUTAM, May 2019, Austin, TX.
10. I. Karaman, “Engineering the transformation hysteresis by precipitation in NiTi and NiTiHf”, IUTAM, May 2019, Austin, TX.
11. B. Haghgouyan, “Fracture toughness and crack growth behavior in NiTi and NiTiHf shape memory alloys”, IUTAM, May 2019, Austin, TX.
12. I. Karaman, “Engineering martensitic transformation through compositional and microstructural control in shape memory alloys – optimum experiment design”, Adaptive Sample Preparation and Target Fabrication for High-Throughput Materials Science, May 2019, College Station, TX.
13. B. Haghgouyan, “Crack growth in shape memory alloys under thermomechanical loading”, SPIE Smart Structures & Nondestructive Evaluation, March 2019, Denver, CO.
14. D. C. Lagoudas, “On the fracture and crack growth behavior in SMAs”, International Conference on Plasticity, Damage, and Fracture, January 2019, Panama City, Panama.
15. B. Young. “Experimental determination of crack growth rates during actuation of NiTi and NiTiHf shape memory alloys”, ASME International Mechanical Engineering Congress & Exposition, November 2018, Pittsburgh, PA.
16. B. Haghgouyan, “Crack growth behavior in NiTi shape memory alloys under Mode-I isothermal loading: effect of stress state, ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems, September 2018, San Antonio, TX.
17. B. Young, “Experimental determination of crack growth rates during actuation of NiTi and NiTiHf shape memory alloys” ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems, September 2018, San Antonio, TX.

1. EXPERIMENTAL WORK

1.1 Structure, Processing and Properties

1.1.1 Two-way Shape Memory Effect in NiTiHf Torque Tubes

Two-way shape memory effect (TWSME) in nano-precipitation hardened, $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ high temperature shape memory alloy (HTSMA) thin walled tubes and its thermal stability were investigated. Torsional TWSME was induced by repeated thermal cycling across their martensitic transformation under applied shear stress. The effects of training parameters and geometric factors, such as the number of training cycles, shear stress levels, and thickness of the tube walls, on the resulting TWSME were evaluated. Thermal stability of TWSME was characterized as a function of annealing treatments at elevated temperatures. It was found that under 200 MPa, 600 thermal cycles were sufficient to reach a two-way shape memory strain (TWSMS) as high as 2.95%, which was shown to be stable upon annealing up to 400°C for 30 min. This TWSMS was 85% of the maximum measured actuation strain under 200 MPa. The microstructure after thermo-mechanical training was investigated using transmission electron microscopy (TEM), which did not indicate a significant change in precipitate structure and size after the training. However, a small amount of remnant austenite was revealed at 100°C below the martensite finish temperature, with a notable amount of dislocations. Overall, it was found that nano-precipitation hardened $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ shows relatively high TWSMS and stable actuation response after much smaller number of training cycles as compared to binary NiTi and nickel lean NiTiHf compositions. Tube wall thickness and training stress levels have been found to have negligible effect on shape memory strains and number of cycles to reach the desired training level, for the ranges studied.

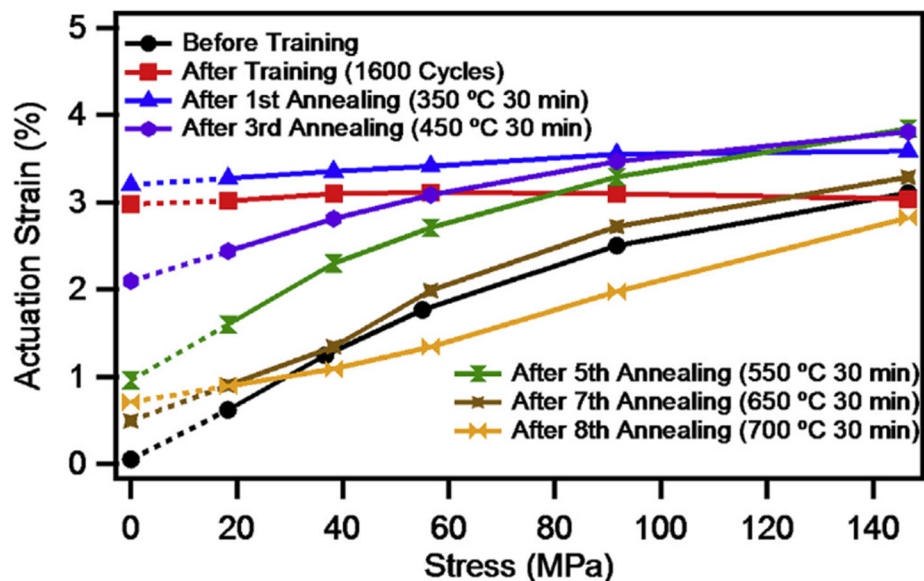


Figure 1. Actuation strain under various stresses in thermo-mechanically trained $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ high temperature shape memory alloy tube after static annealing treatments at various temperatures for 30 min [1].

1.1.2 Effect of Training on Thermomechanical Properties of NiTiHf and NiTiZr

The effects of constant load thermal cycles (training) on the thermomechanical behavior of nano-precipitation strengthened $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ and $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Zr}_{20}$ HTSMAs were compared. Thermomechanical properties were determined as a function of the number of training cycles, which consisted of up to 2000 isobaric thermal cycles at 300 MPa, between lower and upper cycle temperatures of 35 and 300°C, respectively. In addition, the stability of the trained alloys was determined after exposure to thermal treatments at temperatures above the upper cycle training temperature. Training at 300 MPa significantly improved the actuation strain capability of the NiTiHf HTSMA at low stresses (*i.e.*, 50 MPa) and resulted in a two-way shape memory strain (TWSMS) up to 1.9%, but essentially had no effect on the 300 MPa response. Training had less notable benefits in the case of the NiTiZr, producing negligible TWSMS, and resulting in a decrease in actuation strain capability at 300 MPa with repeated cycling. The benefits of training to the NiTiHf HTSMA were maintained after aging at 400°C but were lost after exposure to 500°C and above. Since training was not notably beneficial to the NiTiZr alloy and resulted in a loss in strain capability at 300 MPa, the high temperature annealing treatment actually recovered strain capability in the alloy under high stresses. The superior TWSM response of the NiTiHf HTSMA as compared to the NiTiZr, was attributed to the higher melting temperature, and thus the lower homologous operating temperature of the former, when both alloys were tested over the same temperature range.

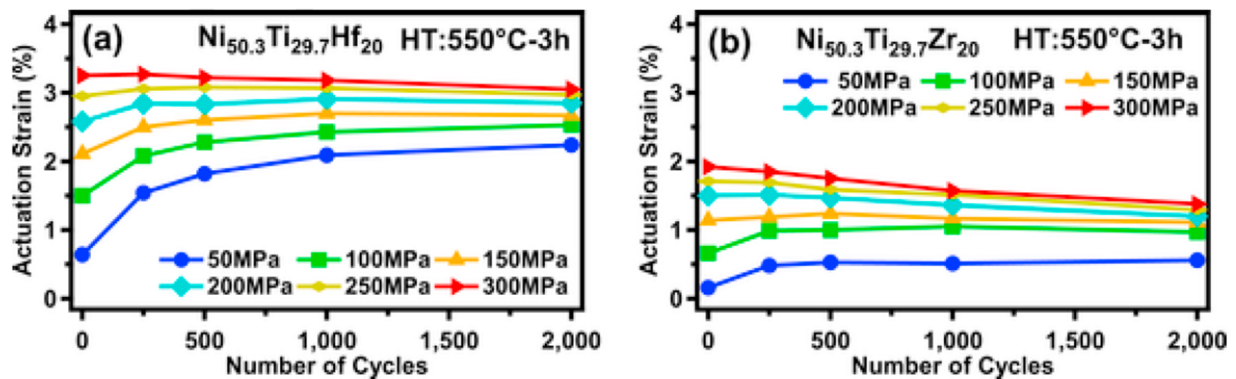


Figure 2. Evolution of actuation strain under various applied stress levels as a function of the number of 300 MPa training cycles for nano-precipitation hardened (a) $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ and (b) $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Zr}_{20}$ [2].

1.2 Failure Mechanisms in SMAs

1.2.1 Thickness Effect on Fracture Toughness of NiTi

The thickness dependence of the fracture toughness in NiTi SMA was investigated. To this aim, compact tension specimens with various thicknesses were tested at room temperature, while in fully martensitic state, and under mode-I loading. Stable crack growth was observed in all specimens, regardless of the thickness. The resistance curves were obtained from load–displacement data and the J -integral values were determined using a recently proposed methodology for SMAs. Critical J values were obtained and compared to reveal the thickness dependence of the fracture toughness. The average J_{Ic} values for different thicknesses obtained from J – R curves are presented in Figure 3a. Comparing the values from each thickness to the mean value obtained from all experiments, no specific trend was observed, suggesting no thickness effect

on the fracture toughness of martensitic NiTi in the thickness range investigated in this work. To investigate the fracture mechanism, the fracture surface analysis was carried out with scanning electron microscopy (SEM). Figure 3b shows SEM images of the fracture surface of NiTi CT specimens with thicknesses. The flat fracture surfaces suggest much less sensitivity of the fracture toughness to the specimen thickness. Moreover, the images reveal a quasi-cleavage fracture, i.e. dominated by cleavage exhibiting small parts with ductile tearing, further explaining the insensitivity of fracture toughness to the specimen thickness. The results suggest modifications towards relaxing the existing thickness requirements for fracture toughness measurements in SMAs.

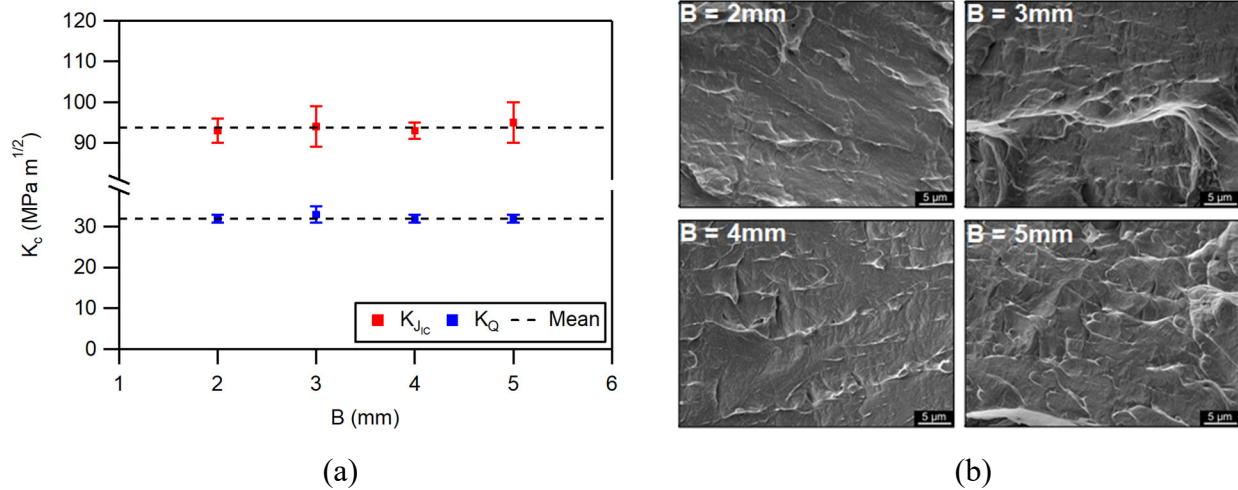


Figure 3. (a) Fracture toughness values for near equiatomic, NiTi shape memory alloy compact tension specimens with different thickness values and (b) SEM images showing the fracture surface of compact tension specimens with different thickness values showing a quasi-cleavage fracture mechanism [3].

1.2.2 Temperature Effect on Fracture Toughness of NiTiHf

The fracture toughness of Ni_{50.3}Ti_{29.7}Hf₂₀ HTSMA was systematically investigated as a function of temperature. A set of nominally isothermal fracture toughness tests were conducted on disk-shaped compact tension specimens at five temperatures corresponding to three thermodynamic conditions: (i) below martensite finish temperature M_f to obtain the fracture toughness of martensite, (ii) above martensite start temperature M_s in austenite, but below the martensite desist temperature M_d (the temperature above which austenite does not transform), in order to find the fracture toughness when stress induced martensitic (SIM) transformation takes place close to the crack tip, and (iii) above M_d , in order to obtain the fracture toughness of austenite. The extent of the inelastic zone near the crack tip was detected using digital image correlation (DIC), and the fracture surfaces were examined. The fracture behavior was highly temperature/phase dependent (Figure 4a). The fracture toughness of the transforming material was higher than that of austenite and martensite, i.e. SIM transformation acts as a toughening mechanism (Figure 4b). This was attributed to the differences in strain hardening behavior in detwinning, martensitic transformation, and plastic deformation regimes of the stress-strain response, where SIM transformation occurs with the lowest strain hardening rate. The fracture toughness values obtained here are lower than those of equiatomic NiTi.

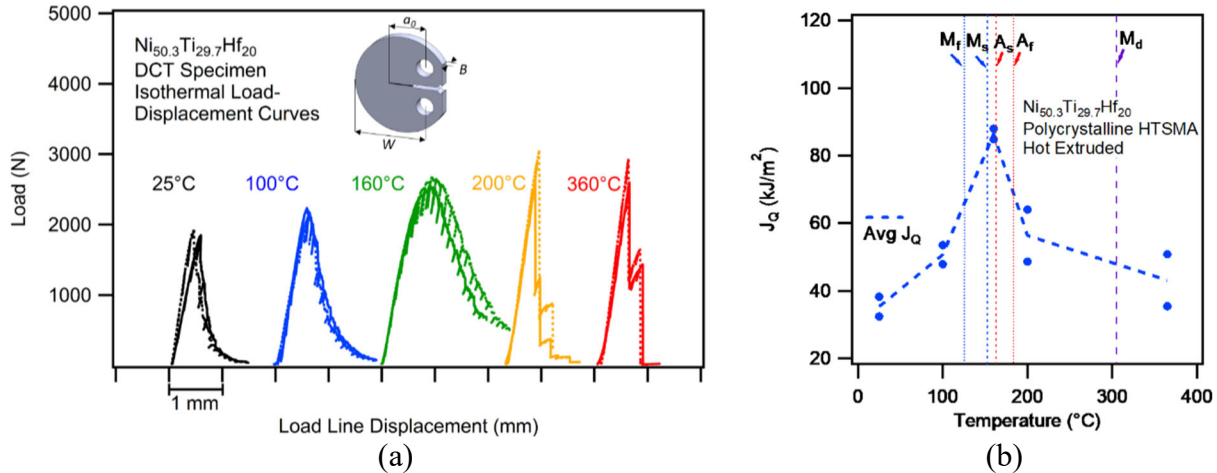


Figure 4. (a) Load-displacement curves and (b) J_Q fracture toughness values of $Ni_{50.3}Ti_{29.7}Hf_{20}$ high temperature shape memory alloy at 25°C ($M_f-100^\circ C$), 100°C ($M_f-25^\circ C$), 160°C ($A_s-5^\circ C$), 200°C ($A_f+15^\circ C$), and 360°C ($A_f+175^\circ C$) of disk-shaped compact tension specimens [4].

1.2.3 Microstructure Effect on Fracture Toughness of NiTiHf

The effect of microstructure on the fracture toughness of a $Ni_{50.3}Ti_{29.7}Hf_{20}$ HTSMA was investigated by conducting experiments on precipitated and solutionized microstructures. A set of nominally isothermal fracture toughness experiments were performed in each microstructure at approximately equivalent temperatures relative to the martensite start temperature (M_s). As shown in Figure 5, in all cases except for the purely austenite material, the solutionized material has higher fracture toughness than the precipitated material. This indicates that these precipitates, which are small, but incoherent with the matrix, can act as stress-concentrators for the crack and decrease overall ductility. For the austenitic material, repeated experiments are necessary to further investigate the effect of precipitation on fracture toughness.

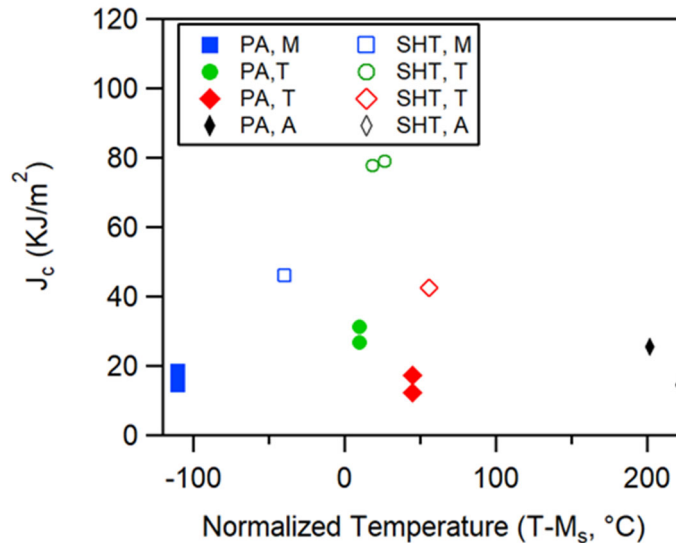


Figure 5. Fracture toughness (J_c) values for precipitated (solid markers) and solutionized (open markers) normalized to M_s temperature. The fracture toughness of solutionized material is higher than that of precipitated material in all cases except for the purely austenite material [5].

1.2.4 Fracture Resistance under Thermomechanical Loading in NiTi

Experimental measurements of the fracture resistance of SNiTi SMA under thermo-mechanical loading conditions were conducted where compact tension specimens were subjected to either isothermal mechanical (Figure 6a) or isobaric thermal loading (Figure 6b). The latter loading path is an idealization of typical loading paths that utilize these alloys as actuators. A single-parameter description of the experimental data was employed based on a path-independent contour integral, which was approximated by the load vs load-line displacement curves recorded from the experiments. A one-parameter interpretation of the experimental data from fracture experiments on SMAs under coupled thermo-mechanical loading was reported. The obtained results, suggest that (i) the employed contour integral should achieve similitude for a wide range of thermomechanical loading conditions and geometric configurations, and that (ii) isothermal conditions provide a more pronounced toughness enhancement as compared to thermal loading under a constant bias load.

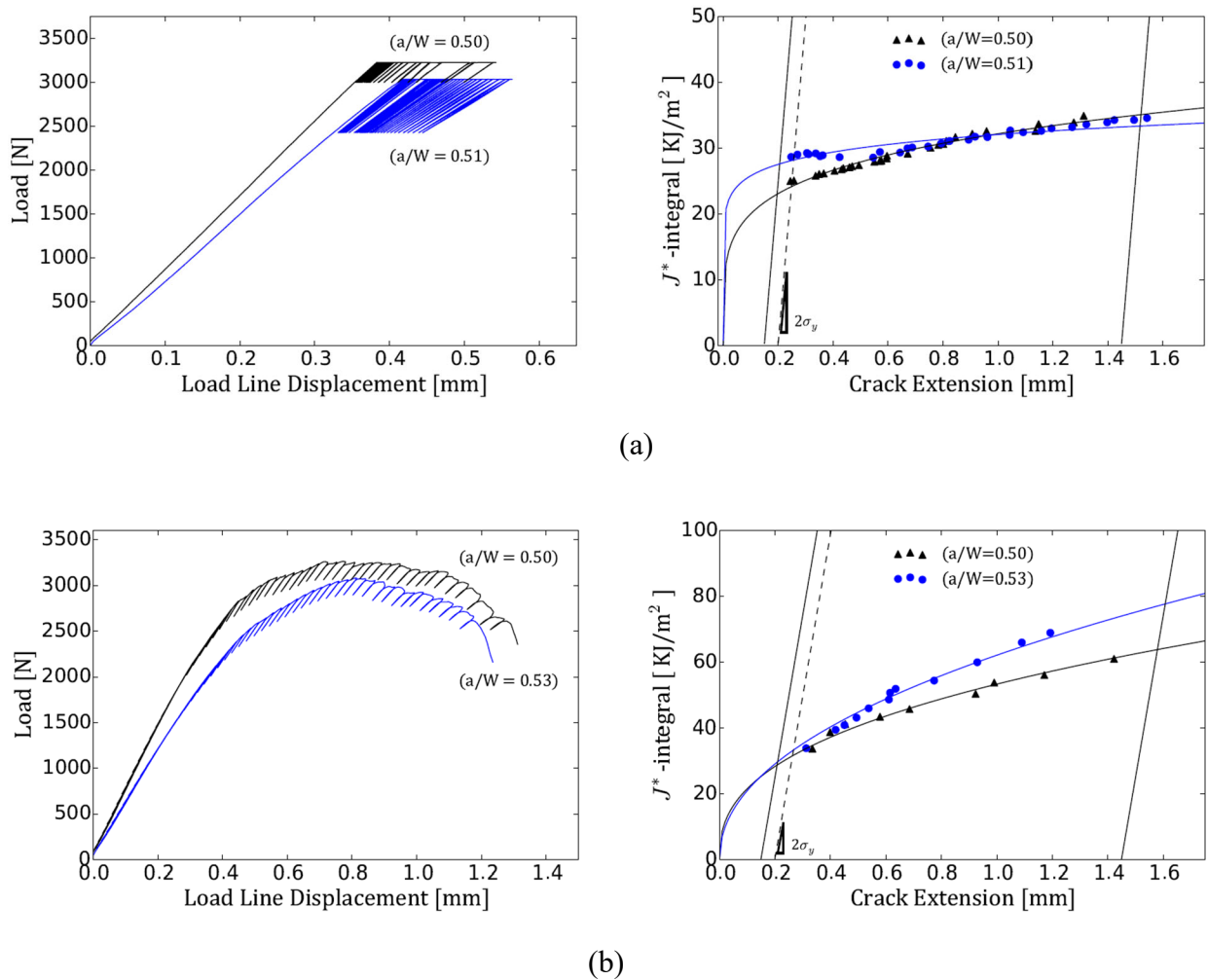


Figure 6. Load vs load-line displacement experimental plots (left) alongside resistance curves for these experiments (right) for (a) isothermal and (b) isobaric loading conditions [6].

1.2.5 Effect of Reversible Phase Transformation on Fracture Toughness of NiTi

The effect of reverse phase transformation on the fracture toughness of a Ni-rich NiTi alloy was explored. Compact tension specimens were tested under isothermal mechanical loading at a temperature at which phase transformation is reversible (superelastic loading). The loading paths included unloading to induce reverse phase transformation beyond that occurring during crack advance. The load vs load-line displacement curves are presented in Figure 7. A critical difference between the obtained response and the respective one of conventional ductile metals and non-superelastic SMAs was found to be the absence of a master curve, i.e. the load-displacement curve after the unloading/reloading cycle did not return to the point at which unloading took place. This response should be attributed to the recovery of the phase transformation strains left in the wake of the growing crack that are not fully restored during reloading, and the different direction and magnitude of the martensite variants formed upon reloading in the active transformation zone. The experimental observations offer evidence that (i) even a partial unloading during stable crack growth may have a profound impact on the fracture response, (ii) the compliance method for constructing the R -curve should be used with caution, and (iii) the validity conditions of R -curves are stricter in hysteretic materials than in conventional structural metals and, thus, the R -curve as the means to study the stability of a real structure is even less effective.

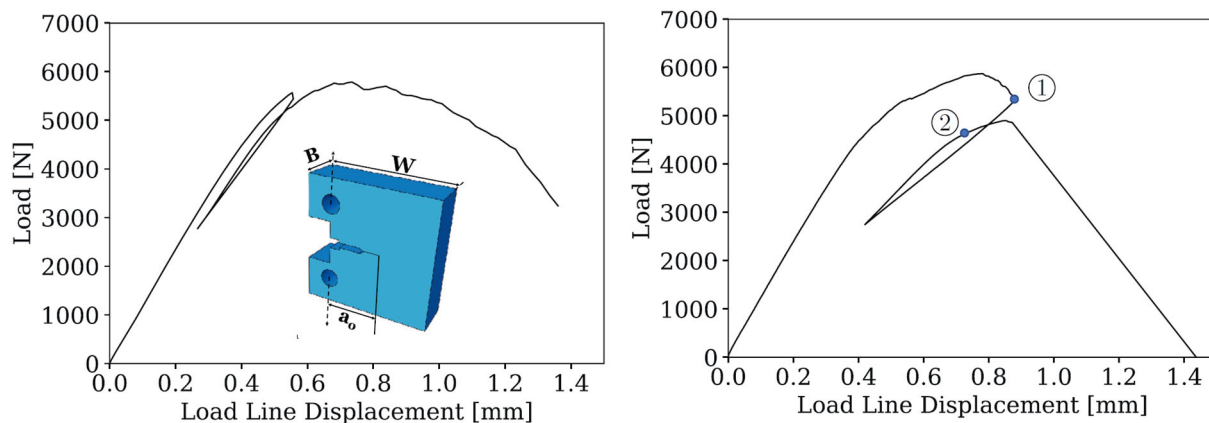


Figure 7. Experimental load vs load-line displacement curves for compact tension specimen of superelastic NiTi unloaded once to 50% of the load at the instant of unloading [7].

1.2.6 Unified theory of mechanical and actuation fatigue crack growth

A new analysis method for actuation and mechanical fatigue crack growth rates is proposed that provides a unified description for fatigue crack growth in shape memory alloys. This allows estimating actuation fatigue crack growth rates, laborious and challenging to measure, from easier to detect mechanical fatigue crack growth rates. To test the proposed analysis method, purely mechanical and actuation fatigue crack growth experiments were performed on a Ni-rich NiTiHf high-temperature shape memory alloy for the first time. In the former, cyclic mechanical loading was performed at a constant temperature (Figure 8a), while in the latter, the load was kept constant while the temperature was cycled in a range in which the stable phase was alternating in every

cycle (Figure 8b). A Paris-type power-law crack growth expression based on the cyclic J -integral was shown to fit the fatigue crack growth rate data from all experiments with a single set of parameters (Figure 8c). The obtained results suggest that a single empirical power-law expression can achieve the required similitude among a wide range of loading conditions and geometric configurations to provide a unified description of thermomechanical fatigue in shape memory alloys. Fatigue fracture surface analysis revealed quasi-cleavage failure for both mechanical and actuation crack growth where frequent presence of hafnium oxides on the surface indicated their deteriorating effect on the crack growth resistance.

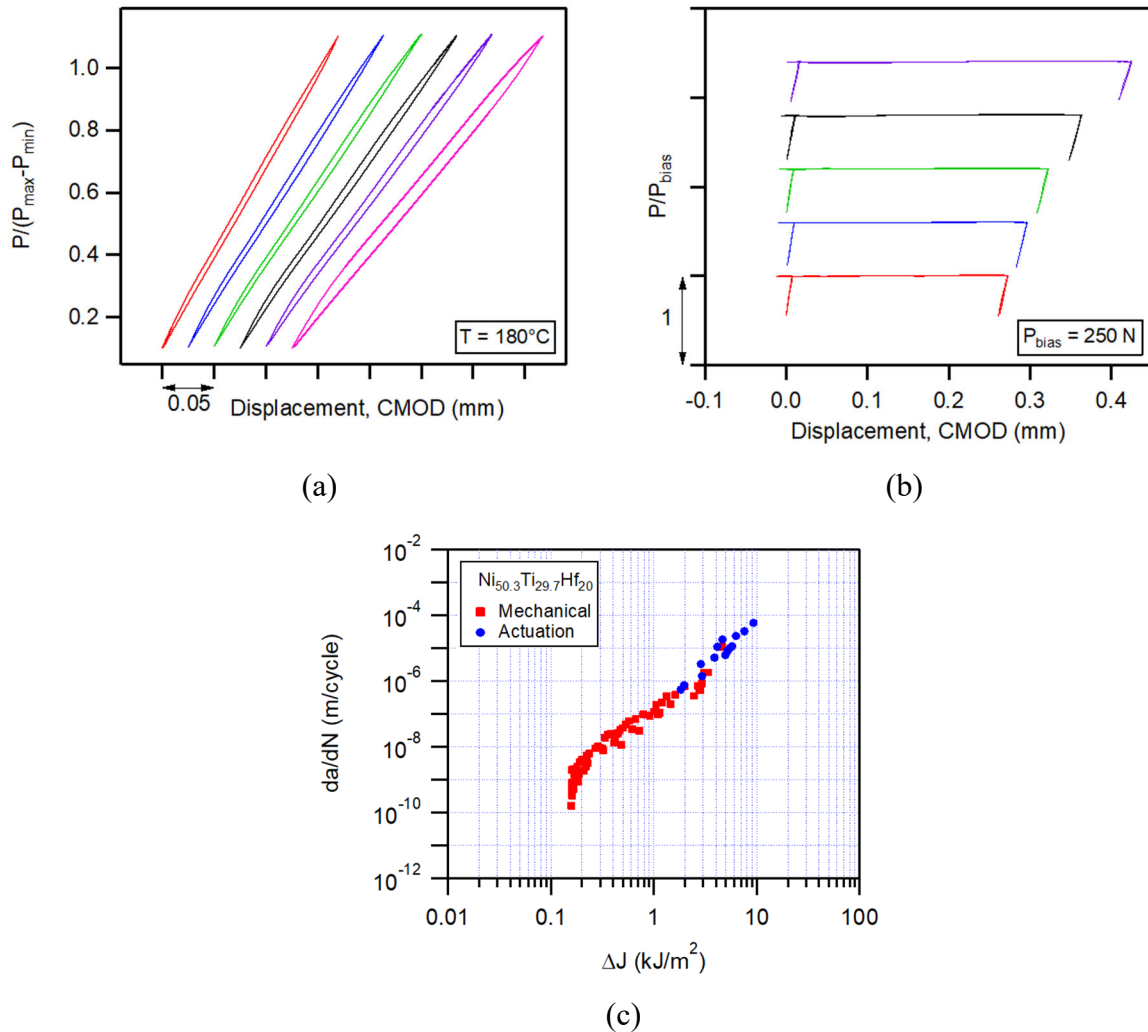


Figure 8. Experimental results from mechanical and actuation fatigue crack growth experiments on NiTiHf DCT specimens: (a) load-displacement curves during mechanical cycling at 180°C , (b) load-displacement data during actuation cycling under a constant bias load of 250 N, and (c) ΔJ vs da/dN plot for mechanical and actuation fatigue crack growth demonstrating unified description for thermomechanical fatigue of SMAs [8].

2. MODELING WORK

2.1 Local Approach

2.1.1 Void Growth and Coalescence

Unit cell simulations are employed to quantify the relative importance of void growth and coalescence in SMAs in the overall failure response of these materials. The numerical simulations involve a single pre-existing void of various size and shape (Figure 9a), assumed to have initiated from a second phase particle, embedded in a SMA matrix. The numerical simulations reveal that at the onset of coalescence, the porosity increases rapidly, the void aspect ratio decreases rapidly, and the load drops abruptly. Coalescence of the voids occurs only by plastic localization due to the “finite” nature of the phase-transformation- induced strains. The point of coalescence is always at the descending part of the effective stress vs effective strain curve, *i.e.*, past the peak stress points (Figure 9b). More importantly for the present study, the strain at the peak volume averaged effective stress increases with decreasing stress triaxiality, decreasing initial porosity and increases with initial aspect ratio and phase transformation (Figure 9c and d).

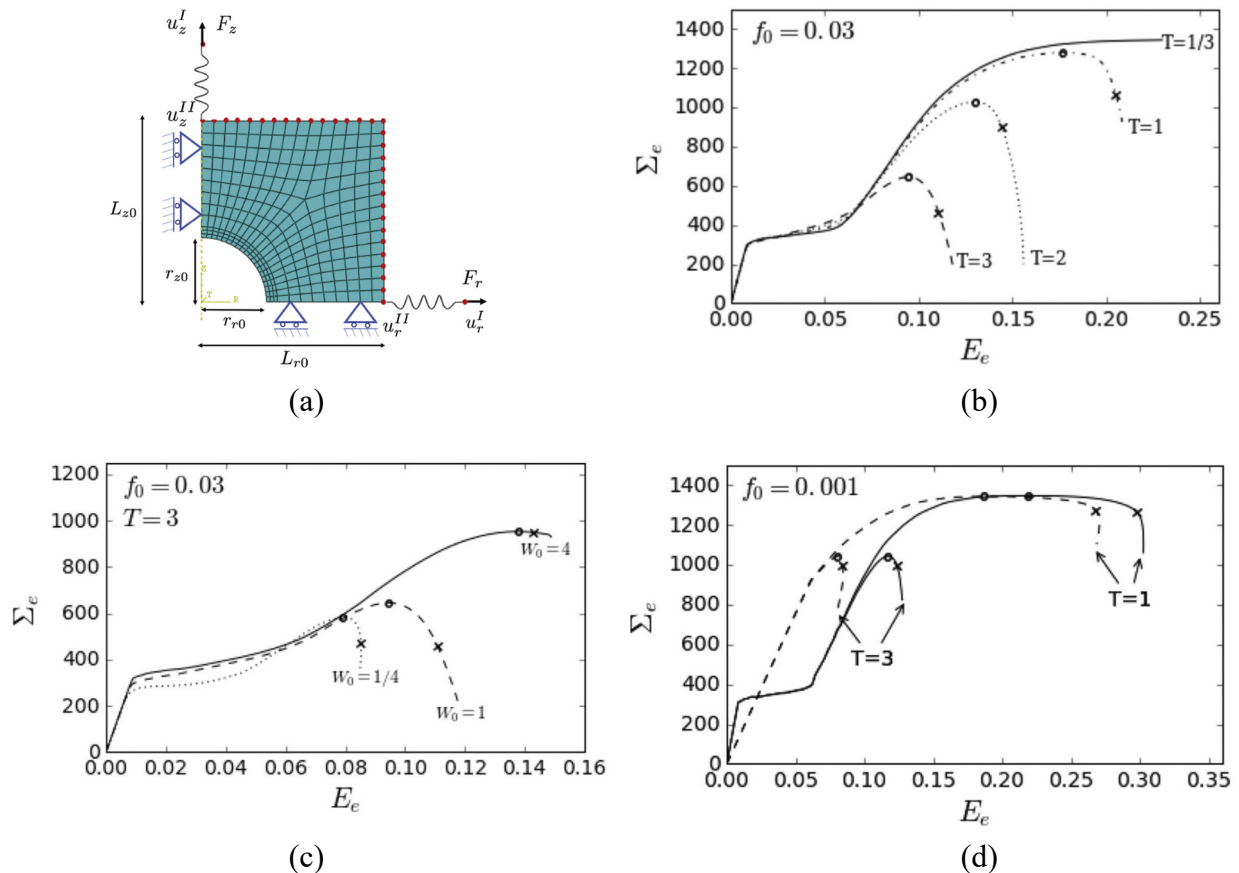


Figure 9. (a) Axisymmetric unit cell and boundary value problem, (b) numerical result showing effective stress vs effective strain result with various triaxialities, (c) numerical results with different void shape spherical, and (d) comparison of elastic-plastic material with phase transforming SMA [9].

2.2 Global Approach

2.2.1 Modeling and Experimental Validation of Stable Crack Growth in SMAs

Crack growth in SMAs under mode-I isothermal loading and in the presence of stress-induced martensitic phase transformation is simulated using finite element analysis. An SMA constitutive model is validated against experimental results for growing cracks in SMAs. The experimental data refers to a compact tension experiment on near-equiatomic NiTi. The constitutive model used is calibrated from uniaxial experiments on the same material system. The simulation is run in a three-dimensional compact tension specimen modeled in Abaqus finite element suite using the virtual crack closure technique, where crack is assumed to extend when the energy release rate reaches its critical value, and an experimentally determined fracture toughness value. The numerical results provide a quantitative description of the observed stable crack growth at different stages of loading in terms of load–displacement curve (Figure 10a), deformation distribution and size and shape of the stress-induced transformation zone close to the crack tip (Figure 10b).

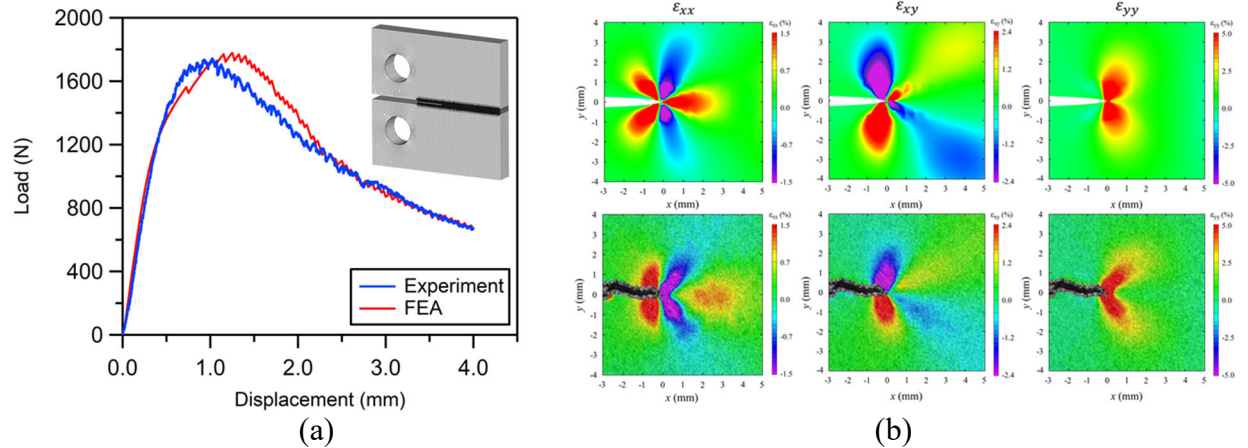


Figure 10. Comparison of experimental and numerical results obtained under mode-I isothermal loading of NiTi SMA: (a) load-displacement data and (b) in-plane strains near the crack-tip at the maximum attained load [10].

2.2.2 Experimental and Numerical Analysis of Actuation Crack Growth in SMAs

The experimental observation of stable crack growth in SMA compact tension specimens during temperature changes under different applied constant bias loads was reported for the first time. The intrinsic damage mechanisms that promote crack advance are those reported in literature for nominally isothermal overload fracture, *i.e.*, cleavage assisted by ductile void growth. Moreover, a numerical analysis was employed, and the resulting simulations were compared to the experimental data with the purpose of building confidence in the insight provided on the role of extrinsic mechanisms that further promote or impede crack advance. As shown in Figure 11, a good agreement between the experimental and numerical responses was reported during crack extension. It is concluded that phase transformation plays a dual role on the crack growth kinetics by promoting crack growth when occurring in a fan in front of the crack tip and providing the toughness enhancement that results in stable crack growth when left in the wake of the advancing crack. While the latter is well known as transformation-induced toughness enhancement, the former has just been recently observed experimentally and is characteristic of SMAs subjected to actuation loading conditions.

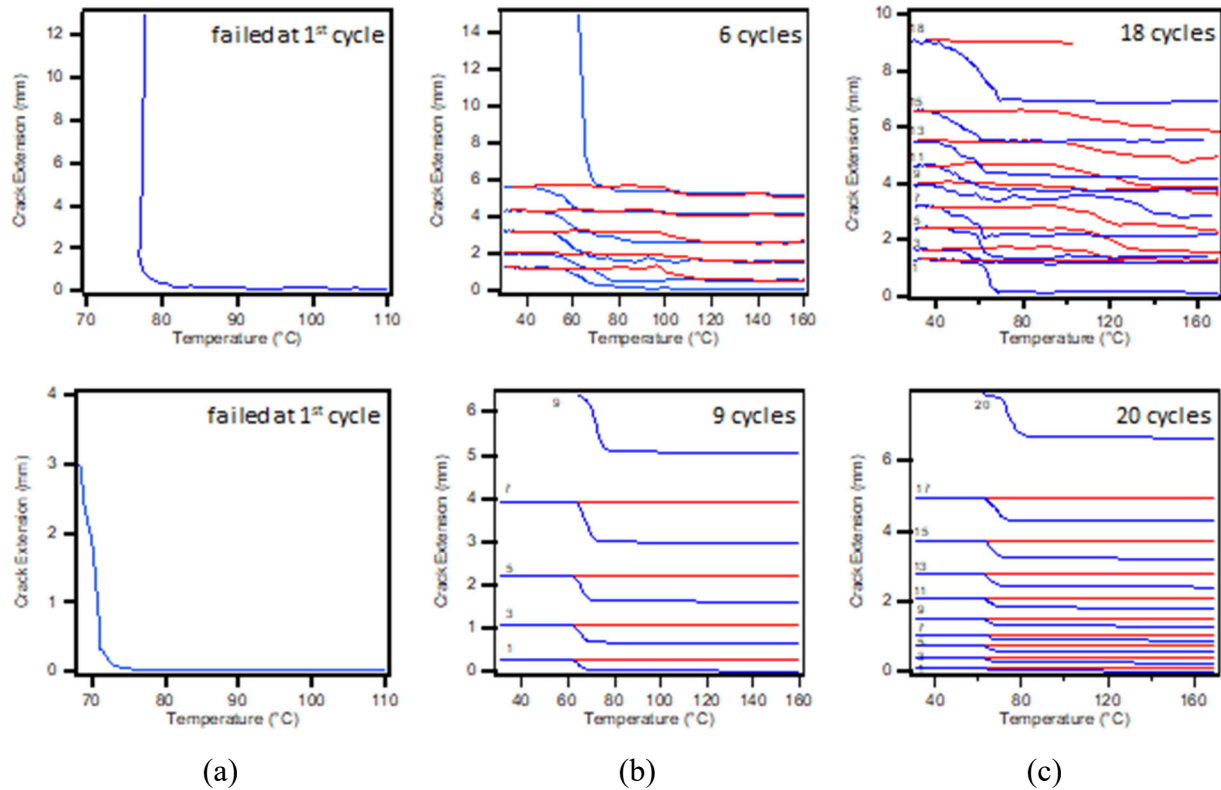


Figure 11. Comparison of experimental (top row) and numerical (bottom row) crack extension vs temperature results obtained under actuation loading of NiTi SMA under (a) 75%, (b) 40%, and (c) 25% of the maximum load obtained during isothermal loading at room temperature [11].

2.2.3 Phase Field Modeling of Actuation Crack Growth in SMAs

To numerically simulate the actuation crack growth in SMA, a phenomenological phase-field model is developed. The model's conformity with the Paris-type power law crack growth expression for a brittle response is reported. Plane strain numerical simulations are performed and the relation between non-dimensional crack growth (a^*) per cycle to non-dimensional cyclic ΔK_I^* , a driving force for the crack growth, normalized with critical driving force K_{Ic}^* is reported. The efficiency of the numerical implementation of the model is verified by crack growth simulations in complex geometries and loading conditions (Figure 12). Crack propagation is observed in a 2D plate with inclined crack by applying cyclic displacement on the upper surface keeping the lower surface fixed and in 2D and 3D notched plate with an oscillating displacement prescribed on the bearing surface of the upper pinhole keeping the lower pinhole fixed. Crack propagation simulation and Paris law for SMAs, based on cyclic ΔJ and accounting for reverse phase transformation, transformation induced plasticity, and latent heat effects, is envisioned.

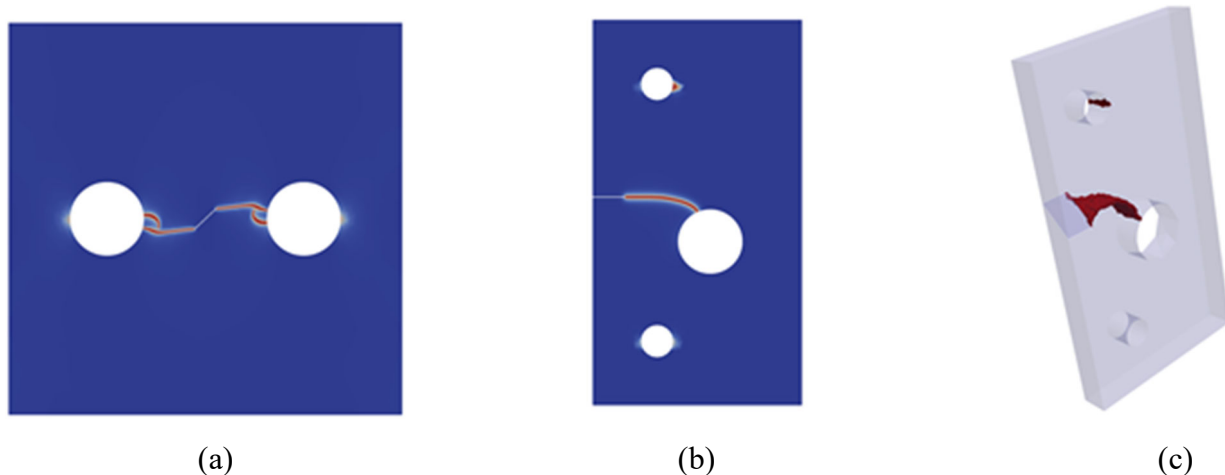


Figure 12. Crack propagation simulation in complex geometry and loading conditions using phase-field modeling. (a) 2D plate with inclined crack, (b) 2D notched plate, and (c) 3D notched plate [12].

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