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Wearless scratch on NiTi shape memory alloy due to phase transformational shakedown

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Cyclic microscratch tests were performed to examine the scratching behavior of NiTi shape memory alloy. It shows a superior wear resistance within the temperature range of 22–120 °C, but the corresponding physical mechanisms are different at low and high temperatures. We introduced the concept of phase transformational shakedown to interpret the wear-resistant behavior. At room temperature, a scratch groove may be caused by repeated scratching, but its depth stops increasing after a certain number of scratching cycles once the phase transformational shakedown state has been achieved. The groove will be self-healed upon heating as a result of the shape memory effect. At 60 and 120 °C, however, no evident scratch groove is observed under the same load due to the pseudoelastic effect and the increase in the phase transition stress with temperature. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2903106]

A challenge facing many and diverse technologies stems from the wear-associated failures.[1] A zero or very low wear rate may greatly enhance the performance and extend the service life of thin films, coatings, and micromachines in surface engineering and microelectromechanical systems. Single layer or multilayer coatings, with a very high hardness are often used to reduce the wear rate.[2,3] However, hard coatings are apt to brittle cracking due to high stress concentrations.

Owing to its remarkable shape memory effect and superelastic behavior, nickel-titanium (NiTi) alloy has found important applications in areas such as medical surgery and microelectromechanical systems.[4–8] However, the wear properties of shape memory alloys (SMAs) are still far from being understood due to the inherent complexity of their deformation behavior, which involves temperature-dependent reversible phase transition and plasticity of both austenite and martensite phases.[9,10]

In this letter, we report our experimental observation of wearless scratching of NiTi SMA under repeated sliding. Tension, microindentation, and microscratch tests were performed at different constant temperatures. It was found that the NiTi alloy exhibits a superior wear-resistance due to the diffusionless reversible transformation between the austenite (A) and the martensite (M) phases. Different physical mechanisms underlying the wear-resistant behavior of SMAs were revealed and discussed using the concept of phase transformational shakedown.[11]

NiTi polycrystalline sheets of 0.5 mm in thickness were purchased from Shape Memory Applications, Inc. (San Jose, CA, USA). The nominal alloy compositions are Ni 50.7 at. % and Ti 49.3 at. %. The grain sizes are about 100 nm, as observed by transmission electron microscope. With a differential scanning calorimeter (DSC 92, SETARAM, France), we measured the martensite start and finish temperatures of the alloy as 2.1 and −34.5 °C during cooling, and the austenite start and finish temperatures as 21.3 and 59.1 °C during heating, respectively. The material was first heated to 120 °C and then cooled down to 22 °C such that it was in the austenite phase at the initial stress-free state. Before tests, specimens were carefully polished to obtain a rms roughness smaller than 15 nm over a $5\times 5\ \mu\mathrm{m}^2$ region.

Uniaxial tensile tests of the NiTi sheets were first performed under different constant temperatures by using a universal testing machine (MTS SINTECH 10/D). Indentation and scratch tests were conducted on a triboindenter (Hysitron Inc., Minneapolis, USA) at three representative temperatures, 22, 60, to 120 °C. The probe was a spherical diamond indenter of 16 μm in radius and the indentation peak load was set as 10 mN. The wear tests were performed by cyclic scratching over a 20 μm distance on the NiTi surface under a constant downward force of 10 mN. After certain number of scratching cycles, the samples were heated to 120 °C for 2 h in order to examine whether the surface deformation beneath the indenter was caused by pure A → M phase transformation or plastic deformation, since the former would be completely recovered.

Figures 1(a) and 1(b) show the typical stress-strain curves and the indentation force-depth curves of the material at 22, 60, and 120 °C, respectively. They all show a significant temperature dependence. At 22 °C, the specimen first undergoes the A → R (rhombohedral) and then R → M phase transitions.[12] The R → M phase transition stress is about 160 MPa and the material shows a typical shape memory effect. A large residual indentation depth will be left after removing the indenter. At 60 °C, however, the phase transition stress increases to about 400 MPa, showing a remark-

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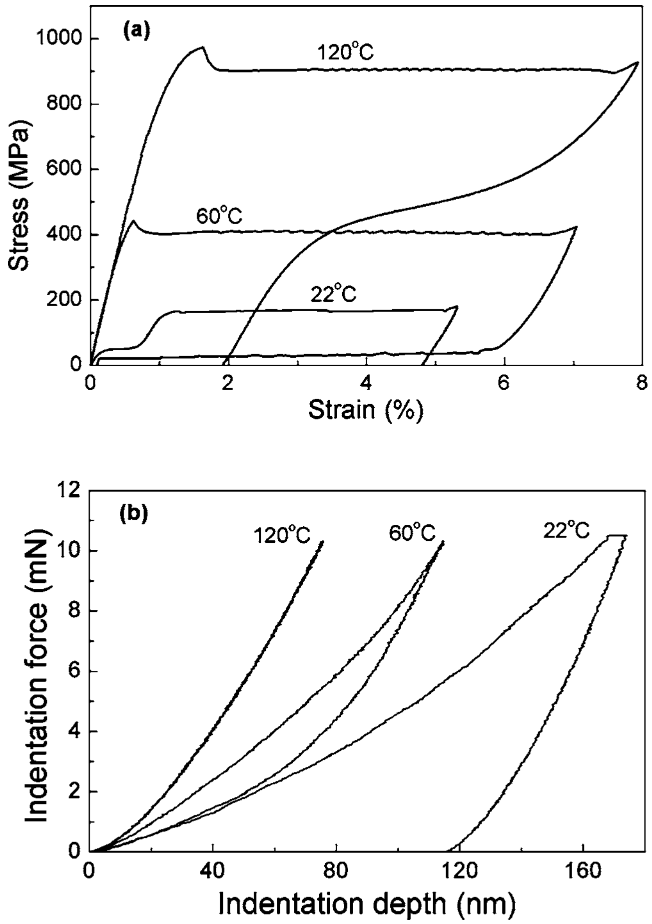

FIG. 1. (a) The tensile stress-strain curves and (b) the indentation force-depth curves of NiTi alloy at three representative temperatures.

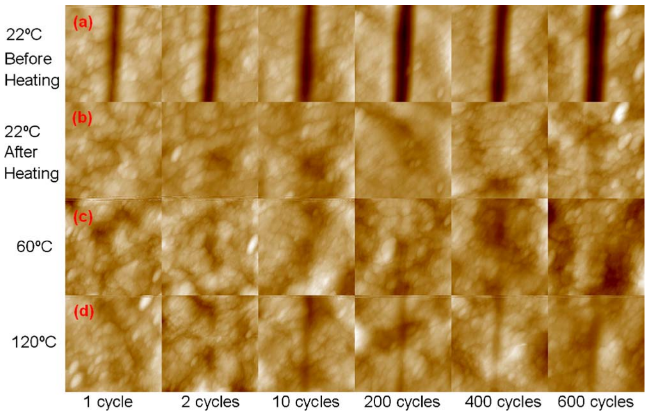

FIG. 2. (Color online) AFM images of surface morphologies on NiTi alloy after different scratching cycles (load: 10 mN, image size: 20×20 μm^2). (a) Scratching at 22 °C, (b) scratching at 22 °C and then heating to 120 °C for 2 h, (c) scratching at 60 °C, and (d) scratching at 120 °C.

able superelasticity. The indentation curve has a closed hysteresis loop with a zero residual indentation depth after complete unloading, indicating a complete reversible $A \rightarrow M/M \rightarrow A$ phase transformation in loading/unloading. At 120 °C, the phase transition stress increases up to about 900 MPa. According to Hertzian contact theory,[13] the applied force of 10 mN will not lead to any phase transition beneath the indenter, so the indentation loading curve is entirely identical to the unloading one.

In the scratch tests, the representative images around the scratch tracks are shown in Fig. 2 for 22 °C (before and after heating to 120 °C), 60, and 120 °C. In the case of 22 °C, the measured maximum depths of the scratch groove before and after heating are given in Fig. 3 as a function of the number of scratching cycles.

Some distinct features of the scratching behavior of NiTi can be observed from Figs. 2 and 3. At 22 °C, a scratch groove was formed due to the $A \rightarrow R \rightarrow M$ phase transformations. The groove depth increased quickly in the first three cycles, with the increments being 59, 20, and 14 nm, respectively. The groove then gradually approached a stable depth of about 120 nm. After heating to 120 °C for 2 h, the scratch groove almost completely recovered (Fig. 3), even the material had experienced a large number of scratching cycles (e.g., 600). Ni *et al.*[14] have observed such a self-healing effect due to $M \rightarrow A$ transformation by heating. Here, the self-healing of the scratch track indicates that the groove was caused only by $A \rightarrow R \rightarrow M$ phase transformation and that the wear rate under the spherical indenter was negligible at 22 °C for the given load of 10 mN. At 60 and 120 °C, on the other hand, no evident scratch groove appeared (Fig. 2), which means an almost wearless sliding under the same given load. The physical mechanisms in the three cases are different and will be discussed below.

Due to the complicated temperature-dependent phase transition properties of NiTi, exact analysis on the deformation mechanisms beneath the scratching indenter is difficult. Here, we introduce the concept of phase transformational shakedown to interpret the scratching behavior at different temperatures. One possible response of SMA under cyclic loading is that both forward ($A \rightarrow M$ or $A \rightarrow R \rightarrow M$) and reverse ($M \rightarrow A$) phase transformations take place in every loading-unloading cycle and the transformation strains tend to cancel out each other. Thus, the resultant transformation strains remain small though phase transformation processes keep evolving with the successive loading cycles. Such an alternating phase transformation response may also take place in a body if it has developed some phase transformation and/or plastic deformation in some initial loading cycles, causing a self-equilibrated residual stress field. The evolution of the residual stress field will make the stress distribution more and more uniform and eventually decrease the magnitude of phase transformation strains to a low level or even exclude phase transformation in the subsequent loading cycles. As a result, the phase transformation strains can evolve only in an alternating manner without any net deformation accumulation over a loading cycle. Such a behavior

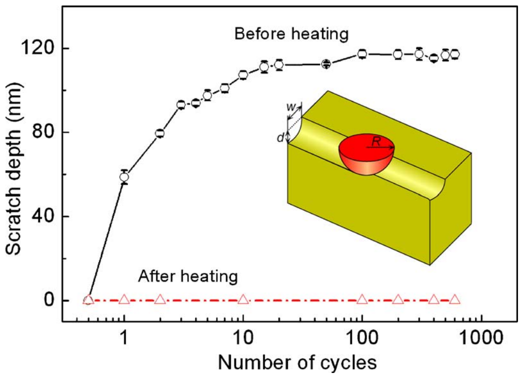

FIG. 3. (Color online) The scratch depths vs the scratching cycles at 22 °C and after heating to 120 °C, respectively.

with stabilized or alternating phase transformation strains is named as phase transformational shakedown.[11] This is an extension of the classical concept of elastic shakedown, which refers to a completely elastic state of an elastic-plastic body subjected to cyclic or variable loads and with stabilized plastic deformation.

According to the above definition, it is apparent that phase transformational shakedown occurs during the scratching at 22 °C. In the first scratching cycle, the elastic stress field has a high stress concentration according to the Hertzian contact theory since there is no preexisting residual stress under the initial planar sample surface. The scratching load will cause a large phase transformation strain (like a plastic strain) which creates a high residual stress field around the groove. In the subsequent scratching cycles, two mechanisms, the residual stress field, and the geometric effect of the scratch groove will reduce the stress concentration. With the increasing number of scratching cycles, the magnitude of the residual stress field becomes higher and the geometric effect becomes more significant. Consequently, the increment of the scratch groove depth in each scratching cycle decreases gradually. Eventually, a stable shakedown state will be achieved beneath the scratching indenter in the sense that after a complete loading cycle, the groove morphology, the residual stress field, and the phase transformation strain field all return to their initial values at the beginning of the cycle. Apparently, the measured scratch depth at 22 °C approached a stable value, as shown in Fig. 3.

For the NiTi SMA, phase transformation under the present loading does not cause evident degradation (such as microcracking) of the material, i.e., damage evolution with transformation process is very slow. It is reasonable to think that wear of SMAs is mainly caused by plastic deformation. For the case of scratching at 22 °C, both the residual stress field induced by $A \rightarrow R \rightarrow M$ transformation and the geometric effect of the groove postpone the occurrence of plastic yield and, therefore, no wear can be observed even after 600 scratching cycles. On one hand, the stabilized scratch groove indicates the achievement of the phase transformational shakedown state under the indenter. On the other hand, its complete and wearless recovery upon heating demonstrates that neither plastic deformation nor mass loss has happened, that is, the groove was caused only by $A \rightarrow R \rightarrow M$ phase transformation.

The scratching response at 60 °C is quite different. The martensite phase will reverse to the austenite once the scratching indenter has passed (unloading) since the alloy exhibits the pseudoelasticity at this temperature. Therefore, no evident scratch groove forms and the residual stress remains negligible in the successive scratching cycles. The stress and strain fields are repeated for all the loading cycles because of the absence of either residual strain development or geometric (groove) effect. The response beneath the indenter is alternating $A \rightarrow M$ and $M \rightarrow A$ transformations, giving a relatively large phase transformation strain but with zero net deformation in each cycle. This is a special case of phase transformational shakedown. It also demonstrates that the alternating phase transformation strain can still postpone the happening of plastic deformation and prevent the sample from wear.

At 120 °C, the plastic yielding of the austenite phase and the $A \rightarrow M$ phase transition take place simultaneously when the stress reaches about 900 MPa (Fig. 1). Due to friction, the stress state beneath the indenter is dominated by compression and shear and the maximum stress under scratching is higher than that under indentation so that $A \rightarrow M$ phase transition process can still occur. Upon unloading, the martensite will return to the austenite with a small plastic strain left in the material. A very shallow scratch groove of 10–15 nm is observed after 600 scratching cycles. It is yet unclear whether this shallow scratch groove results from pure plastic deformation without wear or from mass loss. Anyway, the wear rate would be very low even if there is mass loss. The high wear resistance of the alloy at 120 °C is attributed to (1) the high plastic and high phase transformation stresses and (2) the reversible phase transformation which reduces greatly both the magnitudes of plastic strain and wear rate.[15]

In summary, we have examined the wear resistance of NiTi alloy under cyclic scratching. The scratching behavior and deformation responses beneath the indenter are distinctly different at low (22 °C), mediate (60 °C), and high (120 °C) temperatures. At 22 °C, due to the shape memory effect and phase transformation shakedown, the material shows a superior resistance to abrasive wear. At 60 °C, no evident scratch groove forms due to its pseudoelasticity, and the large reversible transformation strain seems to be the unique reason to prevent the sample from wear. At 120 °C, however, the wear rate is still very low owing to the high plastic yielding stress and the further increased phase transformation stress. The theory of phase transformational shakedown provides a convenient tool to analyze the scratching behavior of SMAs. The wearless and near-wearless scratch of NiTi alloy is attributed to its unique properties of phase transformation.

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