WEAR BEHAVIOR OF NITI THIN FILM AT MICRO-SCALE

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This paper reports experimental study on the hardness and wear behavior of NiTi Thin Film Shape Memory Alloy (SMA) at micrometer scales. A triboindenter (Hysitron Inc., Minneapolis, USA) was used to conduct a series of indentations under various loads (the corresponding maximum indentation depth from 18.52 nm to 333.53 nm) and wear by scanning scratch method at temperatures from 25°C to 120°C. It was found that with increasing temperature, the hardness of NiTi thin film increased while its wear resistance decreased. The observed anomalous variation of wear resistance with hardness value is further analyzed by the interplay of phase transition and plasticity.

Keywords: NiTi; Thin Film; Micro-Wear; Hardness.

1. Introduction

Besides good chemical resistance and biocompatibility, NiTi shape memory alloys (SMAs) possess a variety of other desirable properties, such as a high power to weight (or force to volume) ratio, superelasticity, and high damping capacity. It also has the ability to recover large transformation strain upon heating and cooling.¹² Therefore, in recent years NiTi thin film has attracted strong interest in the field of micromachines;³ and various types of microactuator device have been reported in the literature, including the microgripper,⁴ micropump⁵ and microvalve.⁶

As most of the aforementioned micro-devices must be exposed to and interact with the environment, the understanding and control of tribological behaviors of the material become an important issue of concern. For instance, wear has been recognized as a life-limiting failure mechanism for micromachines⁷ and, consequently, the wear performance is crucial to the efficiency and longevity of micromachines. Although some fundamental studies on the wear behavior of NiTi SMA have been reported in recent years, they are mostly limited to bulk NiTi samples and testing on macro-scale.⁸⁻¹⁰ The purpose of this paper is to give a brief report of the hardness and wear behavior of NiTi thin film at
micrometer scales and try to understand the role of phase transition and plastic deformation in the indentation-scratch-wear process of the thin film.

2. Experiments

The NiTi thin film sample used was fabricated on a silicone substrate by a r.f. magnetron sputter-deposition method. The dimensions of the film are 10mm x 10mm with a thickness of 2µm and the initial state of the film is in austenite phase. The nominal alloy compositions are Ni-51.9 and Ti-48.1 in at%. By measuring and then extrapolating the strain-temperature curves during heating-cooling cycle at various constant stress levels, the phase transition temperatures of the film at the stress-free state are obtained (\(A_f^\circ \approx 28^\circ C, A_s^\circ \approx -5^\circ C, M_s^\circ \approx -60^\circ C, M_f^\circ \approx -100^\circ C\)) and the compositions and the phase transition temperatures are listed in Table 1.

Table 1. Composition (at%) and transition temperatures of the NiTi thin film.

<table>
<thead>
<tr>
<th>Ni (at%)</th>
<th>Ti (at%)</th>
<th>M_s</th>
<th>M_f</th>
<th>A_s</th>
<th>A_f</th>
<th>Room temperature phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>51.9%</td>
<td>48.1%</td>
<td>-100°C</td>
<td>-60°C</td>
<td>-50°C</td>
<td>28°C</td>
<td>Austenite</td>
</tr>
</tbody>
</table>

3. Results and Discussion

Figure 1 shows the surface morphologies (in-situ AFM images) at 25°C of a typical worn area of film at various loads and stages of scanning-scratch cycles.

Fig. 1. Images of wear marks of NiTi thin film at 25°C at different scanning-scratch cycles and loads.

At relatively small load levels of 20µN and 30µN, small ridges formed after 40 cycles of scanning-scratch. The size of the ridges gradually increased and eventually spread over
the worn area in the subsequent cycles of scanning-scratch. However, the average wear depth at this stage still remained very small (less than 10 nm, see Fig. 2), which indicates that the formation of ridges did not remove much material out of the area and mainly plastic flow of material occurred.

![Graph showing wear depth versus number of cycles under various loads at 25°C.](image)

Fig. 2. Wear depth versus number of scanning-scratch cycles under various loads at 25°C.

At 50 µN (Fig. 2) ridge formation was still the main deformation mode at the initial stage. However, the deformation mode changed from ridge formation to ploughing in subsequent cycles of scanning-scratch and a small amount of material piled up at the left edge of the worn area. The wear depth reached 29.4 nm after 200 scanning-scratch cycles.

When the load was further increased to 100 µN and 200 µN, the wear became much more serious (see the wear mark in Fig. 1 and the wear depth in Fig. 2). The images of worn areas after 40 cycles of scanning-scratch show a relatively flat topology. This implies that at the relatively high loading the initial deformation mode is cutting rather than ridge formation.

To investigate the effects of temperature on the wear performance of the film, the same procedures of wear as in Fig. 1 were repeated at 75°C and 120°C. The wear marks after 200 cycles of wear are summarized and are compared with that of 25°C in Fig. 3. It is seen that at small loads such as 20 µN and 30 µN the wear mode changes from ridge formation to ploughing (material piled up at the edge of the worn surface) by increasing the temperature from 25°C to 120°C. While under large loads (100 µN & 200 µN) the wear mode is almost insensitive to the temperature and remained the cutting mode. The wear is much more serious at high temperatures with a much larger amount of material being piled up. Fig. 4 shows the wear depths at different loads and temperatures after 200 cycles of wear. It is clearly seen that the wear performance of the NiTi thin film is strongly temperature dependent and its wear resistance decreased with temperature. For instance, at loads of 100µN and 200µN, the wear depth increased by 90.36% and 79.41% when the temperature is increased from 25°C to 120°C. This is very different from bulk stainless steel for which the wear depth only increased by 8.19% and 7.79% at the
corresponding loads when the temperature is increased from 25°C to 120°C (see the data for steel in Fig. 4).

![Fig. 3. Images of wear marks of NiTi thin film after 200 scanning-scratch cycles under different normal loads and temperatures.](image)

![Fig. 4. Wear depth (NiTi thin film and Stainless Steel) versus normal load after 200 scanning-scratch cycles at various loads.](image)

To understand the above temperature effect on the wear performance of NiTi thin film, hardness of the NiTi thin film was measured by the same Triboindenter at loads ranging from 100 µN to 8000 µN and at temperatures from 25°C to 120°C. The results are plotted in Fig. 5. Except the monotonic increase in hardness at small indentation loads (for loads below 1000 µN in Fig. 5(a)), the measured hardness is almost a constant at each given temperature and it monotonically increased with temperature (Fig. 5(b)). Combining the results of the hardness and wear tests (Fig. 5 and 4), we can deduce that
the wear resistance of NiTi thin film decreases anomalously with an increase in its hardness when the temperature is increased. Such anomalous temperature-dependent wear phenomenon could be explained based on the intrinsic stress-induced phase transition and its interplay with plastic deformation process of the NiTi film in the following.

3.1. Temperature effect on measured hardness of film

It is well known that for ordinary engineering metals, the hardness decreases with temperature because of the decreasing critical stress required for dislocation motion.\textsuperscript{11,12} For the temperature dependence of the hardness of NiTi thin film, we may directly link up this phenomenon with the unique temperature dependence of the stress-induced phase transition process involved in the indentation. Fig. 6(a) shows a typical stress-strain response of NiTi thin film\textsuperscript{13} and bulk sample\textsuperscript{14} in a tensile test.

![Stress-strain curve](image)

Fig. 6. (a) Typical stress-versus-strain curve of NiTi film and bulk sample from a tensile test.
It is expected that during the loading process, the NiTi film will experience four stages of deformation: (1) elastic deformation of austenite; (2) phase transition from austenite to martensite; (3) re-orientation plus elastic deformation of the martensite; and (4) finally plastic deformation of the martensite. Both the elastic and phase transition deformations will recover upon unloading. The residual deformation mainly stems from the plastic yield of martensite at the tip region in the case of indentation. Thus, the projected area $A_c$ (corresponds to the contact depth $h_c$) of NiTi film can be divided into two regions (Fig. 6(b) and 6(c)): the phase transition region $A_t$ and the martensite plastic yield region $A_m$.

It should be emphasized that the martensite plastic yield region $A_m$ is smaller than and located on top of the phase transition region $A_t$. Qian et al., by introducing the recovery ratio $\eta = A_t/A_m$, successfully developed a simple contact model to quantify the role of phase transition in the measured hardness value:

$$H = 4.5\eta + 4.5(1 - \eta)\frac{c_{ph}}{c_m}. \quad (1)$$

From Eq. (1), it is clear that the hardness of NiTi depends on three parameters: tensile transition stress $\sigma_t$ (the same for compression is assumed), tensile or compressive plastic yield stress $\sigma_m$ and the recovery ratio $\eta$. By scaling the load and displacement curves (using Berkovich tip) of the film by their respective maximum values at various temperatures (from 25°C to 120°C), it is revealed that the recovery ratio $\eta$ is independent of temperature as all the curves almost collapsed onto a single curve as shown in Fig. 7 (also see ref. 16).

Fig. 6. (b) A typical indentation curve on NiTi film measured by triboindenter. (c) Schematic showing indentation of NiTi film, where the projected area can be divided into three parts: $A_t$ due to phase transition, and $A_m$ due to austenite plastic yield.

Fig. 7. The non-dimensional indentation curves of NiTi thin film.

Fig. 8. The non-dimensional indentation curves of NiTi thin film and Stainless Steel.
Since the martensite plastic yield stress $\sigma_m$ is almost temperature independent,\textsuperscript{14,15} therefore, the increase in hardness with temperature (Fig. 5) is mainly due to the increase in the transition stress $\sigma_t$ with temperature. In short, different from the hardness of ordinary engineering metals which is mainly affected by the value of plastic yield strength, the hardness of NiTi thin film mainly varies with the material’s phase transition stress which is temperature dependent (see Fig. 5(b)).

3.2. Wear performance of NiTi film at room temperature

Figure 4 demonstrates that the wear performance of NiTi thin film is much better than that of stainless steel at 25°C. This phenomenon is anomalous as the hardness of stainless steel (4.5GPa) is higher than that of NiTi thin film (3GPa). According to the traditional wear formula,\textsuperscript{17} provided that the wear conditions are the same (i.e. with the same tip and upon the same load), the wear loss is inversely proportional to the hardness. However, it fails to explain the difference in wear performance between NiTi thin film and stainless steel because no phase transition process has been taken into account. As shown in Fig. 8, because of the unique phase transition process, the indentation load versus depth curve of NiTi thin film exhibits a much larger recoverable depth and, in turn, recoverable strain than that of stainless steel. Such additional reversible transition strain relax the stress concentration near the indenter tip and postpones the plastic flow by sharing an appreciable portion of the external load so as to minimize wear damage (the “transition shielding effect”).

Furthermore, the low elastic modulus of NiTi thin film is another key factor for the superior wear resistance of such an alloy (as the Young’s modulus is 62GPa in NiTi thin film compared with 200GPa in stainless steel). From a mechanical point of view, wear originated from plastic deformation and damage. For the present wear test, the Berkovich diamond tip can be regarded as rigid and simplified as sphere of radius $R$ at the microscale. The semi-infinite body represents the NiTi thin film or steel with Young’s modulus $E$ (see Fig. 9).

![Fig. 9. Illustration of a rigid spherical tip contacting (a) NiTi thin film and (b) steel.](image-url)
According to the Hertz theory of elastic contact, the maximum pressure is

$$p_o = 0.386 P \frac{1}{3} \left( \frac{E}{R} \right)^{\frac{2}{3}}.$$  \(2\)

Thus, for a given indenter tip radius \(R\), the maximum pressure has the following relation with the Young’s modulus, i.e.

$$\frac{p_o^{\text{NiTi}}}{p_o^{\text{Steel}}} = \left( \frac{E^{\text{NiTi}}}{E^{\text{Steel}}} \right)^{\frac{2}{3}} = 0.46 < 1.$$  \(3\)

By Eq. (3), under the same applied force and contact geometry, the maximum pressure in NiTi thin film is much lower than that of stainless steel. Based on the study of contact problem, the maximum contact pressure instead of the total contact force will directly determine the maximum stress to trigger plastic deformation. For example, the maximum shear stress is equal to 30\% of the maximum contact pressure in a plane strain contact problem between two cylindrical bodies. The maximum pressure, therefore, can be used as an indicator to evaluate the initiation of plastic deformation such as wear in materials. This lower maximum pressure in NiTi thin film delays the plastic deformation or reduces the plastic zone size, thus contributing to the increase in the wear resistance of this material.

### 3.3. Temperature effect on wear performance of NiTi film

Figure 10 implies that the wear performance of the NiTi thin film is strongly temperature dependent (as the hardness increases with temperature), i.e., its wear resistance decreases with temperature. This phenomenon can be attributed to two reasons: 1) The stress or load required to induce phase transition (which can accommodate the deformation) increases with temperature. Accordingly, under the same loading, less material will be transformed and therefore less transformation shielding effect. 2) The Young’s modulus of the NiTi thin film increased from 62GPa to 83GPa when the testing temperature changed from 25°C to 120°C. By
recalling (3), this means a 21% increase in $p_o$ and thus more plastic deformation at the same loading. This forms a strong contrast with stainless steel where the $p_o$ and thus the wear performance is much less sensitive to temperature.

4. Conclusion

In summary, the wear performance and hardness of NiTi thin film and its temperature dependence was investigated for the first time by the Triboindentator. It was found that the wear performance of the film is excellent and superior to stainless steel at room temperature. However, the wear resistance of the film deteriorates with increase in temperature even though its hardness had an obvious increase. Such anomalous wear-hardness relationship for the temperature range of the measurement was found and further rationalized by the role of phase transition and its interplay with plastic deformation during the wear process of the film.

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References