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Effects of Aging Treatments on the High Temperature Wear Behavior of 60Nitinol Alloy

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Abstract
This study was undertaken to investigate the effect of heat treatments on the high temperature wear behavior of 60Nitinol. The samples were hot-worked, aged at two temperatures of 400˚C and 700˚C for 1 h and then water quenched. The microstructure of the alloys was investigated by scanning electron microscope and X-ray diffraction. Sliding wear tests were performed at two temperatures of 25˚C and 200˚C using three types of 60Nitinol disks: hot-worked, aged at 400˚C and aged at 700˚C. All wear tests were performed at a speed of 0.3 m/s under a normal load of 60 N for a total sliding distance of 1000 m using WC-Co pins sliding against 60Nitinol disks. The worn surfaces and microstructure of subsurfaces were studied by scanning electron microscope. Compression and hardness tests were also performed to characterize the mechanical properties of the alloys. The highest fracture strain and lowest hardness was obtained for the sample aged at 700˚C which contained Ni₃Ti₂ precipitants. This sample also showed the maximum wear resistance at wear testing temperature of 200˚C. This was attributed to the formation of a more compact and stable tribological layer on the worn surface of the softer sample.

Keywords: Sliding Wear; Compression; 60Nitinol; Tribological Layer, Oxidative Wear.
INTRODUCTION

Nitinol is a nickel-titanium intermetallic compound invented at the Naval Ordnance Laboratory in the early 1960's (Julien (1-3)). The best-known Nitinol composition is type 55Nitinol, containing a nearly equal atomic mixture of nickel and titanium (Julien (2)). Type 60Nitinol is an intermetallic compound having 60% nickel and 40% titanium by weight, which is lesser known and described. It has many useful properties that have been unrecognized as of potential value. 60Nitinol alloy owns many unique properties such as non-magnetization, excellent corrosion resistance in most common corrosive media, excellent elastic behavior, relatively low elastic modulus with a high hardness and high yield strength even at elevated temperatures (Julien (2); Marinack, et al. (4)). This material is of interest to the aerospace community because it is corrosion-resistant, has moderate density, and can be easily hardened for wear resistance applications (Stanford (5)). It is naturally hard and can be heat treated to hardness on the order of 62 HRC or higher (Julien (1)). 60Nitinol has 26% lower density than steel for weight sensitive applications such as aircraft, satellite and spacecraft (Julien (2)). Hartl, et al. (6,7) have investigated the application of Ni60Ti alloy for chevrons which installed along the trailing edge of jet engine primary and secondary exhaust nozzles of a jet engine.

Recent studies showed that NiTi alloys exhibit high resistance to wear compared with many conventional wear-resistant materials at room temperature (Li (8); Liang, et al. (9); Arciniegas, et al. (10); Li and Liu (11); Qian, et al. (12); Farahat and Zhang (13); Hattori and Tainaka (14); Abedini, et al. (15)). Effect of various parameters including sliding distance, normal load, wear temperature, microstructure of the alloy and counterface material on the wear behavior of near equi-atomic NiTi alloys was investigated by Abedini, et al. (15-19). Formation of compact
tribological layer (mostly contained iron oxides) on the wear surface of the NiTi alloy against bearing steel resulted in a decrease in the wear rate with increasing wear temperature, sliding distance and normal load.

Type 60Nitinol was recently used to fabricate ball bearings (Julien (2)) and cutting instruments (Julien (2)), applications in which the wear performance of the material plays a critical role. This material is also of interest within the aerospace community for use as a bearing material (Stanford (20)). 60Nitinol has found new application as the race in rolling-element which may operate at temperatures higher than 200°C (Miyoshi, et al. (21)). Oil and grease would decompose at temperatures above 200°C, but it was claimed that Nitinol with oxidized surface could extend operating temperature while maintaining low friction and high wear resistance.

The results showed better tribological behavior of oxidized samples against sapphire balls as compared with NiTi base alloy at two wear testing temperatures of 23°C and 500°C. Zeng et al. (22-23) have studied lubrication properties of 60Nitinol alloy. They indicated super-low friction of the alloy under lubricated conditions.

To our knowledge there was no study on the wear behavior of 60Nitinol. In the present study the wear behavior of three heat-treated 60Nitinol was investigated. The main aim was to characterize the effect of heat treatment on the wear resistance of the alloy.

**EXPERIMENTAL PROCEDURE**

60Nitinol (Ti-60 wt.% Ni) alloy was prepared by vacuum induction melting of titanium (99.99%) and nickel (99.98%) metals. The alloy was homogenized at a temperature of 1080°C for 8 h and was then hot-worked at 1080°C (i.e., a reduction of about 30% in length). Disks, 5 mm in thickness, were wire-cut from the hot-worked bar. The disks were then solution annealed
at a temperature of at 1080°C for 6 h and quenched in water. Finally, the disks were aged at 400°C (A400) and 700°C (A700) for 60 minutes, followed by water quenching. All heat treatments were carried out under an argon atmosphere. The heat treatments of different specimens are listed in Table 1. The microstructures of the alloys were characterized by scanning electron microscope (SEM). The samples for SEM observation were prepared by standard metallographic procedures and then etched in a solution containing 3.2% HF, 14.6% HNO₃ and 82.2% H₂O (Robertson, et al. (24)). The phases existing in the microstructure of the alloys were studied by means of X-ray diffraction (XRD) using Cu-Kα (40 kV, 30 mA) radiation. The hardness was measured on polished surface of the specimens using Vickers hardness (HV) tester with an applied load of 30 kg. The hardness value for each specimen was taken from the arithmetical mean of at least three measurements. The wear tests were performed on a high temperature pin-on-disk tribometer using NiTi disks with a diameter of 35 mm. Tungsten carbide (WC-Co) pins, 15 mm in length and 5 mm in diameter, with a spherical tip radius of 3 mm were used as slider. The hardness of the pins was measured to be about 1150 HV. The tests were performed under a normal load of 60 N at a sliding speed of 0.3 m/s for sliding distance of 1000 m at two wear testing temperatures of 25°C and 200°C. The weight loss of the specimens was measured using a precision balance with accuracy of ±0.1 mg. The hardness of the worn surface was also measured using a microhardness tester. Finally, the worn surface and subsurface of the samples were studied by SEM.

The compression tests were also performed to determine the stress–strain relationship and relevant mechanical properties of 60Nitinol specimens. The compression tests were carried out using an MTS universal testing machine at strain rates in the range of 0.04–0.05 s⁻¹ at two
temperatures of 25°C and 200°C. The diameter of cylindrical compression specimens was 4 mm with a length of 6 mm, i.e., a length/diameter ratio of 1.5 (ASTM E9 (25)).

RESULTS AND DISCUSSION

Figure 1 represents the microstructural features of the hot worked (i.e., HW) and the aged (A400 and A700) 60Nitinol samples. The XRD patterns of different samples are shown in Fig. 2. Figure 1a shows that the HW specimen consisted of many observable blocky-shaped precipitates with size up to 1 μm. The XRD pattern of this specimen in Fig. 2a revealed that the precipitates are Ni₄Ti₃. The formation of these precipitations could be due to the high supersaturation of 60Nitinol at room temperature that caused high driving force of nickel atoms to diffuse from matrix (Porter and Easterling (26)). The A400 specimen (i.e., the sample aged at 400°C) contained no observable precipitation in Fig. 1b. However, XRD studies shown in Fig. 2b, demonstrated that the Ni₄Ti₃ precipitates were formed on the microstructure of this specimen. These precipitations were probably very fine to be observable in SEM micrographs even at high magnifications. However, very coarse precipitates with a plate-like morphology can be observed in the microstructure of the A700 alloy in Fig. 1c, which could be related to the Ni₃Ti₂ precipitation according to the XRD results in Fig. 2c.

Table 2 represents the hardness of the 60Nitinol specimens. The A700 and A400 samples have the minimum and maximum hardness, respectively. Higher hardness of more than 35% in the A400 than the A700 alloy could be attributed to the formation of very fine and coherent Ni₄Ti₃ precipitates in sample aged at 400°C as confirmed by the XRD patterns in Fig. 2. Lower hardness of A700 sample could be attributed to the formation of large, plate-like and probably non-coherent Ni₃Ti₂ precipitates (Figs. 1c and 2c). The large size of Ni₄Ti₃ precipitates in the
microstructure of HW specimen after hot working (see Figs. 1a and 2a) could be the main reason for the lower hardness of this specimen as compared to A400 with very fine Ni₄Ti₃ precipitates. The true stress-strain curves of the alloys at two testing temperatures of 25°C and 200°C obtained from the compression tests are shown in Fig. 3. The figure shows that the yield strength of the alloys was in consistent with the hardness shown in Table 2, i.e., A400 and A700 samples showed the highest and lowest yield strength, respectively. This could be related to the size, morphology and type of precipitates in the microstructure of the alloys as discussed earlier for the hardness. A remarkable point in Fig. 3 was the large difference in the fracture strain or plastic deformation of the alloys with different heat treatments. The A400 alloy showed almost no plastic deformation after reaching the yield stress and behaved like a brittle material. The presence of the coherent and fine Ni₄Ti₃ precipitates could increase the compression strength and lower the fracture strain (Delgadillo, et al. (27)) in the sample aged at 400°C for 1 h, i.e., A400. At both temperatures of 25°C and 200°C, Fig. 3 showed the highest plastic deformation and lowest yield strength for A700 specimen, which also showed the lowest hardness in Table 2. Figure 4 shows the weight loss of specimens at two wear temperatures of 25°C and 200°C after sliding distance of 1000 m. The room temperature hardness of the samples is also shown in Fig. 4. The specimens showed almost a same weight loss at room temperature, although the difference in their hardness was noticeable. According to the Archard’s wear equation, the wear of materials has an inverse relationship with their hardness (Hutchings (28)). However, Fig. 4 shows that as the hardness increased by about 35% from A700 to A400, the corresponding room temperature wear decreased by only about 5%. Figure 4 also shows that HW specimen with a
lower hardness of about 14% compared with A400 exhibited a lower wear, which was in contrast with the Archard’s wear equation (Hutchings (28)).

The worn surfaces of HW, A400 and A700 samples tested at a temperature of 25°C are shown in Fig. 5. A less plastically deformed surface could be observed on the worn surface of A400 (Fig. 5b) compared with HW (Fig. 5a) and A700 (Fig. 5c). This could be due to the lower plastic strain of A400 as shown in Fig. 3. In fact, the higher plastic deformation of HW and A700 could lead to higher work hardening and, therefore, a higher hardness of the wear surfaces. The hardness of the worn surfaces of the alloys (i.e., HW, A400 and A700) was between 600-650 HV. This indicates that despite the lower bulk hardness of HW and A700 alloys compared with A400, the hardness of the worn surfaces of the alloys reached a close value. This could lead to improvement of the wear resistance of HW and A700 samples and result in a lower wear than expected.

Figure 4 shows that at the wear testing temperature of 200°C, the wear of the alloys increased as their room temperature hardness increased, i.e., A400 sample with the highest hardness of 650 HV showed the highest wear at temperature of 200°C. Comparing the hardness and the high temperature wear behavior of samples in Fig. 4 suggested that the wear behavior of the specimens at 200°C could not be explained by their room temperature hardness. SEM micrographs of the worn surfaces and cross sections of the 60Nitinol specimens tested at 200°C are shown in Figs. 6 and 7, respectively. Figure 6 shows that some tribolayers (dark regions) were formed on the worn surfaces of samples. The Energy Dispersive X-ray Spectroscopy (EDS) analysis of a typical tribolayer on the worn surface of A400 specimen is also shown in Fig. 6b. The results revealed that the patches formed on the worn surface of 60Nitinol contained
a certain amount of nickel, titanium and oxygen probably indicating formation of mixed oxides of nickel and titanium on the worn surface of the alloys at wear testing temperature of 200°C. Figure 6 shows that there was a lower coverage of tribolayers on the worn surface of A400 (Fig. 6b) than A700 and HW alloys. Figure 7b shows a region of the cross section of the wear surface of A400 where no tribolayer was formed which revealed some near surface cracks. These near-surface cracks could be due to the very low ductility and plastic deformation of A400 sample as confirmed by stress-strain curves in Fig. 3. Figure 7 shows formation of compact tribolayers on the wear surfaces of HW and A700 alloys. Figure 7a shows that the tribolayers formed on the HW worn surface contained fatigue cracks. A heavily plastically deformed substrate supporting a compact tribolayer could be observed for A700 in Fig. 7c. Comparing the cross sections of A700 and HW in Fig. 7 could suggest that a more durable tribolayer was formed on the wear surface of A700 alloy. The analysis of the tribolayers on the worn surfaces showed that they were consisted of mixed oxide of nickel and titanium with debris of WC from the mating pins. The mixing of these oxides and the debris formed a mechanically mixed oxide layer on the worn surfaces. Higher plastic deformation of A700 alloy could lead to a higher ability for formation of mechanically mixed oxide layers on the worn surface. Moreover, the higher work hardening of this alloy could lead to an increased hardness of the substrate, which could better support the tribolayers formed on the worn surface.

The variations of coefficient of friction with sliding distance for HW, A400 and A700 samples are presented in Fig. 8. The figure shows that the instability (i.e., the change in amplitude of friction) in the coefficient of friction of the samples was decreased as the wear temperature
increased. This could be related to the formation of tribological layers on the rubbing surfaces as shown in Fig. 7 and, therefore, reducing adhesion between them.

The compact tribolayers formed on the worn surface of HW and A700 in Fig. 7 at the wear temperature of 200°C could act as a solid lubricant and reduce the contact between mating surfaces (Hutchings (28)). However, the strength that the substrate achieved during sliding wear could control the wear and the behavior of tribolayer under the working conditions. Therefore, it seems that, although the aging treatment of 60Nitinol alloy at 700°C showed the lowest hardness, however, a more stable and compact tribolayer was formed during sliding at the wear temperature of 200°C probably due to the higher work hardening of A700 alloy.

Figure 4 revealed that as the wear temperature was increased from 25°C to 200°C, A700 showed the least increase in wear (about 12%) compared with 33% and 63% increase in the wear of HW and A400 alloys, respectively. This could also be attributed to the formation of a more compact tribolayer on the worn surface of the sample at higher wear testing temperature of 200°C. It seems that the wear of 60Nitinol alloy, especially at elevated temperatures, was affected mainly by the amount of plastic deformation and work hardening of the substrates and, therefore, their ability to form and support the tribolayers during sliding.

CONCLUSIONS

- The microstructure analysis of different samples revealed formation of coarse Ni₄Ti₃ in hot worked sample, very fine Ni₄Ti₃ in sample aged at 400°C and coarse plate-like Ni₃Ti₂ in sample aged at 700°C.
- The results showed that aging at temperatures of 400°C and 700°C caused the highest and lowest hardness, respectively. Aging at 700°C also resulted in the maximum plastic deformation.

- At wear testing temperature of 25°C, the wear results showed that the weight loss of specimens with different hardness was almost in a same range. This was attributed to the higher plastic deformation and work hardening of the softer samples.

- At wear testing temperature of 200°C, the wear of samples was increased as their room temperature hardness increased. Formation of more compact tribolayers on the worn surface of the softer alloy due to the higher plastic deformation was the major reason for this behavior. It was concluded that the plastic deformation is the major parameter affecting the wear behavior of the 60Nitinol alloy, especially at elevated temperatures.
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Fig. 3. The true stress-strain curves of 60Nitinol alloys at two temperatures of: (a) 25°C and (b) 200°C.

Fig. 4. Weight loss of HW, A400 and A700 specimens at 25°C and 200°C under a normal load of 60 N at sliding speed of 0.3 m/s for sliding distance of 1000 m. The room temperature hardness values of specimens are also shown.

Fig. 5. SEM observations of the worn surfaces of: (a) HW, (b) A400, and (c) A700 at wear temperature of 25°C.

Fig. 6. SEM observations of worn surfaces of: (a) HW, (b) A400, and (c) A700 at wear temperature of 200°C.

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Fig. 8. Variations of coefficient of friction with sliding distance for 60Nitinol alloys at two wear testing temperatures of 25°C and 200°C.
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Table 1. Heat treatments of the specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat Treatments</th>
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<td>HW</td>
<td>homogenized at 1080°C for 8 h + hot-worked at 1080°C</td>
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<tr>
<td>A400</td>
<td>homogenized at 1080°C for 8 h + hot-worked at 1080°C + annealed at 1080°C for 6 h + quenched in water + aged at 400°C for 1 h + quenched in water</td>
</tr>
<tr>
<td>A700</td>
<td>homogenized at 1080°C for 8 h + hot-worked at 1080°C + annealed at 1080°C for 6 h + quenched in water + aged at 700°C for 1 h + quenched in water</td>
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Table 2. Hardness of 60Nitinol specimens

<table>
<thead>
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<th>Specimen</th>
<th>HW</th>
<th>A400</th>
<th>A700</th>
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<tr>
<td>Hardness (HV)</td>
<td>560</td>
<td>650</td>
<td>480</td>
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