

New wear-resistant material: Nano-TiN/TiC/TiNi composite

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Near-equiatomic TiNi alloy has been found to exhibit high resistance to wear, especially to erosion. The high wear resistance of the alloy may largely benefit from its pseudoelasticity. Recent studies demonstrate that the wear resistance of TiNi alloy can be considerably enhanced when hard particles such as TiC were added as a reinforcing phase. It was expected that the wear resistance of such a composite could be further improved if the TiNi matrix can be strengthened with retained pseudoelasticity. Attempt was made to develop such a tribo composite, using nano-TiN powder to strengthen the matrix of the TiC/TiNi composite. The composite was made using a vacuum sintering process. Sliding wear behavior of this material was evaluated. It was demonstrated that the *nano-TiN/TiC/TiNi* composite exhibited excellent wear resistance, superior to those of the TiC/TiNi composite and *WC/NiCrBSi* hardfacing overlay. In order to understand the role of the nano-TiN powder, localized mechanical behavior and micro-scale wear of the TiNi matrix with and without nano-TiN powder were investigated using a triboscope. Worn surfaces were examined using SEM to better understand the wear mechanism and to find out clues for further development. © 2001 Kluwer Academic Publishers

1. Introduction

Recent studies have demonstrated that near-equiatomic TiNi alloy exhibits high wear resistance and could be an excellent candidate for various tribological applications [1–11]. A number of researchers have demonstrated superior wear behavior of TiNi alloy in different wear conditions, compared to many conventional materials, such as Co45 alloy, 38CrMoAlA, cast martensitic (CA6NM), austenitic (304L) and duplex (FER-255) stainless steel [2, 8]. It is known that the wear resistance of conventional tribo materials is mainly dependent on their mechanical properties such as hardness, toughness, and work hardening [12]. Hardness is of importance to the abrasion resistance, while the toughness and work hardening are more responsible for the wear resistance under impact or high stresses. However, it appears that these mechanical properties are not only factors responsible for high wear resistance of TiNi alloy, which could greatly benefit from the pseudoelasticity of the alloy [2–5, 8, 11]. The pseudoelasticity of TiNi alloy results from a thermoelastic martensitic transformation. Under external force, B2 phase transforms to a martensitic phase. Such a phase transformation is reversible, accompanied with recoverable strain in the range of 5%–8% [13, 14]. This high magnitude strain may help to accommodate large deformation and absorb impact energy with considerably less damage to the material during wear.

It is also noticed that the high wear resistance of TiNi alloy is not only attributable to its pseudoelasticity but also to other properties such as the strain-hardening capability [1, 15]. More recently, attempts were made to develop a new wear-resistant composite using pseudoelastic TiNi alloy as the matrix and hard TiC particles as the reinforcing phase [16]. It was demonstrated that TiC particles significantly enhanced the wear resistance of TiNi alloy. The wear resistance of *60%TiC/TiNi* composite was about one order of magnitude higher than that of the TiNi alloy, and comparable to that of *WC/NiCrBSi*, a hardfacing overlay widely used in the mining and oil and sand industries. It was expected that the wear resistance of the TiNi-matrix composite could be further improved if the TiNi matrix is strengthened without losing its pseudoelasticity.

In this work, an attempt was made to improve TiC/TiNi composite by modifying its TiNi matrix with TiN nano-particles. The TiNi alloy was employed as the matrix due to its good wear resistance and its pseudoelasticity. TiN nano-powder was added to the matrix to strengthen the TiNi matrix without changing its composition that influences the martensitic transformation. The amount of the nano-TiN powder was small so that the matrix may still keep good pseudoelasticity. Such a matrix may possess improved wear resistance and retained pseudoelasticity to accommodate large-scale

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deformation and bind hard TiC particles effectively, which play the main role in withstanding external load.

Wear behavior of the TiNi-matrix composite modified with nano-TiN powder was evaluated using a pin-on-disc wear tester and compared to TiC/TiNi composite and the WC/NiCrBSi hardfacing overlay. Worn surfaces were examined using a scanning electron microscope. In order to understand the role of nano-TiN powder, mechanical properties and local wear resistance of the composites with and without nano-TiN addition were investigated using a triboscope—a combination of nano-mechanical probe and atomic force microscope. The mechanism responsible for the improved wear resistance of the new tribo composite is discussed.

2. Experiment procedure

Nano-TiN/TiC/TiNi and *TiC/TiNi* composites were made using a vacuum sintering process. Pre-alloyed TiNi alloy powder having its particle size ≤ 180 microns, nano-TiN powder having its particle size < 50 nm, and TiC powder with its size ≤ 45 microns were mixed in air using a ceramic ball mill for one hour. The mixed powder was then pressed to make pin specimens under a pressure of 787 MPa for 30 seconds. The pin specimens were 12 mm long with a diameter of 6 mm. The specimens were then sintered in vacuum (5.4×10^{-4} torr) at 1350°C for 5 hours. The nominal composition of the matrix was Ti-50.6at%Ni. Specimens with different fractions of TiC particles, nano-TiN powder and the TiNi matrix were prepared. Microstructure of the sintered *nano-TiN/TiC/TiNi* specimens were examined using x-ray diffraction and SEM.

Wear behavior of the *nano-TiN/TiC/TiNi* specimens was evaluated under different load levels and at a sliding speed of 60 m/min for 3000 m, using a pin-on-disc tribometer with a water cooling system. Wear resistance of the *TiC/TiNi* composite and that of the WC/NiCrBSi hardfacing overlay were also evaluated under the same condition for comparison. Worn surfaces of the materials were examined using SEM.

In order to investigate the wear behavior of the TiNi matrix of TiC/TiNi composite samples with and without nano-TiN particles, the local wear behavior of the TiNi matrix was investigated on a microscopic level, using a triboscope. The mechanical probe of the triboscope was a pyramidal diamond tip (tip radius: 70 nm). The micro-wear test was performed by scratching the matrix within an area of 400 nm \times 400 nm (scratched line by line within the area) at a speed of 4 μ m/s and under a load of 50 μ N. The wear resistance of a specimen was evaluated by measuring its volume loss in the scratched area. The main purpose of this test is to investigate the correlation between the wear resistance of the matrix and the integral wear resistance of the corresponding composite.

Indentation behavior of the TiNi matrix was investigated using the triboscope. This test was performed to investigate changes in pseudoelasticity and hardness of the TiNi matrix when nano-TiNi powder was added, towards a better understanding of the effect of nano-TiN addition on the wear resistance of the composite.

3. Experiment results and discussion

3.1. Microstructure

The sintered 5vol% nano-TiN/55vol%TiC/TiNi composite was examined using x-ray diffraction. Fig. 1 illustrates a X-ray diffraction pattern (Co-K α_1), which demonstrates that there was no new phase in the composite after sintering. Since the amount of nano-TiN powder was small, no TiN peaks were detected. It is also possible that small TiN peaks may not be distinguished, since they could be overlapped with the TiC peaks due to similar structures of TiC and TiN. Microstructure of the sintered composite was examined using a Hitachi S2700 SEM. A metallograph of 5% nano-TiN/55%TiC/TiNi specimen is presented in Fig. 2. One may see that the TiC particles were distributed homogeneously throughout the matrix. The composition analysis shows that no chemical reaction occurred between the TiNi matrix, TiC and nano-TiN particles.

3.2. Sliding wear

Volume losses of 1% nano-TiN/59%TiC/TiNi, 3% nano-TiN/57%TiC/TiNi, 5% nano-TiN/55%TiC/TiNi, 7% nano-TiN/53%TiC/TiNi and 9% nano-TiN/51% TiC/TiNi specimens were evaluated using a pin-on-disc wear tester under several loads, respectively. Results of the wear test are illustrated in Fig. 3, which illustrates that the volume loss of 5%-nano/55%TiC/TiNi was the least. It turned out that the optimal fraction of nano-TiN particles was 5%. Therefore, the following research was focused on the 5% nano-TiN/55%TiC/TiNi specimens.

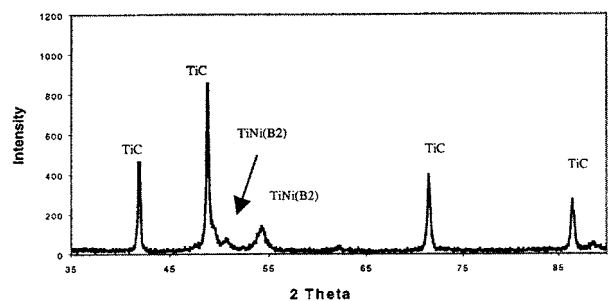


Figure 1 X-Ray pattern of 5% nano-TiN/55%TiC/TiNi composite.

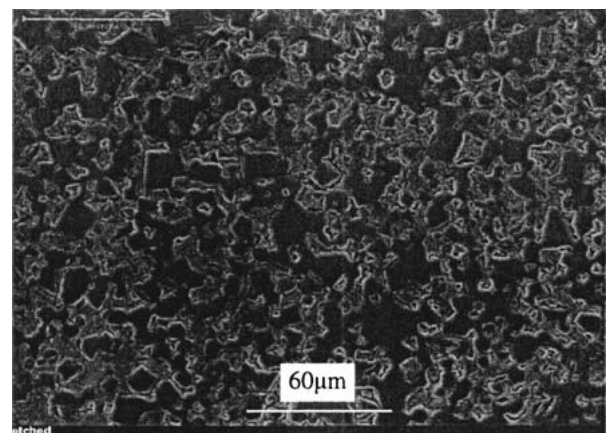


Figure 2 A SEM micrograph of 5% Nano-TiN/55%TiC/TiNi composite. The sample was polished and etched to make the interphase boundary visible.

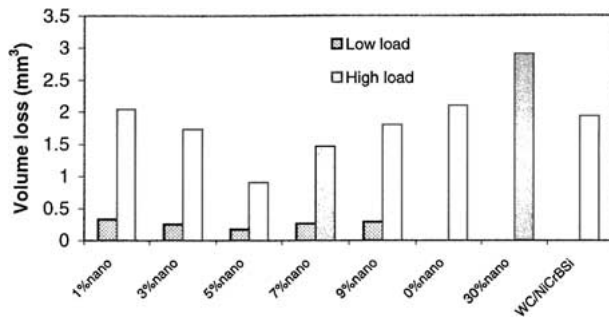


Figure 3 Volume losses of TiC/TiNi composites with different fractions of nano-TiN particles worn under low and high loads [low load: 0.05 kN, high load: 0.3 kN]. Volume loss of the WC/NiCrBSi hardfacing overlay worn under the high load is also given.

In order to obtain more information on the effect of nano-TiN particles on the wear resistance, 30% nano-TiN/60% TiC/TiNi composite was also investigated for comparison. It was observed that this composite was rather brittle because there was not enough TiNi matrix, which acted as a binder to retain the reinforcing TiC particles. Therefore, in order to compare 5% nano-TiN/55% TiC/TiNi to those containing a higher-fraction of nano-TiN particles, we kept the fraction of TiNi matrix at 40% to make it effective as a binder. 30% nano-TiN/30% TiC/TiNi composite rather than 30% nano-TiN/60% TiC/TiNi was then studied. Volume losses of sintered 30% nano-TiN/30% TiC/TiNi, 60% TiC/TiNi, and WC/NiCrBSi hardfacing overlay under a high normal load of 0.3 kN were measured for the purpose of comparison. Results of the wear test are also presented in Fig. 3. It was demonstrated that the 5% nano-TiN/55% TiC/TiNi composite was remarkably superior to the 60% TiC/TiNi composite with no nano-TiN particles. However, 30% nano-TiN/30% TiC/TiNi composite was worse than the 60% TiC/TiNi composite. The result indicates that the nano-TiN powder played a beneficial role in improving the wear resistance of the TiC/TiNi composite, but too much nano-TiN powder was detrimental. Compared to the WC/NiCrBSi hardfacing overlay which is widely used in the mining and oil sands industries, 5% nano-TiN/55% TiC/TiNi composite showed markedly higher wear-resistance. The wear resistance of TiC/TiNi composite benefited from its pseudoelastic matrix, which however becomes less functional with an increase in load because of the promotion of plastic deformation. When a small amount of nano-TiN powder was added, it may strengthen the matrix without losing substantial pseudoelasticity, thus leading to the wear resistance higher than that of the TiC/TiNi composite.

Surfaces of the tested materials worn respectively under a low load (0.05 kN) and a higher load (0.3 kN) were examined using a scanning electron microscope. Fig. 4 illustrates micrographs of the worn surfaces. At the low load, the worn surfaces of 60% TiC/NiTi, 5% nano-TiN/55% TiC/NiTi and 30% nano-TiN/30% TiC/NiTi all are smooth and no significant difference was observed between these materials. Under the high load, however, the situation changed. Worn surfaces of 60% TiC/NiTi and 30% nano-TiN/30% TiC/NiTi composites show severe wear track and it appeared that the reinforcing TiC particles were torn off from the matrix. While the

worn surface of the 5% nano-TiN/55% TiC/NiTi was relatively smooth and no tear-off of TiC particles was observed. In this case, TiC particles could be worn away gradually during the wear process. The worn surfaces of the tested materials under the low and higher loads are consistent with the sliding wear test.

3.3. Mechanical properties

3.3.1. Pseudoelasticity

Effect of nano-TiN particles on the pseudoelasticity of TiNi-matrix of the composites under study was investigated. The pseudoelasticity results from a thermoelastic martensitic transformation, which is evaluated usually using tensile test. The response of the martensitic transformation to stress, however, differs under different loading conditions [17, 18]. Therefore, the tensile test may not precisely reflect the pseudoelastic response of TiNi alloy to external force during wear. In the case of wear, the stress state is complicated, involving compressive, tensile, and shearing components. It is thus necessary to evaluate the pseudoelastic behavior of the TiNi matrix by employing a more suitable method.

In this work, a nano-indentation technique was used to evaluate the pseudoelasticity of the TiNi matrix. The indentation process is somewhat similar to the penetration of an asperity into the surface layer during abrasive wear. Due to the complex stress state, it may be more appropriate to evaluate the pseudoelasticity using two parameters: $\eta = W_{rc}/W_t$ and W_t . η is the ratio of the recoverable deformation energy (W_{rc}) to the total deformation energy (W_t) throughout an entire loading and unloading cycle [19]. The deformation energy W_t is represented by the area enclosed by the loading curve and the maximum penetration depth, as illustrated in Fig. 5; while W_{rc} is the area enclosed by the unloading curve and the maximum penetration depth. W_t is a measure of the capability to accommodate deformation, while the parameter η represents the portion of the total deformation that is recoverable. Clearly, these two parameters can be used to evaluate the degree of pseudoelasticity. A material with larger values of η and W_t will behave more pseudoelastically and has higher capacity to absorb impact energy and accommodate larger deformation with less damage. It should be indicated that η is not constant and it varies with changes in the applied load and eventually becomes stable as Fig. 6a illustrates, while W_t always increases as the applied load increases. One may notice that there is no data in Fig. 6 for the TiC/TiNi specimens under high load. This occurred because the matrix of this composite without nano-TiN particles was soft and a further increase in load resulted in the displacement running out of the measurable range of the triboscope.

As illustrated in Fig. 6a, η of the NiTi matrix of 60% TiC/NiTi composite was the highest, followed by that of the 5% nano-TiN/55% TiC/NiTi; while η of the 30% nano-TiN/30% TiC matrix was the lowest. Fig. 6b illustrates W_t values of the matrices of these composites. One may see that the matrix of 60% TiC/TiNi composite had the largest W_t , corresponding to the highest capability to accommodate deformation energy. W_t

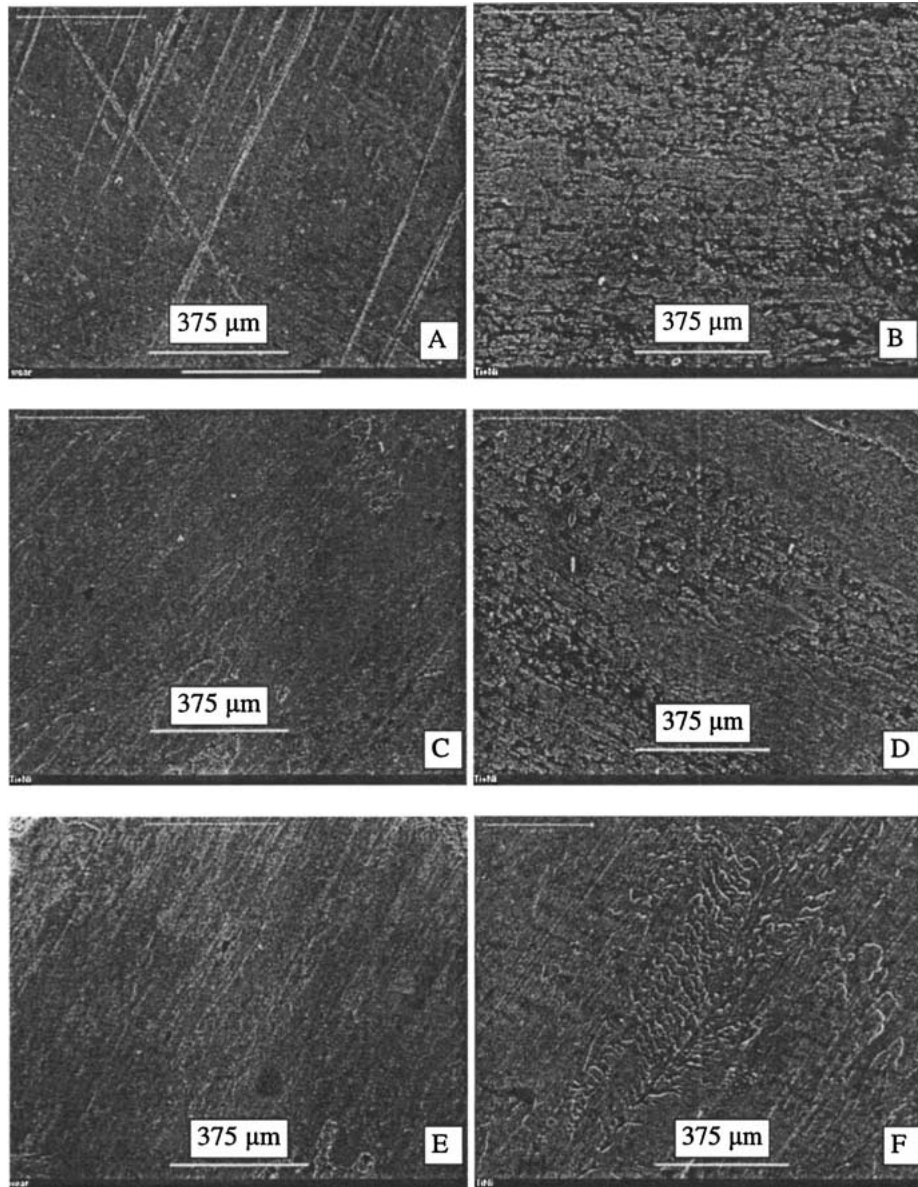


Figure 4 Worn surfaces of the composites under high load and under low load. (A) 30% nano-TiN/30% TiC/TiNi under low load, (B) 30% nano-TiN/30% TiC/TiNi under high load, (C) 60% TiC/TiNi under low load, (D) 60% TiC/TiNi under high load, (E) 5% nano-TiN/55% TiC/TiNi under low load, and (F) 5% nano-TiN/55% TiC/TiNi under high load. [Low load: 0.05 kn, high load: 0.3 kN].

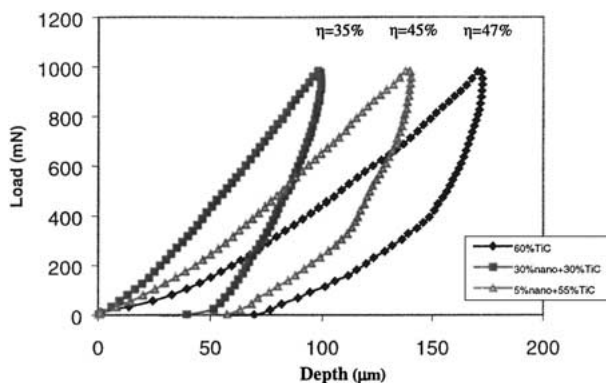


Figure 5 Load-depth curves of the composites.

of the 5% nano-TiN/TiC/TiNi was lower and that of the 30% nano-TiN/TiC/TiNi was the lowest. The indentation test indicates that the nano-TiN powder decreased the pseudoelasticity of the TiNi matrix. The more the nano-powder, the lower was the pseudoelasticity. Such

a decrease in pseudoelasticity is a result of the obstacle set by the nano-TiN particles to the stress-introduced martensitic transformation. As a matter of fact, TiC particles in the composites should also retard the martensitic transformation to some degree. As K. L. Fukami-Ushiro *etc.* observed [20] in their study on a TiC/TiNi composite, the critical stress for inducing the martensitic transformation is slightly higher than that for inducing the martensitic transformation in a TiNi alloy having the same composition. When a lot of nano-TiN particles are dispersively distributed in the matrix, the martensitic transformation would be more difficult to be induced.

3.3.2. Hardness

Although the pseudoelasticity is of importance to the wear resistance of TiNi alloy, the hardness of this alloy also plays a role in resisting wear. The hardness of pseudoelastic TiNi alloy could not be properly

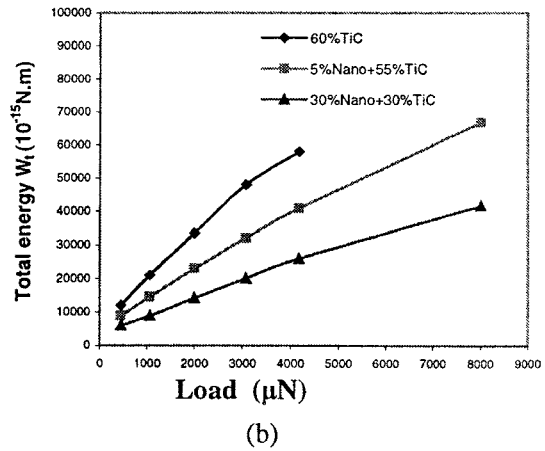
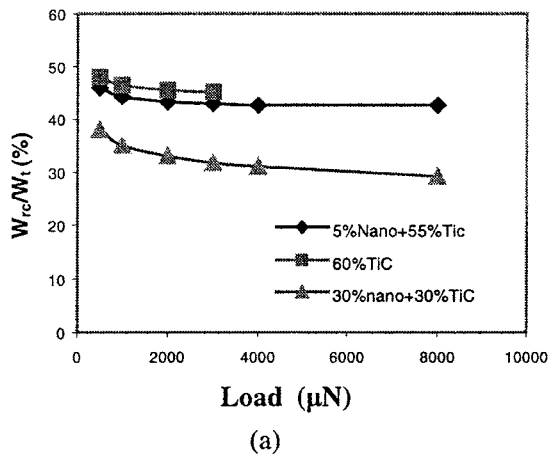


Figure 6 Effects of nano-TiN particles on the pseudoelastic behavior of TiNi matrix of the composites under study. (a) The ratio of recoverable energy to the total deformation energy against the loading force. (b) The total deformation energy against the loading force.

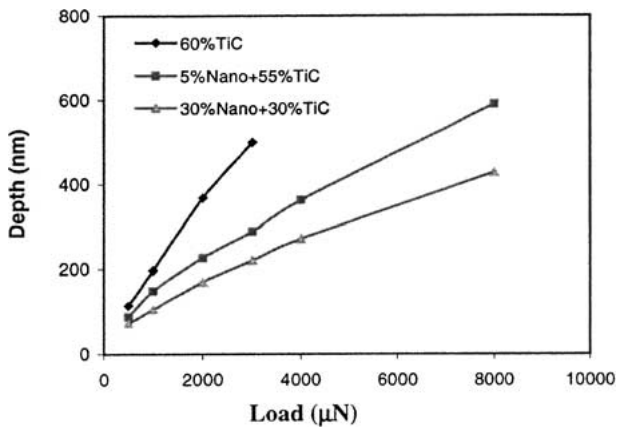


Figure 7 The depth against the indentation load.

determined using conventional hardness tester because of the recoverable pseudoelastic deformation. In this work, the hardness of TiNi matrix was evaluated based on its indentation behavior. The instantaneous penetration depth under a certain load is a measure of the hardness. The smaller the penetration depth, the harder is the material. As illustrated in Fig. 7, the nano-TiNi powder apparently decreased the penetration depth of the TiNi matrix, or increased the matrix's hardness. The nano-TiN powder could pin dislocations during plastic deformation and thus strengthened the matrix. In addition, the nano-TiN particles may also limit the growth of martensite and this could further harden the matrix. As a result, the matrices of 5% nano-TiN/55%TiC/TiNi and 30% nano-TiN/30%TiC/TiNi had higher hardness than that of 60%TiC/TiNi. The more the nano-TiN powder, the harder was the matrix.

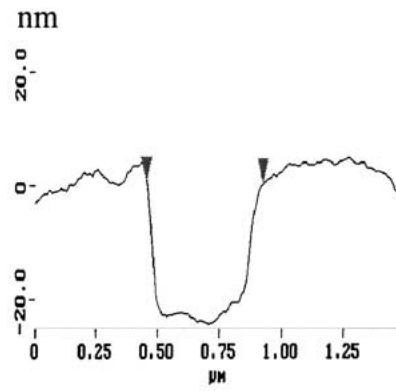
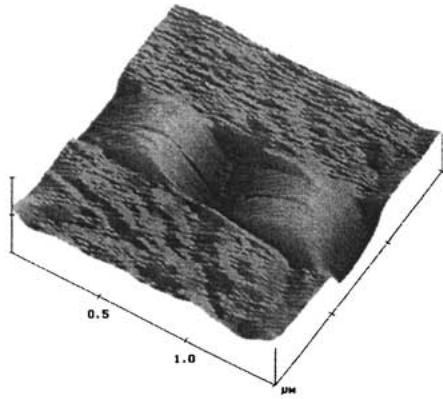
3.4. Microscale-wear behavior of the TiNi matrix

The integral wear resistance of a TiNi-matrix composite is strongly influenced by the mechanical and tribological properties of the matrix. In order to investigate the correlation between the integral wear resistance of and the wear resistance of the TiNi matrix, localized wear of the TiNi matrix was in-

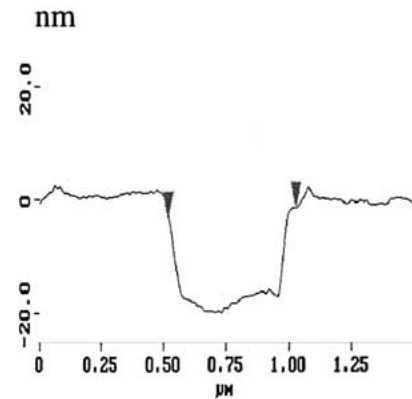
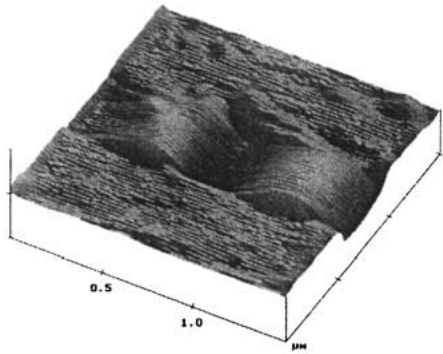
vestigated on microscopic level using the triboscope. The wear test was performed by scratching a very small area ($0.4 \mu\text{m} \times 0.4 \mu\text{m}$), so that the influence of the reinforcing TiC phase could be minimized. Fig. 8 illustrates representative wear tracks and corresponding cross-section profiles experienced wear under a normal load of $50 \mu\text{N}$. The wear loss was counted by estimating the volume loss in the worn area. It was determined that volume losses of the TiNi matrices with 0%, 5%, and 30% are $3.7 \times 10^6 \text{ nm}^3$, $2.9 \times 10^6 \text{ nm}^3$ and $1.4 \times 10^6 \text{ nm}^3$, respectively. It was demonstrated that 5% nano-TiN powder effectively enhanced the wear resistance of the NiTi matrix. This increase in wear resistance mainly resulted from the increase in hardness by the nano-TiN powder. 30% nano-TiN powder resulted in a more significant increase in wear resistance of the matrix. However, although the TiNi matrix containing 30% nano-TiN powder had the highest local wear resistance, the corresponding composite performed the worst (see Fig. 3), i.e. its integral wear resistance was the lowest. The reason for this contradiction has been discussed in the following section.

3.5. Further discussion

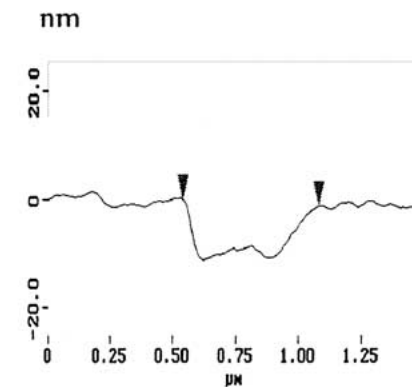
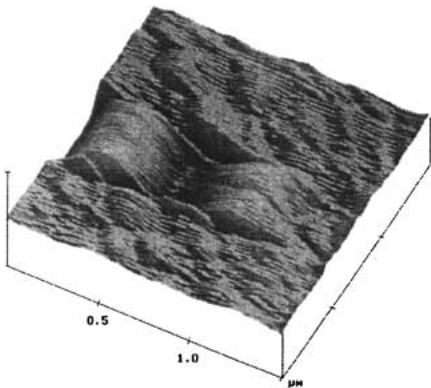
Wear resistance of a composite is an integral property not only dependent on mechanical and tribological properties of each individual phase and the interfacial bond but also dependent on the coordination between the phases. In general, the matrix plays a role as a binder to hold the reinforcing phase as well as to accommodate large-scale deformation and to absorb impact energy. While the reinforcing phase (e.g., hard particles) mainly withstands the external force. It is often found that matrices of many conventional composites are weaker and worn first followed by the removal of the reinforcing particles. Many attempts have been made to improve the wear resistance of a matrix by modifying its composition for higher hardness. However, the degree of such improvement is rather limited, since this modification is somewhat contradictory. If hardness of the matrix is increased, its ductility will decrease, thus reducing its function as a binder to hold the reinforcing phase, to absorb the impact energy and to accommodate large-scale



TiNi matrix with 0% Nano-TiN



TiNi matrix with 5% Nano-TiN



TiNi Matrix with 30% Nano-TiN

Figure 8 Micro-scale wear tracks and the cross-section profiles.

deformation. However, if the hardness of the matrix remains low, it will become the weak region and quick wearing of the matrix will result in rapid removal of the reinforcing particles. Recent work has demonstrated that pseudoelastic TiNi alloy could be an ideal binder material because of its high wear resistance and rubber-like behavior. However, although the pseudoelasticity

greatly benefits the wear resistance, TiNi alloy can still be damaged under high stress when the strain exceeds a critical level and results in plastic deformation. Therefore, the wear resistance could be enhanced if the hardness of TiNi alloy can be enhanced without losing sufficient pseudoelasticity. In this work, the TiNi matrix of TiC/TiNi composite was modified with nano-TiN

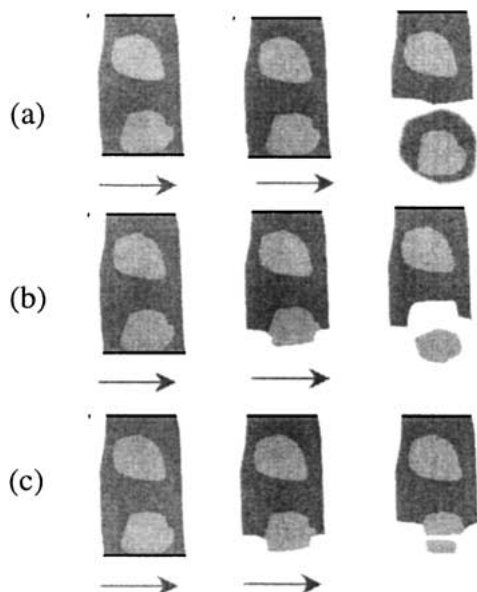


Figure 9 Three models of TiC particle-removal from the composites. (a) Large debris removal, (b) particle drop-out, and (c) gradual worn-out.

powder for a better balance between the pseudoelasticity and hardness. Expected results were obtained.

3.5.1. 60%TiC/TiNi composite

The TiNi matrix of this composite has high pseudoelasticity but low hardness. As a result, the matrix of the composite has relatively low wear resistance in contrast with the matrix containing nano-TiN powder, as demonstrated by the micro-scale wear test (see Fig. 8). Since the matrix was not strong enough, the reinforcing particles could be torn off along with the matrix as wear debris under high loads, as schematically illustrated in Fig. 9a. This may also be seen from the worn surface of the composite under high loads (see Fig. 4D). In such a case, the reinforcing hard particles may play no role in resisting wear. Therefore, this composite has relatively lower integral wear resistance than the composite containing a small amount of nano-TiN powder.

3.5.2. 30% nano-TiN/30%TiC/TiNi composite

By adding 30% nano-TiN powder to replace 30% TiC particles, the matrix was significantly strengthened. However, although such a matrix has the highest wear resistance because of its high hardness, its role as a binder to hold the TiC particles was considerably reduced because of low flexibility. Under external load, the hard matrix has low capability to accommodate large-scale deformation and absorb impact energy. The integral wear resistance of this composite is therefore low, especially under high-stress wear condition as shown in Fig. 4B, where many craters on the worn surface are visible. One may consider that in this case the wear occurred with a particle-drop-out mechanism involved as schematically illustrated in Fig. 9b. As mentioned earlier, we also tested 30% nano-TiN/60%TiC/TiNi and found that this composite was

very brittle. The high brittleness of this composite is another evidence to support the above-addressed point.

3.5.3. 5% nano-TiN/55%TiC/TiNi composite

The wear test demonstrated that 5% nano-TiN/55%TiC/TiNi composite performed the best. The degree of pseudoelasticity of the TiNi matrix containing 5% nano-TiN powder was relatively lower than that of pure TiNi matrix but superior to that of the TiNi matrix containing 30% nano-TiN powder. On the other hand, this matrix was harder than the TiNi matrix but softer than that containing 30% nano-TiN powder. Obviously, a good balance between the pseudoelasticity and hardness can result in significantly improved integral wear resistance. Such a balance could be obtained by adding an adequate amount of hard nano-powder and 5% nano-TiN powder appeared to be the optimal. Although the wear resistance of this matrix is lower than that of the matrix containing 30% nano-TiN powder, 5% nano-TiN powder makes the TiNi matrix in good coordination with the reinforcing phase (TiC). Such a matrix possesses enhanced hardness without losing substantial pseudoelasticity and it is therefore a flexible binder and resistant to wear. Under wear attack, the matrix was gradually worn away and so were the reinforcing particles, as schematically illustrated in Fig. 9c. As a result, the TiC particles are effective as a reinforcing phase and this composite has shown optimal performance. As illustrated in Fig. 4F, the worn surface of 5% nano-TiN/55%TiC/TiNi composite was smooth and few craters appeared on the worn surface. In this case, both the matrix and the reinforcing phase acted effectively, resulting in a significantly improved wear resistance.

In summary, it is clear that the integral wear resistance of TiC/TiNi composite is strongly affected by properties of the matrix and the reinforcing phase as well as the coordination between the phases. A good balance between the pseudoelasticity and hardness of the TiNi matrix may considerably improve the integral wear resistance of the composite. It has turned out that a small amount of nano-TiN powder benefits the TiC/TiNi composite and makes it remarkably superior to WC/NiCrBSi hardfacing overlay, a widely used industrial wear-resistant material.

4. Conclusion

An attempt was made to improve the wear resistance of a newly developed TiC/TiNi tribo composite by modifying the TiNi matrix with nano-TiN powder. Results of this research is summarized as follows:

1. 5% nano-TiN powder apparently improved the integral wear resistance of the TiC/TiNi composite. However, 30% nano-TiN powder is detrimental to the integral wear resistance.
2. The nano-TiN powder increased the hardness of the TiNi matrix at the expense of its pseudoelasticity. The hardness and wear resistance of TiNi matrix containing TiN nano-particles increased with an increase in

the amount of the nano-TiN powder, while its pseudoelasticity decreased as the amount of nano-TiN powder increased.

3. The integral wear resistance of TiNi-matrix composite is strongly influenced by both hardness and pseudoelasticity of the matrix. A good balance between hardness and pseudoelasticity of the TiNi matrix may significantly improve the integral wear resistance of the composite.

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