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Microstructure and wear properties of laser clad Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coatings

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ABSTRACT Wear resistant Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coatings with a microstructure consisting of Ti_2Ni_3Si primary dendrites and interdendritic Ti_2Ni_3Si/Ni_3Ti eutectic were fabricated on a substrate of 0.2% C plain carbon steel by a laser cladding process with Ti-Ni-Si alloy powders. The Ti_2Ni_3Si/Ni_3Ti coatings have excellent wear resistance and a low coefficient of friction under metallic dry sliding wear test conditions with hardened 0.45% C carbon steel as the silide-mating counterpart. The excellent tribological properties of the coating are attributed to the high hardness, strong covalent-dominant atomic bonds of the ternary metal silicide Ti_2Ni_3Si and to the high yield strength and strong yield anomaly of the intermetallic compound Ni_3Ti.

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1 Introduction

Transition metal silicides represent the largest family of intermetallic compounds and are considered as a new candidate class of high- and/or ultrahigh-temperature structural materials because of their combinations of high melting point, low density, high elastic modulus, and excellent creep and oxidation resistance [1–6]. From tribology and surface engineering points of view, however, these ordered transition metal silicides-based alloys, especially those ternary metal silicides with the topologically-closed-packed (TCP) *hP*12 $MgZn_2$ type Laves phase crystal lattice such as W_2Ni_3Si, Mo_2Ni_3Si, Ti_2Ni_3Si, etc., can be shown to have outstanding abrasive wear resistance. This is due to their intrinsic high hardness, strong anomalous hardness–temperature relationship, adhesive wear resistance, and low coefficient of friction. Their strong covalent-dominated atomic bonds and high hardness also play a significant role. They are a new class of advanced wear resistant coating-materials for those tribological mechanical components working under aggressive service conditions [7–11]. Room-temperature brittleness and poor processing capability are the main obstacles that restricted these monolithic intermetallic alloys from industrial applications as tribological components [1–6]. Introducing a ductile second metallic phase [2, 6, 12–18], and/or fabricating a multiphase intermetallic microstructure [2, 19, 20], have been postulated and have been shown to be the most effective means of enhancing the toughness and ductility of most intermetallic alloys. Preliminary results conducted in the authors' group also demonstrated that some of the multiphase transition metal silicides-based intermetallic alloys have displayed excellent tribological properties and processing abilities, as novel wear resistant coating materials [7–11, 21–25].

The binary intermetallic compound Ni_3Ti with a $D0_{24}$ crystal structure is well-known for its high yield strength and especially for its strong yield anomaly (i.e., increasing yield strength with increasing temperature) and excellent thermal, chemical, and microstructural stability, and is widely utilized as an important strengthening phase called η-phase for many nickel and Fe-based superalloys [26–28]. A multiphase intermetallic alloy with the hard ternary metal silicide Ti_2Ni_3Si as the wear resistant reinforcing phase and the relatively more ductile Ni_3Ti phase as the matrix is expected to be a promising wear resistant coating material having outstanding tribological properties. In this paper, the Ti_2Ni_3Si/Ni_3Ti intermetallic coatings were fabricated on a substrate of 0.2% C low carbon steel by a laser cladding process using Ti-Ni-Si alloy powders as the precursor materials. Wear properties of the coatings were evaluated under dry sliding wear test conditions, coupling a hardened 0.45% C carbon steel at room temperature. The wear behaviors are subsequently discussed.

2 Experimental procedures

Laser cladding experiments were conducted on a 5 kW continuous-wave CO_2 laser material processing system, equipped with a 4-axis computer numerical controlled (CNC) table, in a dynamically-sealed argon shielded processing chamber, using a coaxial powder-blowing process with high-purity (99.999 wt. %) argon as the powder-carrying gas. A commercial hot rolled 0.20% C plain carbon steel, 100 mm × 15 mm × 10 mm in size, was selected as the substrate material. Commercial pure 32% Ni–60% Ti–8% Si (in wt. %) alloy powders with an average particle size of 70 to 140 μm were selected as the precursor materials for fabricating the intermetallic coatings using the powder-blown laser cladding process. The coaxially delivered Ti-Ni-Si powders

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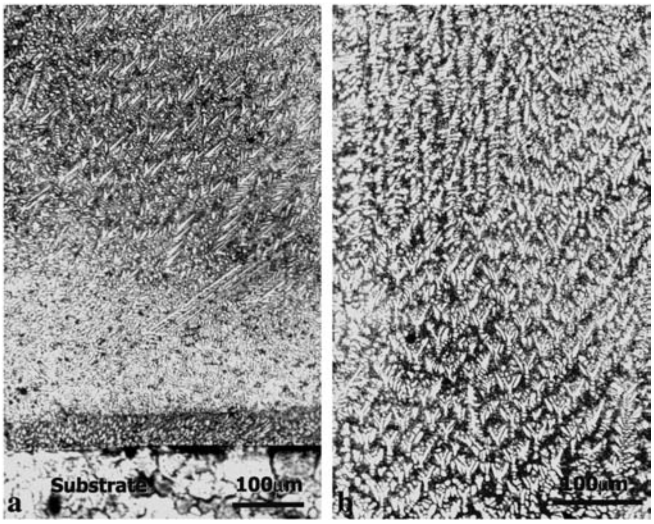

FIGURE 1 OM photographs showing the overall microstructure in the lower part **a** and upper part **b** of a longitudinal cross section of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic composite coating

were melted onto the surface of the 0.20% C steel with a very thin superficial melting of the substrate under the irradiation of a focused laser beam, producing a laser clad Ti-Ni-Si alloy melt-pool with negligible dilution from the substrate. Subsequently this lead to the fabrication of a Ti_2Ni_3Si/Ni_3Ti intermetallic coating, metallurgically bonded to the steel substrate after rapid solidification of the melt-pool following the forward movement of the scanning laser beam. The laser cladding parameters were: laser beam power 2.5 kW, beam diameter 3 mm, beam scanning speed 200 mm/min, flow rate of the argon carry-gas $150 \sim 180$ L/h, and the flow rate of the powder stream was $3.8 \sim 4.2$ g/min. Five overlap tracks were clad side by side with an overlap ratio of approximately 30% in order to cover the whole 100 mm × 10 mm surface of the substrate specimen. After the first layer was clad, another two layers were successively deposited in order to produce a thicker coating with a final coating thickness of approximately 1.9 mm.

Transverse and longitudinal metallographic cross sections and wear test specimens of the Ti_2Ni_3Si/Ni_3Ti coatings were prepared by electric discharging machining (EDM). Metallographic samples were prepared using standard mechanical polishing procedures and were etched in H_2O–7 vol.%HF–43 vol.%HNO_3 at ambient temperature for approximately 25–55 seconds. The microstructure was characterized using the Neophot II optical microscope (OM) and the JSM-5800 and KYKY-2800 scanning electron microscopes (SEM). Phases of the coatings were identified by X-ray diffraction (XRD) using the Rigaku D/max 2200 pc automatic X-ray diffractometer using a Cu target K_α radiation operated at 40 kV, with a scanning rate of 0.5°/s on the top surface of the coating specimen after mechanical grinding. Chemical compositions of the phases were analyzed by energy dispersive X-ray analysis (EDS) using a Noran Ventage DSI spectrometer. The volume fraction of the Ti_2Ni_3Si primary dendrites in the laser clad Ti_2Ni_3Si/Ni_3Ti coatings was measured using commercial contrast-based computer image analyzing software on high-contrast optical photographs (×500 magnifications). Hardness profiles along the coating depth direction were measured using a MH-6 semi-automatic Vickers microhardness tester with a test load of 200 grams and a load-dwell time of 15 seconds.

Wear properties of the Ti_2Ni_3Si/Ni_3Ti coatings were evaluated on a MM-200 block-on-wheel dry sliding wear tester at room temperature, where the top flat surface of the square block Ti_2Ni_3Si/Ni_3Ti coating specimen (10 mm × 10 mm × 10 mm in size) is pressed, under an applied test load of 98 N, against the outer periphery surface of a hardened 0.45% C plain carbon steel wheel (HRc53) rotating at a speed of 400 rpm. The outer diameter of the steel wheel is 40 mm and the relative sliding speed between the coating specimen and the contact-coupling wheel is 0.838 m/s. The wear test cycle lasted for 60 minutes and the total wear sliding distance is approximately 3016 m. Details of the dry sliding wear tester and the test procedures were reported elsewhere [10, 24]. A solution treated single-phase austenitic stainless steel AISI 321with an average Vickers hardness number of HV180 was selected as the reference material for all wear tests in order to eliminate the system errors of the wear tester. Parallel wear tests on a quenched and tempered low-alloy high-carbon tool steel of nominal composition (wt. %) of 1.0 C–1.5 Cr, and with an average Rockwell hardness number of HRc58 $\sim$ 60, were also conducted for comparison purposes. Each coating specimen is matched with a reference specimen and each test was repeated twice. Wear mass loss was measured using an electronic balance (Sartorius BS110) with an accuracy of

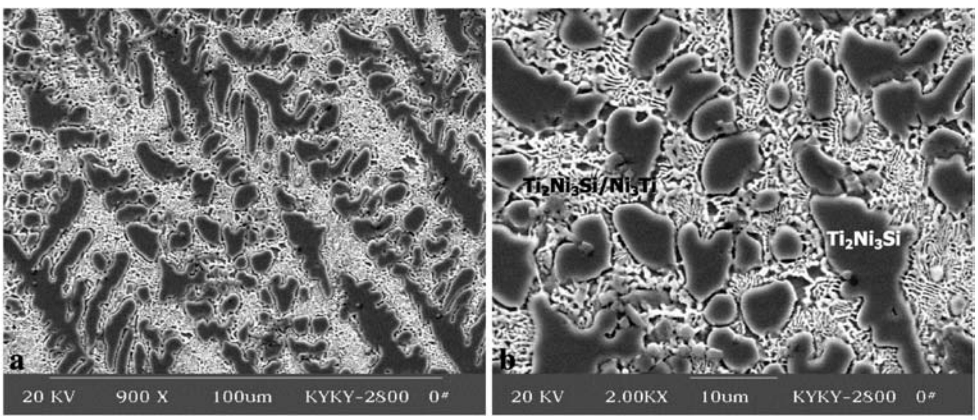

FIGURE 2 SEM micrographs showing the typical microstructure of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic composite coating, **a** low and **b** high magnifications

0.1 mg. Relative wear resistance (i.e., ratio of wear mass loss of the reference material to that of the coating material), was used to rank the wear resistance of the coating in reference to the reference materials. Worn surface and wear debris particles collected during the wear testing process were examined under an SEM to assist in the analysis of wear mechanisms.

3 Results

The laser clad Ti-Ni-Si intermetallic coating, with an average coating thickness of approximately 1.9 mm and a high quality metallurgical bonding to the substrate, has an uniform microstructure consisting of pre-eutectic primary dendrite and interdendritic irregular lamellar eutectics, as shown in Fig. 1, and more clearly in Fig. 2. Results of X-ray diffraction analysis indicate that the main phase constituents of the coatings are the titanium nickel ternary silicide Ti_2Ni_3Si, and the binary intermetallic compound Ni_3Ti, as indicated in Fig. 3. Energy dispersive X-ray analysis (EDS) indicates that the average chemical composition (at. %) of the primary dendrite is approximately 28% Ti–53% Ni–12% Si–7% Fe and is identified as the ternary silicide Ti_2Ni_3Si, a topologically closed packed (TCP) phase having the *hP*12 $MgZn_2$ type Laves crystal lattice [2, 5, 29], whereas the interdendritic eutectic is Ti_2Ni_3Si/Ni_3Ti, as shown in Fig. 3. The volume fraction of the Ti_2Ni_3Si primary dendrites is approximately 46%. The laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating has a high hardness and a relatively uniform hardness distribution in the upper two thirds of the coating, as indicated in Fig. 4. The average Vickers hardness number of the upper part of the coating is over HV950. The hardness gradually decreases from approximately HV900 to HV600 in the bottom part of the coating. This is due to the lower volume fraction of the primary dendrites and the dilution of the coating by the substrate during the laser clad-deposition of the first layer of the coating.

Wear test results indicate that the laser clad Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coating displayed excellent tribological properties under room-temperature dry sliding wear test conditions when slide-coupling with the rotating hardened 0.45% C carbon steel mating wheel. As indicated in Table 1, wear mass loss of the Ti_2Ni_3Si/Ni_3Ti intermetallic coating is much lower than that of both the austenitic stainless steel AISI321, and even the hardened tool steel 1.0% C–1.5% Cr. Relative wear resistance of the intermetallic coating is up to 60 times higher than the austenitic stainless steel, and up to 12 times higher than the hardened tool steel. Moreover, the

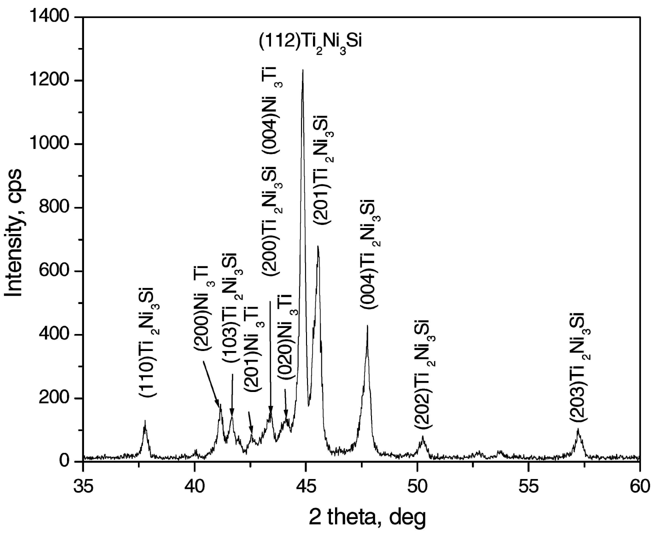

FIGURE 3 X-ray diffraction patterns of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating

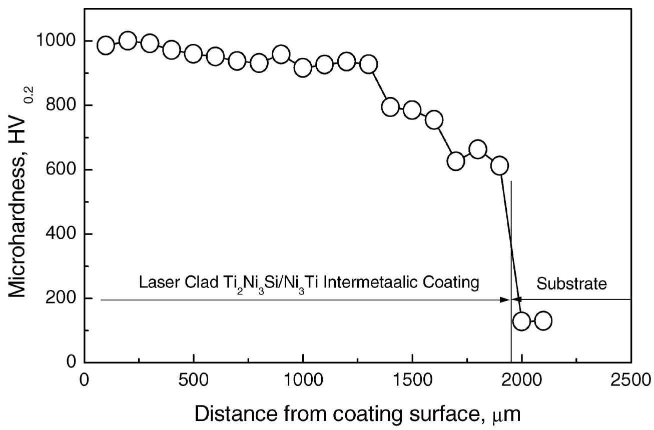

FIGURE 4 Hardness profile of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating

Test materials	Wear Mass Loss, g	Relative Wear Resistance
Ti_2Ni_3Si/Ni_3Ti coating	0.0055	60.9
Hardened Tool Steel 1.0% C–1.5% Cr	0.0278	12
Austenitic Stainless Steel AISI 321	0.3348	1

TABLE 1 Wear test results of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating and the hardened high carbon tool steel 1.0% C–1.5% Cr in comparison to the AISI321 austenitic stainless steel

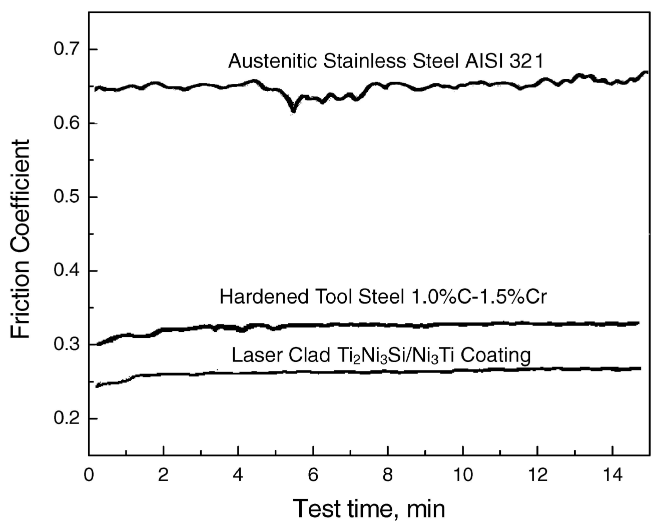

FIGURE 5 Profiles of friction coefficients versus wear test time of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating, the austenitic stainless steel AISI321, and the hardened tool steel 1.0% C–1.5% Cr reference materials, during the dry sliding wear process coupling with a hardened 0.45% C carbon steel wheel

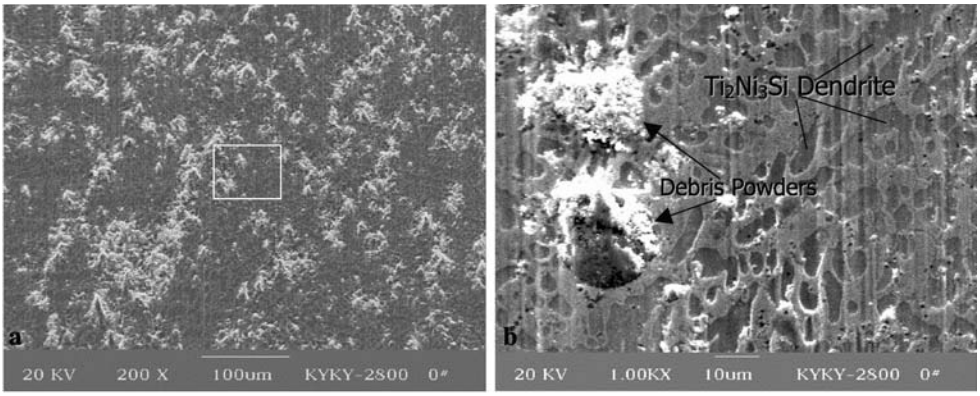

FIGURE 6 SEM micrographs showing the worn surface morphologies of the Ti_2Ni_3Si/Ni_3Ti intermetallic coating after the dry sliding wear test with hardened 0.45% C steel for 60 min, **a** low and **b** high magnifications

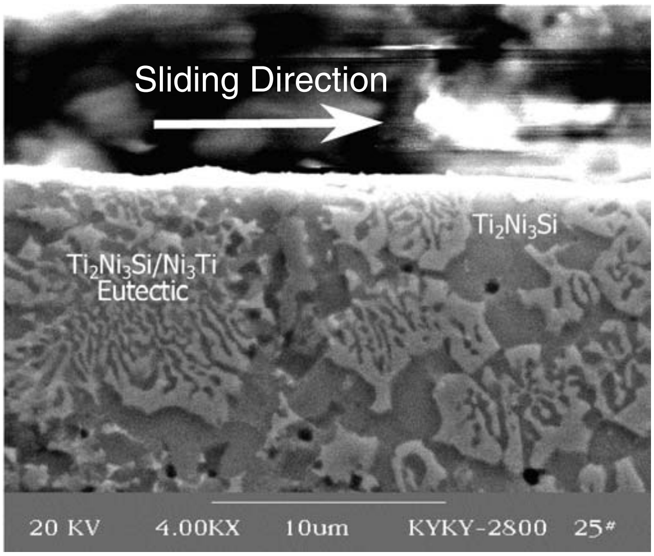

FIGURE 7 SEM micrograph showing worn subsurface microstructure of the Ti_2Ni_3Si/Ni_3Ti coating on a cross section perpendicular to worn surface along the sliding direction

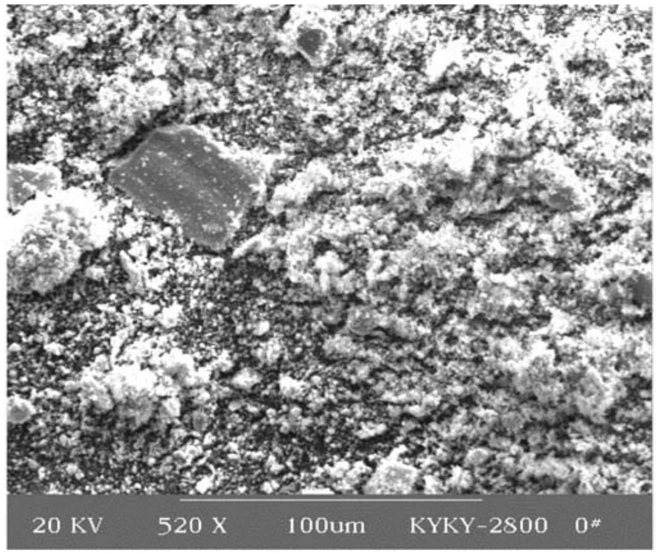

FIGURE 8 SEM micrograph showing the wear debris morphologies of the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating after dry sliding wear test coupling with a hardened 0.45% C carbon steel counterpart

coefficient of friction of the intermetallic coating was noticeably reduced in comparison to the hardened tool steel 1.0% C–1.5% Cr, and was much more lower than the austenitic stainless steel, as indicated in Fig. 5.

The worn surface of the Ti_2Ni_3Si/Ni_3Ti intermetallic coatings after a dry sliding wear test cycle of 60 min is very smooth with almost no adhesive and abrasive wear characteristic features observable (e.g., micro-cutting, micro-plowing, deformation ridges or materials tearing traces, etc.), as indicated in Fig. 6a. The worn surface resembles a polished and etched metallographic section apart from some tiny loosely stuck wear debris particles, or islands of weakly consolidated powder-agglomerates, on which the pre-eutectic Ti_2Ni_3Si primary dendrites and even the interdendritic eutectics could be revealed, as shown in Fig. 6b. Such special worn surface morphologies were also observed for the laser clad intermetallic coatings reinforced by the ternary metal silicides having the *hP*12 Laves phase crystal structure of Mo_2Ni_3Si [7, 8] and W_2Ni_3Si [9, 10]. This unique worn surface morphology implies that both the ternary silicide Ti_2Ni_3Si and the binary intermetallic Ni_3Ti, are free-from wear attacks in terms of metallic adhesion, plastic deformation, and abrasive wear via the mechanisms of micro-cutting and micro-plowing during the metallic dry sliding wear process. Worn subsurface microstructure on longitudinal sections along the sliding direction, as shown in Fig. 7, show no evidences of local subsurface plastic deformation, brittle fragmentation, or phase preferential wear to both Ti_2Ni_3Si primary dendrites, and the interdendritic Ti_2Ni_3Si/Ni_3Ti eutectic. SEM observations indicate that all wear debris consists of tiny powders or loosely consolidated powder-agglomerates, as shown in Fig. 8. EDS analysis indicates that the fine wear debris particles are highly enriched in Fe, O, and with a minor amount of Ti, Ni, and Si. Indicating that the debris originated primarily from the contacting surface of the mating carbon steel counterpart.

4 Discussions

The laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating exhibited excellent wear resistance and a low coefficient of friction under metallic dry sliding wear test conditions, with hardened 0.45% C steel as the slide-mating counterpart, as indicated in Table 1 and Fig. 5. It is the fully intermetallic nature of the coating's phase constituents and their unique tribological behavior that provided the Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coatings with excellent tribological properties under metallic dry sliding wear test conditions.

Many intermetallic compounds having the *hP*12 $MgZn_2$ type Laves phases crystal structure, such as the ternary metal silicide Ti_2Ni_3Si, are reported to have the capability to retain 85% of their ambient yield strength at temperatures as high as half of their homologous melting temperature, which is much higher than all other intermetallics [1, 2, 5, 6, 12]. The intermetallic compound Ni_3Ti with the $D0_{24}$ crystal structure is famous for its increasing yield strength with increasing temperature, and for its excellent chemical and phase stabilities up to high temperatures very close to its melting point, and was widely used as a primary precipitation strengthener for many nickel- and Fe-base superalloys [26–28]. It is actually the strong atomic bonds, intrinsic high hardness, and the unique abnormal hardness–temperature relationship of the phase constituents of the laser clad intermetallic coating, i.e., the ternary metal silicide Ti_2Ni_3Si and Ni_3Ti, that provide the coating with excellent abrasive and adhesive wear resisting capabilities, and a low coefficient of friction under metallic dry sliding wear test conditions coupling with a metallic counterpart.

It is believed that when the dry slide-interacts with a metallic surface, the local temperature at the contacting surface is high and the hardness of the materials near the contact surface will quickly be decreased for conventional wear resistant metallic materials due to the very localized temperature rise. This results in serious contact surface plastic deformation, local contact-welding between the contacting asperities, material transferring, debonding or tearing of the adhered junctions, and consequently leads to serious material removal from the friction surface as patch-like or thin flake-like wear debris.

The laser clad Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coating, has an excellent capability to resist abrasive wear attacks such as micro-cutting, micro-plowing, repeated surface plastic deformation. This is because the ternary metal silicide Ti_2Ni_3Si has as its main phase constituents a topologically closed packed (TCP) phase with a *hP*12 $MgZn_2$ type Laves phase crystal structure, and an average hardness of over HV980, and the constituent Ni_3Ti which is an intermetallic compound with a $D0_{24}$ crystal structure, and an increasing yield strength with increasing temperature, in addition to excellent chemical and phase stabilities up to high temperatures, very close to its melting point. Due to the high intrinsic hardness of these phase constituents of the coating, it also exhibits an excellent abrasive wear resistance when dry sliding with a metallic mating counterpart.

Moreover, the anomalous hardness–temperature dependence relationship of the intermetallic phases of Ti_2Ni_3Si and Ni_3Ti, is beneficial to the tribological properties by guaranteeing that the coating materials on or in the vicinity of the contacting surface still keep the original, or even higher hardness and yield strengths. Hence providing the coating with excellent resistance to abrasive wear attacks (micro-cutting, micro-plowing, etc.), and adhesive wear attacks such as surface contact plastic deformation, cold-welding, junction formation at the contacting asperities, and materials transferring, etc, when dry sliding with the surface of the hardened 0.45% C carbon steel coupling wheel under applied pressure. This is in spite of the friction-induced high local temperature at the contacting surface.

The above discussions are justified by the very low wear mass loss, compared to the comparison metallic materials of austenitic stainless steel, and hardened tool steel (as shown in Table 1), and also by the very smooth worn surface not showing any noticeable cutting or plowing-induced grooves (as shown in Fig. 5). Furthermore, the strong and dominant covalent atomic bonds, high hardness of the Ti_2Ni_3Si, high yield strength, and abnormal strength-temperature dependence of the Ni_3Ti, gives the coating excellent resistance to metallic adhesion, or local contact plastic deformation-induced cold-welding, junction formation, and materials-transferring at the contacting asperities, etc. The coating also has excellent adhesive wear resistance and a low coefficient of friction when slide-coupling with a metallic counterpart. This is evidenced by the very smooth and clean worn surface without any metallic adhesion characteristics observable as shown in Fig. 5b, and by the considerably lower friction coefficient of the coating compared to the test reference materials (as indicated in Fig. 6).

In summary, the laser clad Ti_2Ni_3Si/Ni_3Ti intermetallic coating can only be worn very slowly through the mechanism of slight soft-polishing or rubbing of the slide-mating surface of the 0.45% C steel counterpart during the dry sliding wear process. This is because both the ternary silicide Ti_2Ni_3Si, and the binary intermetallic Ni_3Ti, can be neither plastically deformed, metallically adhered, transferred nor micro-cut, plowed under the dry sliding interaction with the metallic friction counterpart. Consequently this imparts the coating with excellent abrasive and adhesive wear resistance and a low coefficient of friction.

5 Conclusions

Wear resistant Ti_2Ni_3Si/Ni_3Ti intermetallic composite coatings were fabricated on a substrate of 0.2% C low carbon steel by laser cladding. The microstructure of the coatings consists of ternary metal silicide Ti_2Ni_3Si primary dendrites, and interdendritic Ti_2Ni_3Si/Ni_3Ti eutectic. The T_2Ni_3Si/Ni_3Ti intermetallic coatings have excellent abrasive and adhesive wear properties and a low coefficient of friction under dry sliding wear test conditions because of the intrinsic high hardness, strong covalent dominant atomic bonds, and the strong hardness anomaly of the phase constituents of the laser clad Ti_2Ni_3Si/Ni_3Ti multiphase intermetallic coatings.

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