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Mechanical and Tribological Properties of Cold-Sprayed Ti Coatings on Ti-6Al-4V Substrates

N.W. Khun, A.W.Y. Tan, and E. Liu

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A cold spray process was used to deposit titanium (Ti) coatings of different thicknesses on commercial Ti-6Al-4V (Ti64) substrates. The hardness of the Ti coatings was measured using a Vickers micro-indenter. It was found that the thicker Ti coatings had higher hardness probably due to the better uniformity and higher density of the coatings. The tribological results showed that the friction and wear of the Ti coatings tested against a steel ball under dry condition became lower with higher thickness probably due to the higher wear resistance of the thicker coatings associated with their higher hardness. The specific wear rates of all the Ti coatings were significantly lower than that of the Ti64 substrate as a result of the higher wear resistance of the Ti coatings associated with their cold-worked microstructures and the formation of high wear resistant oxide layers on their wear tracks during the wear testing.

Keywords cold spray, friction, hardness, microstructure, thickness, Ti coating, wear

1. Introduction

Titanium (Ti) and its alloys possess unique properties of light weight, high specific strength, high elastic modulus, and high corrosion resistance, which make them potential for aerospace applications (Ref 1). However, the poor resistance of Ti and its alloys to high temperature oxidation and abrasive wear limits their applications (Ref 2-4).

Generally, aerospace components are very expensive and require long delivery periods. Aerospace components require repair for reasons such as wear, impact damage, or corrosion during service. Traditionally, thermal spray and welding processes have been used to repair damaged aerospace components (Ref 5-7). However, thermal repair processes have major drawbacks for heat-sensitive materials or aerospace components. In addition, metallic bulk forms or coatings produced by thermal processes have high thermal residual stresses and high porosity or oxidation levels, which in turn can degrade their mechanical properties and consequently affect the service life of repaired aerospace components with them (Ref 5-7). Although it is necessary to produce relatively thick coatings for repair of aerospace components, high thermal residual stress prevents a deposition of thick coatings on the

components. Therefore, cold spray process is considered in the aerospace industry as an alternative process to overcome these drawbacks because metallic coatings can be deposited on metallic substrates at a supersonic speed without significant changes in microstructures, and oxidation or porosity level of the coatings which are normally associated with thermal processes (Ref 5-7). Moreover, cold spray process holds a promise for use in repair applications because it is a low temperature process without significant melting of spray particles to produce metallic coatings with cold-worked microstructure, very little oxidation, low porosity level, lack of thermal residual stress, and high thickness. It was reported that cold-sprayed aluminum (Al) alloy coatings had better mechanical and tribological properties than their Al-6061 alloy substrate (Ref 8). Li et al. (Ref 9) found that the wear resistance of cold-sprayed iron (Fe) Al alloy coating was higher than that of heat-resistant 2520 stainless steel even under a working temperature of 800 °C. Bakshi et al. (Ref 10) investigated the tribological properties of cold-sprayed composite coatings containing Al and Al-silicon (Si) and found that Al-Si coatings had lower wear resistance than Al and Al/Al-Si ones. A study conducted by Melendze et al. (Ref 11) showed that tungsten carbide (WC)-based composite coatings fabricated by cold spraying had highest wear resistance for their highest WC content. Recently, Khun et al. (Ref 4) investigated the effect of Al-Cr-Fe quasicrystal content on the wear resistance of cold-sprayed Ti coatings on commercial Ti substrates and reported that increasing the quasicrystal content increased the wear resistance of the Ti coatings. It is clear that the available data on the tribological properties of cold-sprayed Ti coatings are still very limited probably due to the private nature of the cold spray technology and its research and development stage. Therefore, an understanding of the tribological properties

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of cold-sprayed Ti coatings with respect to thickness is important for successful tribological applications.

In this study, Ti coatings of different thicknesses were fabricated on commercial Ti-6Al-4V (Ti64) substrates using a PCS1000 cold spray system. Their mechanical and tribological properties were systematically investigated with respect to thickness using Vickers micro-hardness and ball-on-disk micro-tribological tests, respectively.

2. Experimental Details

2.1 Sample Preparation

Commercial Ti (99.9%, Titan Singapore) powder was used as the starting powder. The Ti coatings of different thicknesses of about 100 μm to 6200 μm were deposited on the commercial Ti64 substrates with 50 mm $\times$ 50 mm $\times$ 8 mm (Grade 5, Titan Singapore) using a PCS1000 cold spray system. The open cold spray process parameters were described in Table 1. The coating with a thickness of “0” μm in the relevant figures represented the Ti64 substrates.

2.2 Characterization

The surface and cross-sectional microstructures of the samples were observed using optical microscopy (OM, Zeiss Axioskop 2, JVC color video camera). Prior to the OM observation, the samples were submerged in liquid epoxy contained in Teflon molds with 25 mm in diameter and 15 mm in thickness each (Struers). After curing of the epoxy at room temperature (RT $\sim$ 22 to 24 $^{\circ}\text{C}$) for one day, the molded samples were ground using 320 grit papers followed by chemical-mechanical polishing (CMP) with a DiaPro solution containing 9- μm diamond particles and

then an OP-S suspension solution containing 0.04- μm colloidal silica particles (Struers). Kroll's reagent was used to etch the polished samples for the microstructural evaluation.

The microstructures, and fracture and wear morphologies of the samples were studied using scanning electron microscopy (SEM, JEOL-JSM-5600LV). For the morphological evaluation of the fractured Ti coatings, the cross-section of each coated sample was cut with wire electro-discharge machining from the backside of the Ti64 substrate to the half thickness of the coating. The coating was then broken by bending for the observation of the fractured coating morphology.

The OM was used to measure the cross-sectional porosity levels of the coatings. Ten OM micrographs (before etching) per sample were analyzed using ImageJ software to get an average porosity level.

The surface topographies and wear tracks of the samples were measured using surface profilometry (Talyscan 150) with a diamond stylus of 4 μm in diameter. Root-mean-squared surface roughness, R_q , of the samples was averaged from three measurements on each sample.

The surface and cross-sectional hardnesses of the samples were measured using a Vickers micro-indenter (Future-tech FM-300e) under a normal load of 200 g (1.96 N). The surface hardness was averaged from six indentation measurements per sample. The average cross-sectional hardness was taken from 2 to 3 indentation measurements on each sample.

The chemical structures of the samples were investigated using X-ray photoelectron spectroscopy (XPS) (Kratos Axis Ultra). The X-ray source used was a monochromatic Al K α line ($h\nu = 1486.71$ eV) powered with 10 mA and 15 kV. A pass energy of 40 eV was used for detail scans of C 1s, O 1s, and Ti 2p.

Table 1 Cold spray process parameters used for fabrication of Ti coatings

Working gas	Gas temperature	Gas pressure	Nozzle/scan speed	Spray angle	Stand-off distance	Step distance
Helium	350 $^{\circ}\text{C}$	2 MPa	100-400 mm/s	90 $^{\circ}$	25 mm	1 mm

Fig. 1 (a) SEM micrograph showing size distribution of Ti particles used for fabrication of Ti coatings and (b) EDX spectrum of Ti particles

The tensile bond strengths of the coatings were measured using a FM1000 adhesive according to RPS927/5.

The friction and wear of the samples were measured using a ball-on-disk micro-tribometer (CSM high temperature) (Testing conformed to DIN50324 and ASTM G99) and their values were averaged from three measurements per sample. The polished samples used for the microstructural evaluation were used for the tribological tests. The samples were tested against a 100Cr6 steel ball of 6 mm in diameter in a circular path of 1 mm in radius for 30,000 laps at a sliding speed of 3 cm/s under a normal load of 1 N.

3. Results and Discussion

Figure 1(a) shows the SEM micrograph of Ti particles used as starting powder for the fabrication of the Ti coatings on which irregular-shaped Ti particles with a significant variation in sizes from about 10 to 44 μm are clearly seen. The EDX spectrum measured on the Ti particles (Fig. 1b) confirms that the spray particles used in this study are pure Ti particles.

Figure 2(a) shows the fracture morphology of the Ti coating with a thickness of about 6200 μm on which the presence of some craters is indicative of a relatively strong

Fig. 2 (a and b) SEM micrographs showing (a) fracture morphology and (b) surface microstructure of Ti coating with a thickness of about 6200 μm , (c and d) OM micrographs showing (c) surface and (d) cross-sectional microstructures of the same coating, and (e and f) OM micrographs showing cross-sectional microstructures (including interfaces) of Ti coatings with thicknesses of about (e) 100 and (f) 6200 μm

bonding between sprayed Ti particles (Ref 12). Figure 2(b) shows the surface microstructure of the same Ti coating polished without etching on which interfaces between sprayed particles are apparently found.

Figure 2(c) and (d) show the surface and cross-sectional microstructures of the etched Ti coating with a thickness of about 6200 μm on which individual particles with a visible interface can be clearly seen. It is noticed that the Ti coating contains a significant number of pores in its microstructures.

Figure 2(e) and (f) show the cross-sectional microstructures of the Ti coatings with thicknesses of about 100 and 6200 μm observed before etching. The both Ti coatings consistently have a significant number of pores in their microstructures. Interfaces between the Ti coatings and Ti64 substrates are apparently found in Fig. 2(e) and (f). Comparison of Fig. 2(e) and (f) shows that the coating/substrate interface seems to be slightly wider for the higher thickness of about 6200 μm . Since a poor interfacial bonding between the coating and substrate gives rise to a significant gap between them, the slightly wider interface between the Ti coating with a higher thickness of about 6200 μm and Ti64 substrate can be correlated to the lower bond strength of the coating on the Ti64 substrate. The bond strength of the Ti coatings was measured with respect to thickness. The bond strength of the Ti coating with a thickness of about 100 μm is about 18 MPa. The increased thickness of the Ti coating to about 6200 μm results in a 41 % decrease in its bond strength. It is clear that the bond strength of the Ti coatings is influenced by their thickness because the higher thickness probably results in the higher residual stress in the Ti coatings (Ref 5, 6).

The porosity levels of the Ti coatings with thicknesses of about 100, 700, 1000, and 6200 μm are about 21.3 ± 2.7 , 14.9 ± 4.1 , 14.8 ± 4.7 , and 10.1 ± 6.8 %, respectively. The decreased porosity level of the Ti coatings with the increased coating thickness is attributed to that a longer deposition duration probably gives rise to better uniformity and higher density of the coatings.

Figure 3 illustrates the fitted XPS Ti 2p, O 1s, and C 1s peaks of the Ti coating with a thickness of about 6200 μm measured on its fracture morphology shown in Fig. 2(a).

Fig. 3 Fitted XPS peaks of Ti coating with a thickness of about 6200 μm measured on its fracture morphology shown in Fig. 2(a): (a) Ti 2p, (b) O 1s, and (c) C 1s

Fitting of the XPS peaks was performed by decomposing each of them into several components with a Gaussian line shape and a Shirley background. As shown in Fig. 3(a), two spin-orbit coupling peaks of the Ti 2p are recorded at 458.2 eV (full-width-at-half-maximum (FWHM): 2.4 eV) for the Ti 2p_{3/2} peak and 463.8 eV (FWHM: 3.4 eV) for the Ti 2p_{1/2} peak. In Fig. 3(b), the single O 1s peak with a FWHM of 3.6 eV at 529.7 eV is attributed to O-Ti bonds (Ref 13). In Fig. 3(c), the C 1s peak is decomposed into three components assigned to C-C bonds at 284.8 eV (FWHM: 1.7 eV), C-O bonds at 286.1 eV (FWHM: 1.6 eV), and C=O bonds at 287.6 eV (FWHM: 3.1 eV), respectively (Ref 13, 14). The C 1s and O 1s peaks come from surface carbon and oxygen contaminants adsorbed after exposure to air (Ref 13, 14).

Figure 4 shows the surface hardness of the Ti coatings as a function of thickness. The hardness of the Ti64 substrate is about 371 Hv. The Ti coating with a thickness of about 100 μm has relatively low surface hardness of about 120 Hv probably due to its high porosity level (Ref 4). The Ti coating with a thickness of about 700 μm has similar surface hardness of about 120 Hv to that of the one with a thickness of about 100 μm . The surface hardness of the Ti coatings with thicknesses of about 1000 and 6200 μm are

Fig. 4 Surface hardness of Ti coatings as a function of thickness

Fig. 5 Hardnesses of Ti coatings with thicknesses of about (a) 100, (b) 700, (c) 1000, and (d) 6200 μm measured along their cross-sections. The gray lines represent respective coating/substrate interfaces and the positive values are “measured distances” for the Ti coatings from their respective interfaces

Fig. 6 Surface topographies of polished (a) Ti64 substrate and (b) Ti coating with a thickness of about 6200 μm

about 146 and 280 Hv, respectively, which imply that the higher thickness gives rise to the higher surface hardness of the Ti coatings due to the better uniformity and higher

density of the coatings (Ref 15, 16). In addition, the increased thickness suppresses a complication arisen from the Ti64 substrate for increasing the surface hardness of the Ti coatings.

Figure 5 shows the hardnesses of the Ti coatings measured along their cross-sections as a function of distance. For all the samples, the hardness of the Ti64 substrate is consistently higher than those of the Ti coatings, which is in agreement with the results presented in Fig. 4. Comparison of Fig. 4 and 5 shows that the surface hardnesses of the Ti coatings are higher than their cross-sectional hardnesses, especially for the thicker coatings, probably due to the more cold-worked structures in the direction of the impact during the cold spray deposition.

Figure 6 shows the surface topographies of the polished Ti64 substrate and Ti coating with a thickness of about 6200 μm . Comparison of Fig. 6(a) and (b) clearly shows that the Ti coating has a significant number of pores on its surface topography although such pores are not found on the surface topography of the Ti64 substrate. Therefore, the Ti coating has a larger R_q value of $0.38 \pm 0.01 \mu\text{m}$ than ($0.1 \pm 0.01 \mu\text{m}$) the Ti64 substrate.

The tribological properties of the Ti coatings of different thicknesses were investigated by sliding them

Fig. 7 Friction coefficients of Ti coatings, tested against a 100Cr6 steel ball of 6 mm in diameter in a circular path of 1 mm in radius for 30,000 laps at a sliding speed of 3 cm/s under a normal load of 1 N, as functions of (a) thickness and (b) the number of laps

Fig. 8 Specific wear rate of Ti coatings tested under the same conditions as described in Fig. 7 as a function of thickness

against a steel ball for 30,000 laps at a sliding speed of 3 cm/s under a normal load of 1 N. Their friction coefficients are presented in Fig. 7(a). The average friction coefficients of the Ti coatings with thicknesses of about 100, 700, 1000, and 6200 μm are about 0.72, 0.74, 0.71, and 0.61, respec-

tively. It is supposed that the increased thickness could result in a slightly decreased friction of the Ti coatings through their decreased wear since a larger contact area between two rubbing surfaces via higher surface wear gives rise to a higher friction (Ref 4). In addition, the opposite trends between the hardness (Fig. 4) and friction (Fig. 7a) of the Ti coatings with respect to thickness imply that the increased hardness of the Ti coatings with increased thickness gives rise to their decreased friction via their reduced contact with the steel balls (Ref 4).

As shown in Fig. 7(a), the friction coefficients of the Ti coatings are apparently higher than that of the commercial Ti64 substrate, which is confirmed by the lower friction coefficient (about 0.55) of the Ti64 substrate than those of the Ti coatings. Normally, an adhesion between two smooth surfaces in contact can generate a high friction via an effective interfacial shear strength between them (Ref 4, 17-19). Roughening of rubbing surfaces via surface wear can lessen the interfacial shear strength by reducing a real contact area between them and subsequently decrease the friction (Ref 20, 21). In addition, wear debris generated via surface wear can be released to an interface between two rubbing surfaces so that the wear debris decrease the interfacial shear strength by serving as spacers between the rubbing surfaces and freely rolling or sliding under a lateral load and result in a subsequent decrease in the friction (Ref 20, 21). It can be understood that the significantly higher wear of the Ti64 substrate than that of the Ti coatings gives rise to the lower friction via the higher roughening of rubbing surfaces and larger amount of wear debris. This finding is in agreement with the results reported by Khun et al. (Ref 4, 22).

Figure 7(b) shows the friction coefficients of the Ti64 substrate and Ti coatings of different thicknesses as a function of the number of laps. The Ti64 substrate has a significant increase in the friction with increased laps at the beginning as a result of the promoted initial wear of rubbing surfaces and then a stable friction for the rest. It is clear that the Ti64 substrate exhibits a stable friction throughout the wear test due to its stable wear. As shown in Fig. 7(b), the frictional behaviors of the Ti coatings with thicknesses of about 100, 700, and 1000 μm are apparently different from that of the Ti64 substrate. At the early stage, these Ti coatings exhibit an increased friction with increased laps and a decreased friction before reaching a stable condition for a certain number of laps. Then, their friction significantly increases with further increased laps as a result of the promoted surface oxidation process and dramatically decreases again because the formed surface oxide layers are somewhat harder and decrease the friction by lessening further wear of rubbing surfaces, and reducing a contact area and preventing a direct contact between them (Ref 23). Eventually, they reach a stable condition for the rest. The Ti coating with a thickness of about 6200 μm does not exhibit such frictional behavior during the test. It is, however, supposed that the lowest wear of the Ti coating with a highest thickness of about 6200 μm results in an earliest formation of surface oxide layers on its wear track during the rubbing against the steel ball so that a relatively stable friction is found

Fig. 9 Surface morphologies (above) and topographies (below) of (a) Ti64 substrate and Ti coatings with thicknesses of about (b) 100, (c) 1000, and (d) 6200 μm tested under the same conditions as described in Fig. 7

throughout the wear test till the oxide layers are broken down for a significant increase in the friction at the end of the test. It clearly suggests that the formation of the high-wear-resistant surface oxide layers significantly contributes to the lower wear of the Ti coatings than that of the Ti64 substrate.

The specific wear rate of the Ti coatings as a function of thickness is presented in Fig. 8. The Ti coatings with thicknesses of about 100, 700, 1000, and 6200 μm have specific wear rates of about 18.9×10^{-14} , 22.4×10^{-14} , 17.6×10^{-14} , and 10.7×10^{-14} m^3/Nm , respectively. It is found that the wear of the Ti coatings decreases with increased thickness, which in turn decreases the friction of the coatings via the decreased contact between the steel balls and the coatings. The similar trends between the friction (Fig. 7a) and wear (Fig. 8) of the Ti coatings with respect to thickness indicate that the frictional behavior of the Ti coatings is closely related to their wear behavior.

The much higher specific wear rate (about 93.9×10^{-14} m^3/Nm) of the Ti64 substrate than those of the Ti coatings (Fig. 8) clearly indicates that the cold-sprayed Ti coatings have the higher wear resistance than the commercial Ti64 substrate.

Figure 9 shows the wear morphologies of the Ti64 substrate and Ti coatings of different thicknesses. Comparison of Fig. 9(a)-(d) shows that the Ti64 substrate has a larger wear track than the Ti coatings, confirming that the Ti coatings have the higher wear resistance. In addition, it can be confirmed that the apparently rougher wear morphology and significantly larger amount of wear debris on the wear track of the Ti64 substrate (Fig. 9a) are responsible for its lower friction coefficient compared to those of the Ti coatings (Fig. 7a). Abrasive lines on the wear track of the Ti64 substrate is indicative of the abrasive wear caused by the repeated sliding of the steel ball (Ref 4, 19, 22, 23). As shown in Fig. 9(b)-(d), the Ti coating with higher thickness has a smaller wear track, which is in

agreement with the wear results presented in Fig. 8. Tribolayers are clearly found on the wear tracks of all the Ti coatings (Fig. 9b-d), which are formed by compacting the wear debris on the wear tracks due to the repeated sliding of the steel balls on the wear tracks (Ref 4, 22-27). The tribolayers are not apparently found on the wear track of the Ti64 substrate because the much higher wear of the Ti64 substrate does not allow the formation of the tribolayers during the wear test.

Figure 10 illustrates the EDX spectra measured on the untested and tested areas of the Ti64 substrate and Ti coatings of different thicknesses. In Fig. 10(a), the EDX spectrum measured on the untested area of the Ti64 substrate contains C (6.56 at.%), Al (7.78 at.%), Ti (82.52

at.%), and V (3.14 at.%) peaks. It is clear that the Ti, Al, and V peaks result from the Ti64 matrix as the C peak comes from surface carbon contaminant. The EDX spectrum measured on the wear track of the Ti64 substrate (Fig. 10b) has an additional O peak (3.11 at.% C, 20.92 at.% O, 3.65 at.% Al, 70.42 at.% Ti, and 1.9 at.% V), which implies that an oxidation process has occurred during the wear test (Ref 23).

In Fig. 10(c), the EDX spectrum measured on the untested area of the Ti coating with a thickness of about 100 μm is composed of Ti (95.47 at.%) and C (4.53 at.%) peaks. Although the XPS enables to detect surface oxygen contaminant as found in Fig. 3, the EDX is not able to detect it. The possible reason is that the amount of ad-

Fig. 10 EDX spectra of (a and b) Ti64 substrate and Ti coatings with thicknesses of about (c and d) 100 and (e) 6200 μm measured on their (a and c) untested and (b, d and e) tested areas at locations A and B in Fig. 9(a), locations C and D in Fig. 9(b), and location E in Fig. 9(d)

Fig. 11 OM images showing wear morphologies of counter steel balls tested against (a) Ti64 substrate and (b) Ti coating with a thickness of about 6200 µm under the same conditions as described in Fig. 7

sorbed O is not high enough to discriminate overlapping of the O and Ti peaks on the EDX spectrum so that the Ti and C peaks can only be seen on the EDX spectrum. However, the EDX spectra measured on the wear tracks of the Ti coatings with thicknesses of about 100 µm (7.84 at.% C, 60.06 at.% O, 4.22 at.% Ti, 0.4 at.% Cr, and 27.48 at.% Fe) and 6200 µm (5.53 at.% C, 63.78 at.% O, 9.85 at.% Ti, 0.31 at.% Cr, and 20.53 at.% Fe) have several additional peaks for Fe, Cr, and O elements as shown in Fig. 10(d) and (e). The Fe and Cr peaks probably result from the wear of the steel ball. The EDX spectra measured on the wear tracks of the Ti coatings do not significantly change with thickness. Comparison of Fig. 10(b), (d), and (e) clearly shows that the EDX spectra measured on the wear tracks of the Ti coatings (Fig. 10d and e) have much stronger O peaks than the one obtained from the wear track of the Ti64 substrate (Fig. 10b), which is indicative of the occurrence of much more severe oxidation process during the wear test. In addition, the relatively depressed Ti peaks on the EDX spectra measured on the wear tracks of the Ti coatings (Fig. 10d and e) can be correlated to a significant coverage of oxide layers on the wear tracks of the coatings, which supports the claim that the formation of high-wear-resistant oxide layers is responsible for the lowered wear of the Ti coatings than that of the Ti64 substrate. The Fe peaks on the EDX spectra measured on the wear tracks of the Ti coatings (Fig. 10d and e) and the lack of the Fe peak on the EDX spectrum measured on the wear track of the Ti64 substrate (Fig. 10b) clearly indicate that the higher wear resistance of the Ti coatings results in the higher wear of their counter steel balls.

Figure 11(a) and (b) show the wear morphologies of the counter steel balls tested against the Ti64 substrate and Ti coating with a thickness of about 6200 µm, respectively. The steel ball tested on the Ti coating has much higher wear than the one rubbed on the Ti64 substrate as a result of the higher wear resistance of the Ti coating (Ref 28). In addition, more significant abrasive lines on the worn surface of the steel ball tested against the Ti coating is an indication of its more severe abrasive

wear. It can be deduced that the wear resistance of the Ti coatings significantly influences the wear of their counter steel balls.

4. Conclusions

In this study, the Ti coatings of different thicknesses were deposited on the commercial Ti64 substrates via a high-pressure cold spray process. The mechanical and tribological properties of the Ti coatings were systematically investigated. The hardness of the Ti coatings significantly increased with increased thickness probably due to the promoted uniformity and density of the coatings and the lessened complication arisen from the substrates. But, the Ti coatings had the relatively low hardness probably due to the high porosity levels of the coatings. The tribological results clearly showed that the wear resistance of the Ti coatings tested against the steel ball was significantly higher than that of the Ti64 substrate due to the cold work hardening caused by the cold spraying and the formation of high wear-resistant oxide layers on the wear tracks of the coatings during the wear tests. The wear resistance of the Ti coatings was enhanced with thicker coating as a result of the improved hardness of the coatings. It could be concluded that the cold spray process could produce the higher wear-resistant Ti coatings as the mechanical and tribological properties of the cold-sprayed Ti coatings were apparently influenced by the thickness.

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