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TRIBOLOGICAL BEHAVIOR OF OIL-LUBRICATED, TiN-COATED STEEL*

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Abstract

The effects of titanium nitride (TiN) coatings on the tribological behavior of M50 and 52100 steels under both dry and synthetic polyol ester-based oil lubrication were evaluated using a reciprocating sliding pin-on-flat test machine. Under dry conditions, the TiN coating reduced the wear, which occurred by abrasion as well as the oxidation of the sliding surface. It also reduced the amount of wear-debris accumulation at the contact interface. During oil lubrication, wear and roughening of the contact area, usually associated with the boundary lubrication regime, was eliminated by the TiN coating. Formation of boundary film by the chemical interaction between the oil additives and wearing surface was also prevented by the TiN coating.

Introduction

The use of a hard ceramic coating to improve the performance and prolong the useful life of cutting tools and other metal-working and shaping tools is now a common practice in industries. Several kinds of coatings are currently being used and many others are being developed [e.g. 1]. Perhaps one of the most commonly used ceramic coating material is TiN. Various deposition methods, such as physical vapor deposition (PVD), chemical vapor deposition (CVD), ion-beam-assisted deposition (IBAD), etc., are being employed to produce a durable, well-adhering coating.

In the recent past, the use of hard ceramic coatings has expanded beyond tool applications. Studies have shown that the rolling-contact-fatigue (RCF) life of bearings could be significantly improved by a hard coating [2,3]. In fact, these coatings are now increasingly being used in bearing applications.

Other prospective areas of tribological applications for hard coatings are those that involve sliding contact. Several studies have evaluated the sliding friction and wear behavior of coated systems usually under dry conditions [e.g. 4-7]. In practice, most applications involving sliding contacts, however, involve liquid lubrication. Only limited studies have been done to evaluate the tribological characteristics of lubricated coated systems in sliding contact [8,9]. The present study investigates the oil-lubricated behavior of TiN coatings on AISI M50 and 52100 steel substrates.

Experimental Details

Two steel materials that are currently being used extensively for tribological applications were used in the study. These are the tool steel AISI M50 and bearing steel AISI 52100. Several flat specimens of nominal dimensions 50 x 25 x 6 mm and cylindrical pins of about 9 mm diameter and 15 mm length were made from each of the two types of steel. Both sides of the flat specimens were ground and polished to a surface finish of 0.035 μm R_a. One end of the pins was rounded to a radius of curvature of 127 mm and polished to a finish of 0.08 μm R_a.

One side of the flat specimens was coated with TiN using a high rate reactive sputtering deposition method, details of which have been previously described [10-12]. Coating deposition was done in an MRC 902 M magnetron sputtering system using a VP-grade titanium target. Two micrometer (2μm)-thick coatings were deposited at a rate of approximately 0.48 μm/minute at 10

kW power, a substrate bias of -100V and at a total pressure of 8 mTorr with a N_2 partial pressure of 0.17 mTorr. The TiN coatings produced under these conditions have a dense columnar structure with a Vickers hardness of 2200-2400 kgf mm^{-2}. The surface roughness of the coatings was measured to be 0.038 μm R_a, thus the coating process did not significantly change the surface roughness.

Friction and wear tests were done using a pin-on-flat contact configuration in reciprocating sliding. A detailed description of the test device has been previously given [13]. Tests were conducted by sliding uncoated pins on both TiN-coated and uncoated flat specimens under dry and oil-lubricated conditions. The lubricated tests were done with the flat specimen immersed in a commercial synthetic polyol-ester-based oil during the test, thereby creating a fully flooded condition. The oil is fully formulated with a proprietary blend of additives. All the dry tests were done with a normal load of 10N while the lubricated ones were done at 10N and 50N. Other test parameters include reciprocating sliding frequency of 1 Hz, stroke length of 25mm, creating an average sliding speed of about 0.05 m/s, ambient temperatures of room temperature (≈ 23°C) and 250°C. All tests were done for a total of 5000 cycles.

The friction coefficient was monitored continuously throughout the duration of each test. The amount of wear on the pin was estimated from the scar diameter measured by an optical microscope [13]. Tests were interrupted after 500, 1000, 2000, 3000, 4000 and 5000 cycles and the pin wear measured from which the "instantaneous" wear rate could be estimated. The wear in the flats was estimated by measuring the cross-sectional area of the wear track using a surface profilometer. All tested specimens were examined by both optical microscope and scanning electron microscope (SEM), equipped with energy dispersive spectrometer (EDS) and wavelength dispersive spectrometer

(WDS) capabilities. Both the damage and wear processes, as well as the chemistry of the surfaces, were analyzed.

Results

Friction: Figure 1a shows the frictional behavior of unlubricated sliding of an M50 steel pin on both uncoated and TiN-coated M50 steel flats. At room temperature, for the uncoated flat, sliding started with a friction coefficient of 0.7 but increased rapidly to a steady value of about 0.95 after about 100 cycles. With the TiN-coated flat, a similar trend of a quick rise in friction was observed, but with lower starting and steady friction coefficients of 0.3 and about 0.7 respectively. At 250°C, the steady friction with the uncoated flat dropped significantly to a value of 0.4 compared to 0.95 at room temperature. With the coated flat, however, a slight increase in friction was observed at 250°C when compared to the room temperature value (0.7 at room temperature vs. 0.85 at 250°C). The sliding frictional behavior for the 52100 steel with and without TiN coating during dry sliding is shown in Figure 1b. There was only a slight decrease in the steady friction coefficient with the uncoated flat at 250°C when compared with the room temperature value of about 0.5. With the coated flat, the steady friction coefficient at room temperature was about 0.65. At 250°C, the friction coefficient for the first 2200 cycles was about 0.5, but increased to a higher value of about 0.75 for the remainder of the test. This transition coincides with the wearing-through of the TiN coating in the central portion of the wear track.

With oil lubrication, the friction behavior in the types of steel was very similar. Figure 1c shows the typical characteristics. At room temperature the friction coefficient for M50 steel ranged in value from 0.02 for coated flats to about 0.025 for uncoated flats at 50N. For 52100 steel, the friction coefficient

ranged from 0.007 for the uncoated flat to about 0.01 for coated ones. At 250°C, the friction coefficient was significantly higher than the room temperature value in both the coated and uncoated cases. This was most likely due to the large decrease in the viscosity of the oil as the temperature increases, allowing more direct contact and interaction between the two sliding surfaces. For both coated and uncoated flats, sliding started with a relatively high friction coefficient, but decreased gradually to a lower steady value. Although the starting friction coefficient for M50 steel uncoated flat was higher than the coated flat (0.12 for coated and 0.25 for uncoated), the steady friction coefficients for both were about the same at 0.08. With 52100 steel, both the starting and the steady friction coefficient with the coated flat were lower than the ones for the uncoated flat (Figure 1c). However, by the end of the test both were approaching the same value of about 0.1. The higher friction at the early stage of the tests in both kinds of steel was probably due to a break-in process.

Wear: Figure 2 shows the "instantaneous" specific wear rate in the pins during various test conditions. For M50 steel (Figure 2a), the pin wear rates after sliding with coated and uncoated flats showed very similar behavior. The wear rate during the first 1000 cycles for dry sliding was of the order of 10^{-5} mm^3 N^{-1} m^{-1}, but decreased to a steady value on the order of 10^{-6} mm^3 N^{-1} m^{-1}. The initially higher wear rate was probably due to the higher nominal contact pressure at the early stage of the test. The only exception to this trend was the uncoated flat test at 250°C, in which the wear rate appeared to increase linearly after the initial decrease. With oil lubrication, the trends of the pin wear rates were similar to those of the unlubricated tests, but two to three orders of magnitude lower. When sliding against a TiN-coated, lubricated flat at 250°C, the pin showed peculiar wear behavior. After the initial drop in the wear rate following break-in (a value of about $3 \times 10^{-8} mm^3 N^{-1} m^{-1}$), it increased sharply to

a steady value of $10^{-6} mm^3 N^{-1} m^{-1}$ after 2000 cycles. The final value was comparable to the wear rate under dry sliding.

For 52100 steel pins (Figure 2b), the specific wear rate during dry tests was nearly constant at $10^{-5} mm^3 N^{-1} m^{-1}$, except when sliding against a coated flat at room temperature wherein the wear rate decreased over the first 3000 cycles from about 2×10^{-5} to about 2×10^{-6} $mm^3 N^{-1} m^{-1}$. When lubricated, the wear rate decreased by about three orders of magnitude, with more wear occurring in the pins that slid against coated flats. In addition, for both coated and uncoated cases, the wear rate at the early part of sliding was high but decreased to a steady value after about 1500 cycles.

The average wear rate over the duration of tests for the flat specimens is shown in Table 1. Under dry conditions, the wear rate in the coated and uncoated flats for M50 and 52100 steels was of the order of 10^{-5} - 10^{-6} mm^3/Nm. When lubricated, the wear in the uncoated flats was reduced to about 10^{-7} mm^3/Nm, while wear was unmeasurable in the coated flats. The wear tracks were identifiable, but no loss of material or significant surface damage was detected by both profilometry and microscopy.

Microscopy: Under dry conditions, the worn surface of the pin was rough with deep parallel scratches and grooves in the sliding direction. Detailed examination revealed that some locations in the worn area were covered by a layer of debris that was rich in oxygen and Fe according to EDS and WDS analysis. In addition, Ti was also detected in the debris on the pins that were rubbed against coated flats. With oil lubrication, the pin worn surface was not as rough as in the case of dry tests but some scratches in the sliding direction could also be seen (Figure 3). Closer examination showed no accumulation of debris, but oxygen was still detected by the WDS analysis.

The wear track on the uncoated flat tested under dry conditions showed the same characteristics as for the pin described above. Deep scratches and grooves were the main macro-features. At higher magnification, the presence of a debris layer rich in oxygen was observed. For the TiN-coated flats, the wear track was in general very smooth compared to the original coating surface. Upon closer examination, however, broad grooves and small debris accumulation were observed (Figure 4). The extent of debris accumulation on the coated-flat wear track was much less than for the uncoated one. Analysis showed that the debris contained Fe and oxygen in addition to Ti.

Worn surfaces of the flat specimens during oil lubrication are shown in Figure 5. In the uncoated flats, the formation of a new layer on the wear track was seen in Figure 5a. The EDS analysis showed the new surface layer is rich in S, which is probably one of the components of the additives in the oil. WDS analysis also showed that the new layer contains a significant amount of oxygen. For the TiN-coated flats, very little change was observed on the wear track except for a few scratches (Figure 5b). The EDS analysis showed only Ti and WDS analysis showed only N. This observation was consistent with the fact that no wear could be measured on any of the oil-lubricated, coated flats (Table 1). In addition, no chemical changes were observed.

Discussion

Some effects of TiN coatings on the tribological behavior of steel during both dry and oil-lubricated sliding contact were elucidated in the present study. Under dry conditions, the primary material removal mechanism involves abrasion, as evidenced by the scratches and grooves observed on the worn surfaces. Some of the debris generated accumulates and reattaches to the worn surface. This debris layer consists primarily of iron oxide when both of the

sliding couples are uncoated, and iron oxide and TiN when one of the couples is coated. Several other investigators have observed similar occurrences of debris accumulation during sliding contact of TiN-coated systems and the increase in friction has been attributed to generation and accumulation of debris [4-6]. Presumably the higher friction in the uncoated flat is due to more damage and accumulation of debris. Microscopy of the worn surfaces support this view. The exact reason for the large decrease in friction at 250°C for M50 steel is not understood yet, but it may be connected to increased oxidation of the surface at this temperature.

The extent of damage and amount of wear on the uncoated flat specimen was much greater than for the TiN-coated flat, as one would expect since the hardness of the TiN layer (≈ 2300 kgf mm^{-2}) is much higher than the hardness of the steels (870 kgf mm^{-2} for M-50 and 730 kfg mm^{-2} for 52100). Since abrasion is the primary material removal mechanism, increased hardness will reduce the amount of material removed. However, there was also more debris reattachment and accumulation for the uncoated flat than for the TiN-coated flats. This, we suggest, is the reason for the fact that the measured wear in both the coated and the uncoated flats was about the same under dry conditions (Table 1).

Another major difference between the coated and the uncoated surfaces during dry sliding was the oxidation of the worn surface. In all the uncoated sliding components, a significant amount of oxygen was detected on the worn surface and a visible change in color to dark brown from the initial metallic appearance was commonplace. For the TiN-coated component, no oxygen was detected, nor was there any change in appearance. Although oxidation of TiN to form TiO_2 in a TiN-on-TiN sliding system has been suggested [5], no such observation was made in the steel-on-TiN couples used in the present study.

The occurrence of a significant amount of wear in all the lubricated tests suggests that the tests were under boundary or mixed lubrication regimes. The extent of surface contact was increased even further at 250°C, due to the large decrease in the oil viscosity.

One of the major differences between the TiN-coated and the uncoated flats in the lubricated test was the change in the surface roughness of the wear track. For the uncoated flats, the interaction between the two surfaces led to roughening, resulting in even more intimate contact between the two surfaces and some wear on the flat. In the TiN-coated flats, however, the higher hardness of the TiN surfaces prevented any change in the roughness and there was no wear. Wear did occur in the mating pin surface, however.

Another major difference between coated and uncoated flats was the chemical change on the surface during oil lubrication. Chemical interaction between additives in the oil, oxygen from the environment and the steel surface of the uncoated sliding couples led to the formation of boundary film. This may be responsible for the lowering of the friction coefficient with time and also the reduction in the wear rate after the running-in in the early stage. With TiN coatings, however, there was no formation of boundary film due to lack of chemical interaction between the TiN and the additives in the oil. Differences in chemical interaction between the oil (or its contents) and the wearing surface is not limited to the oil used in the present study. A similar observation was made with perfluoropolyalkylether (PFPE) oils in which chemical interaction between the oil and Fe led to deterioration of the lubricant; with TiN coating no such interaction was observed, thereby prolonging the durability of the oil [14]. In the present study, the lack of boundary film formation on TiN-coated flats may be responsible for the slightly higher wear in the mating pin when compared to the ones sliding against the uncoated flat with boundary film.

A major difference between the dry and lubricated tests was the role of the generated wear debris. Although in some of the lubricated tests, wear occurred in both the pins and the flats, accumulation and reattachment of debris that was prevalent in the dry tests was not observed in any of the oil-lubricated tests. This was due to the mechanical flushing of the debris from the contact interface by the lubricant. This prevented the increase in the friction coefficient associated with the debris accumulation during dry tests, but instead the friction either remained steady or decreased after the wearing-in for all oil-lubricated tests.

Conclusion

Results of the present study showed that tribological behavior of steels is significantly influenced by TiN coatings both under dry and oil-lubricated (particularly under boundary or mixed lubrication) conditions. The high hardness of the coating reduced the abrasion of the surface during dry tests and prevented the usual surface roughening and wear associated with boundary lubrication. The TiN coating prevented oxidation of the wearing surface and reduced the extent of debris accumulation (compared with an uncoated surface) under dry sliding contact conditions. Also, a lack of chemical interaction between the coating surface and additives in the oil lubricant prevented the formation of boundary films. This could be a problem for an uncoated mating surface when the beneficial effect of the boundary film is missing. Mechanical flushing of the wear debris during oil-lubricated tests prevented the accumulation of wear debris on the surface.

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Material	Temperature	Load	Surface Treatment	Specific Wear Rate (mm^3/Nm) Dry	Lubricated
M50 Steel	Room	10N	Uncoated	1.4×10^{-5}	NMW*
			Coated	5.92×10^{-5}	NMW
		50N	Uncoated	**	1.6×10^{-7}
			Coated	-	NMW
	250°C	10N	Uncoated	4×10^{-6}	-
			Coated	4×10^{-5}	-
		50N	Uncoated	-	3.6×10^{-7}
			Coated	-	NMW
52100 Steel	Room	10N	Uncoated	NMW	NMW
			Coated	1.35×10^{-5}	NMW
		50N	Uncoated	-	2.4×10^{-7}
			Coated	-	NMW
	250°C	10N	Uncoated	7×10^{-5}	-
			Coated	2×10^{-5}	-
		50N	Uncoated	-	1.04×10^{-6}
			Coated	-	NMW

*NMW means no measurable amount of wear occurred
** - means no tests were done under such conditions

Table 1: Average specific wear rates in the flat specimen

Captions for Figures

Figure 1: Variation of the friction coefficient with the number of sliding cycles for steel pins sliding on TiN-coated and uncoated flats (a) M50 under dry conditions, (b) 52100 under dry conditions, and (c) 52100 under oil-lubricated conditions.

Figure 2: Variation of the "instantaneous" specific wear rate with the number of sliding cycles for (a) M50 steel pins and (b) 52100 steel pins.

Figure 3: SEM micrograph of a typical pin worn surface tested under oil-lubricated conditions.

Figure 4: SEM micrograph of a typical worn surface under dry conditions for TiN-coated surface showing limited debris accumulation.

Figure 5: (a) SEM micrograph of uncoated flat tested under lubricated conditions showing formation of boundary film, (b) SEM micrograph of TiN-coated flat tested under oil-lubricated conditions.

Figure 1a

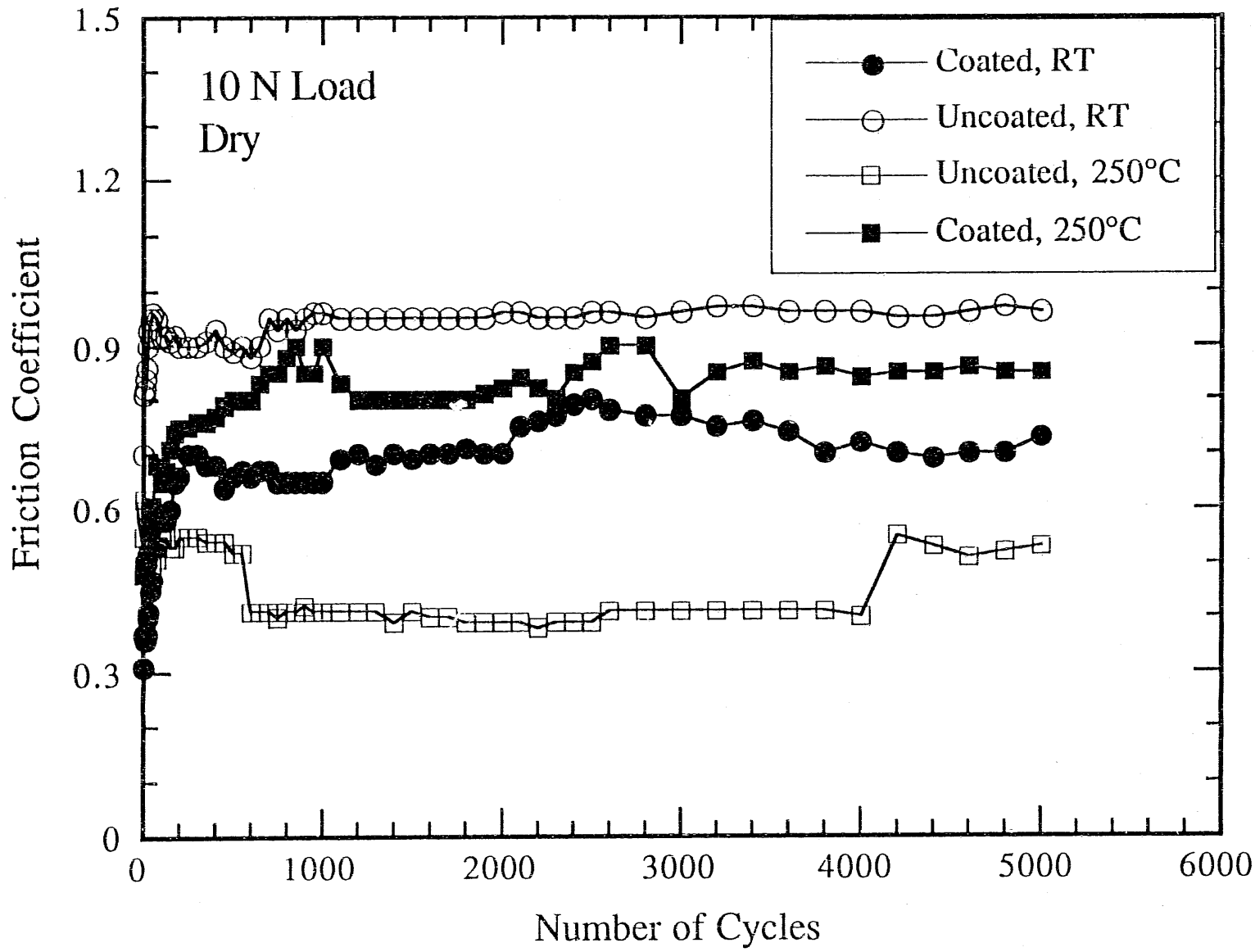

10 N Load
Dry
Coated, RT
Uncoated, RT
Uncoated, 250°C
Coated, 250°C
Friction Coefficient
1.5
1.2
0.9
0.6
0.3
0
0
1000
2000
3000
4000
5000
6000
Number of Cycles

Figure 1b

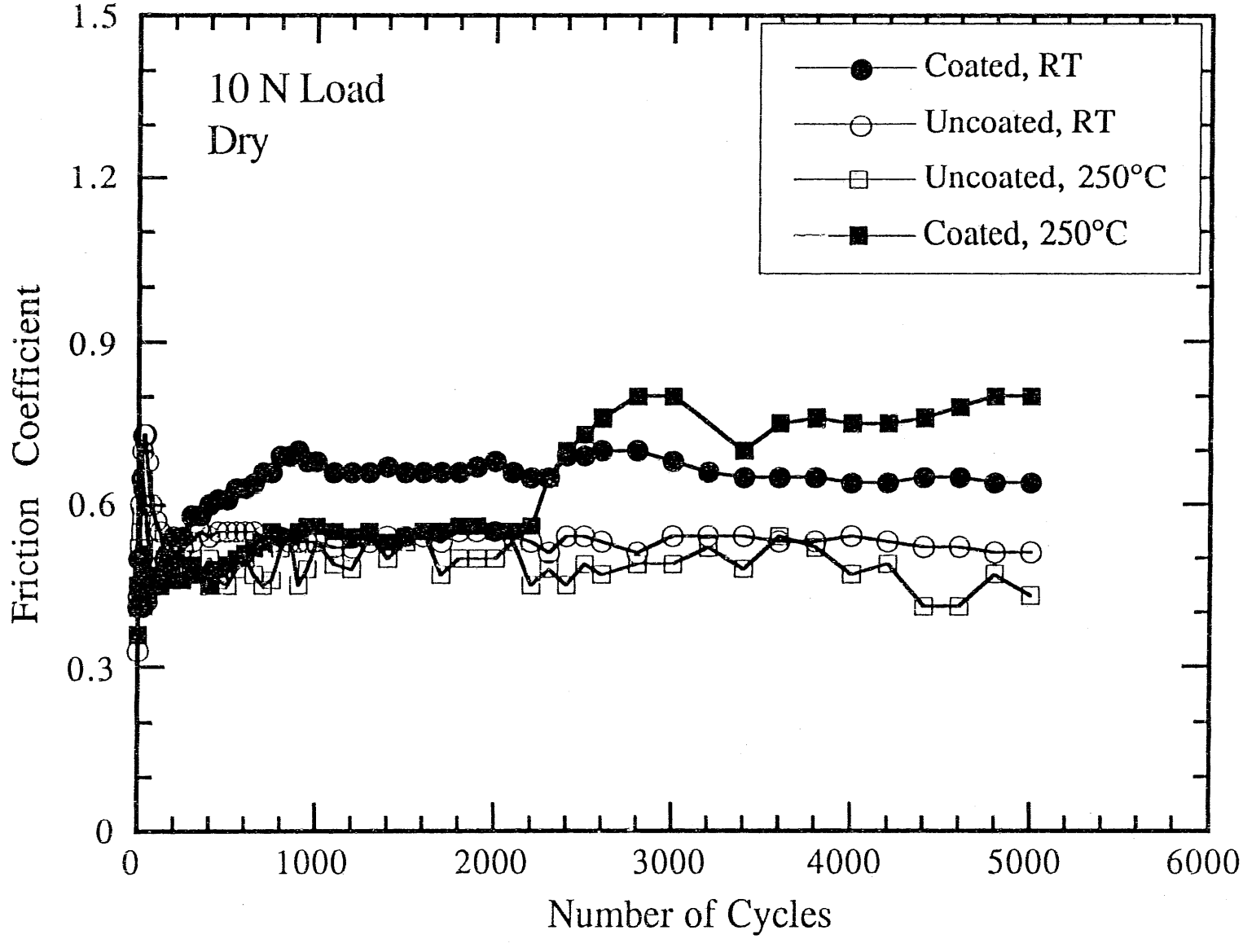

10 N Load
Dry
Coated, RT
Uncoated, RT
Uncoated, 250°C
Coated, 250°C
Friction Coefficient
1.5
1.2
0.9
0.6
0.3
0
0
1000
2000
3000
4000
5000
6000
Number of Cycles

Figure 1c

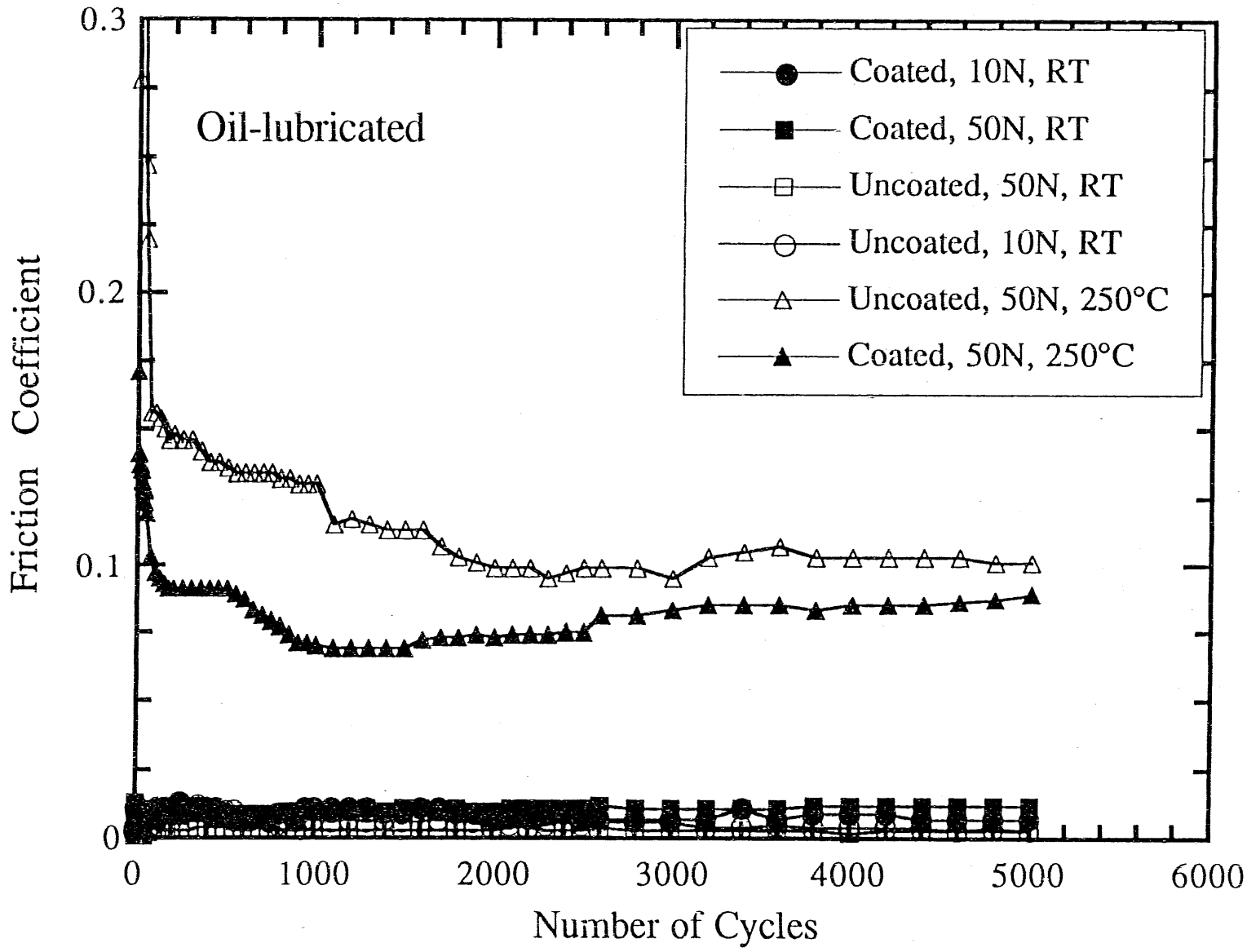

Oil-lubricated
Coated, 10N, RT
Coated, 50N, RT
Uncoated, 50N, RT
Uncoated, 10N, RT
Uncoated, 50N, 250°C
Coated, 50N, 250°C
Friction Coefficient
0.3
0.2
0.1
0
0
1000
2000
3000
4000
5000
6000
Number of Cycles

Figure 2a

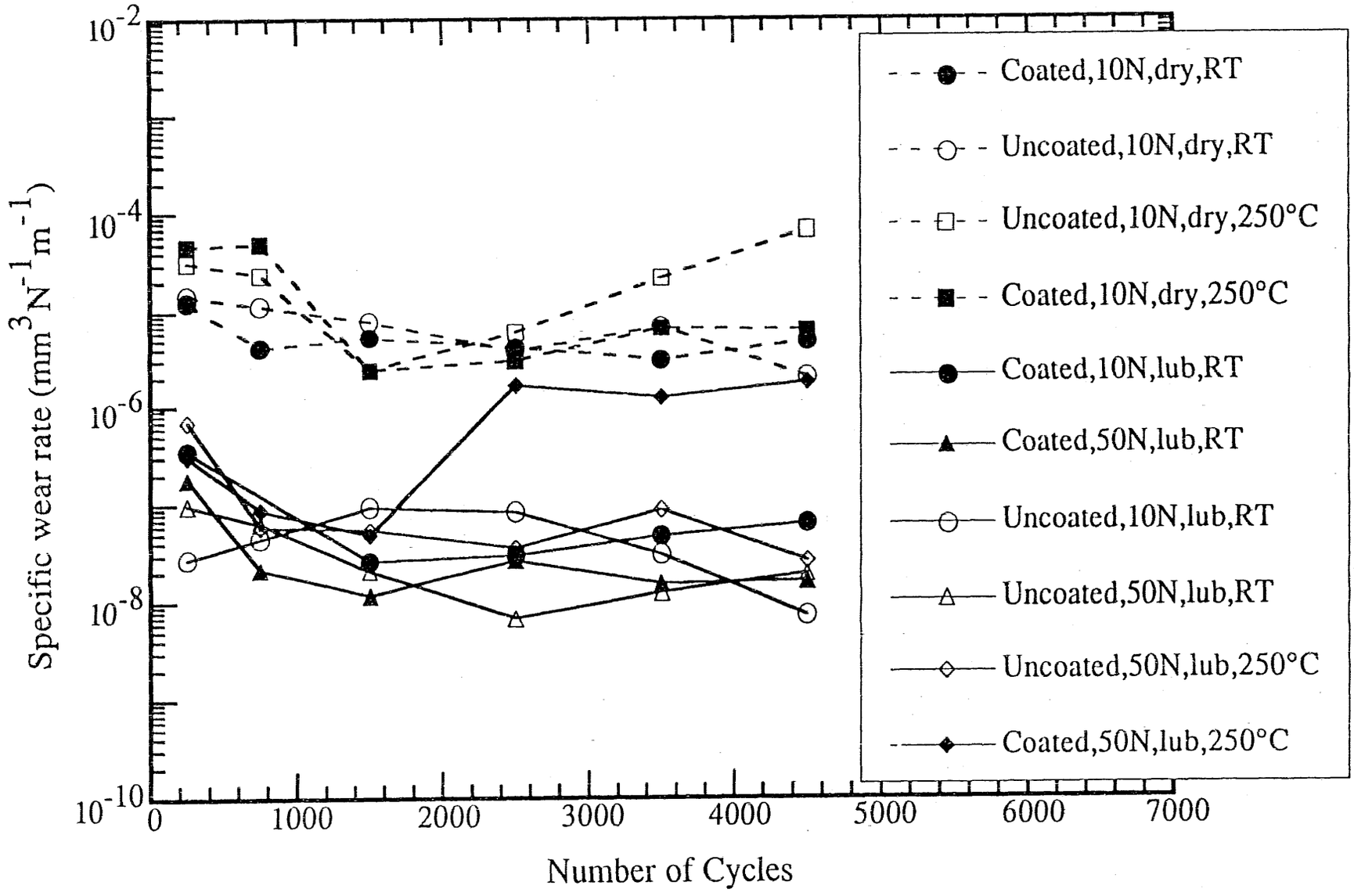
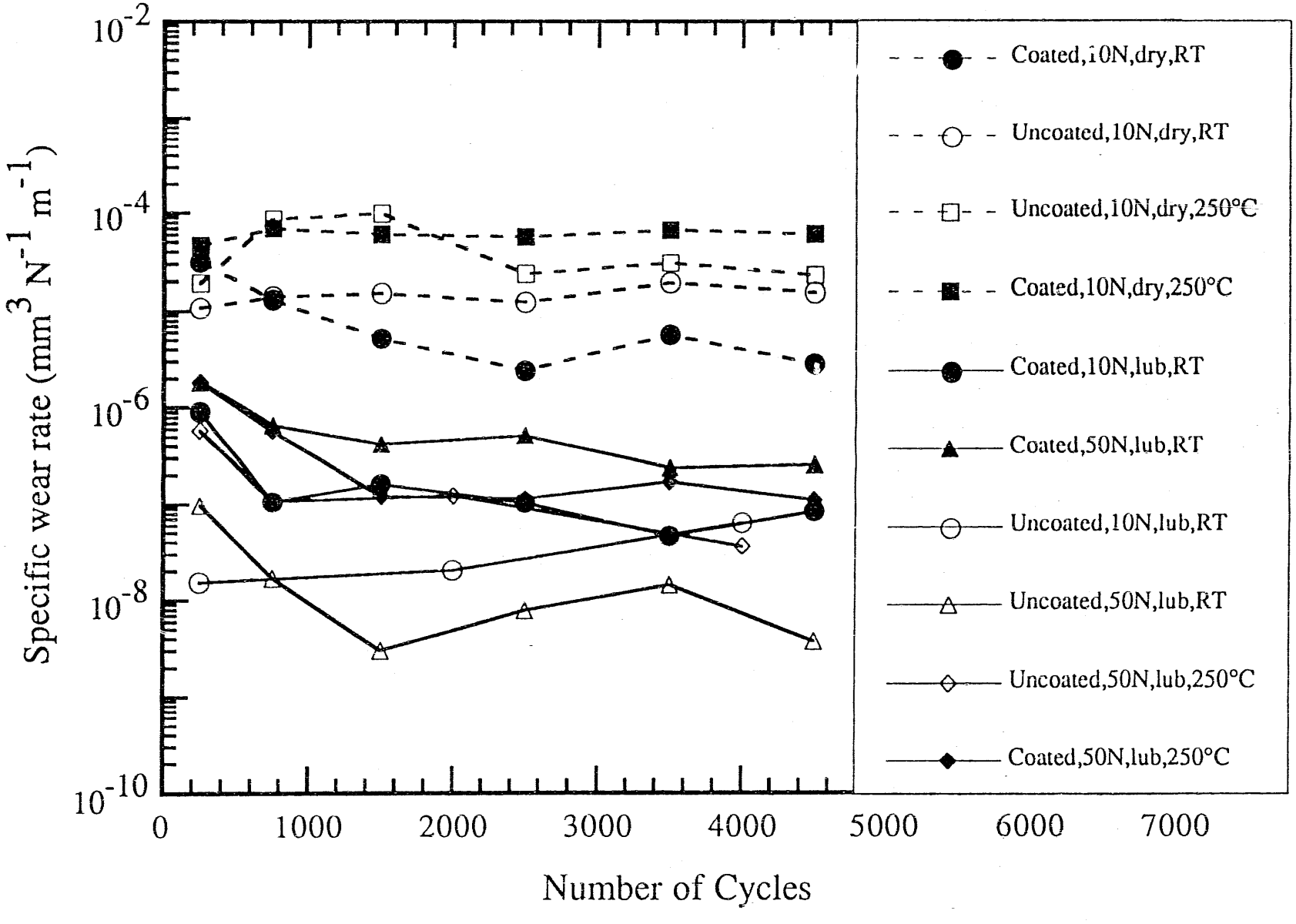

Specific wear rate ($mm^3N^{-1}m^{-1}$)
10^{-2}
10^{-4}
10^{-6}
10^{-8}
10^{-10}
0
1000
2000
3000
4000
5000
6000
7000
Number of Cycles
Coated,10N,dry,RT
Uncoated,10N,dry,RT
Uncoated,10N,dry,250°C
Coated,10N,dry,250°C
Coated,10N,lub,RT
Coated,50N,lub,RT
Uncoated,10N,lub,RT
Uncoated,50N,lub,RT
Uncoated,50N,lub,250°C
Coated,50N,lub,250°C

Figure 2b


Specific wear rate (mm3 N-1 m-1)
10-2
10-4
10-6
10-8
10-10
0
1000
2000
3000
4000
5000
6000
7000
Number of Cycles
Coated,10N,dry,RT
Uncoated,10N,dry,RT
Uncoated,10N,dry,250°C
Coated,10N,dry,250°C
Coated,10N,lub,RT
Coated,50N,lub,RT
Uncoated,10N,lub,RT
Uncoated,50N,lub,RT
Uncoated,50N,lub,250°C
Coated,50N,lub,250°C

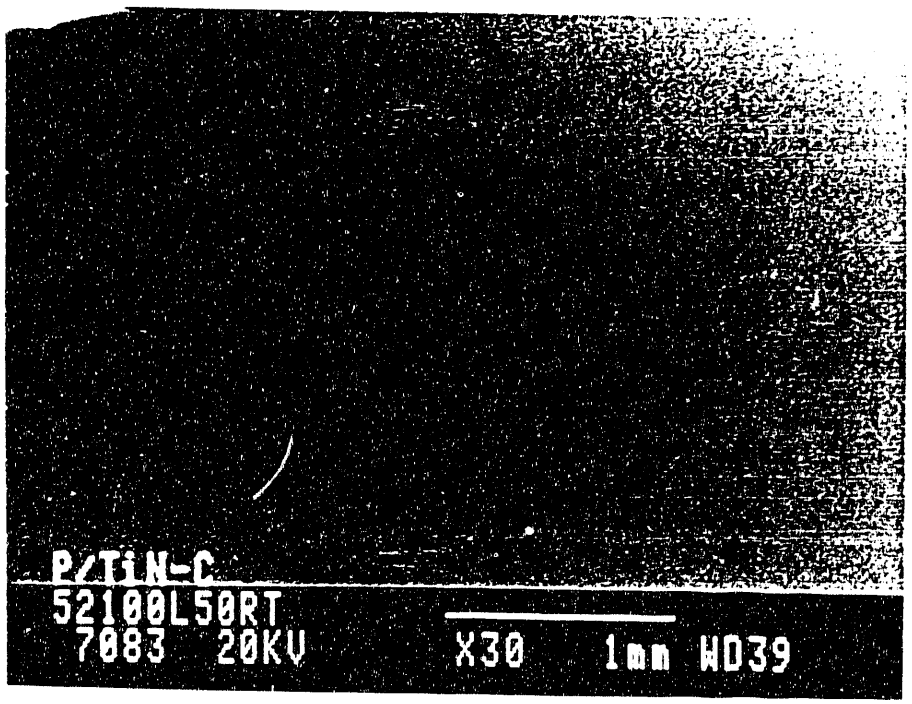


Figure 3

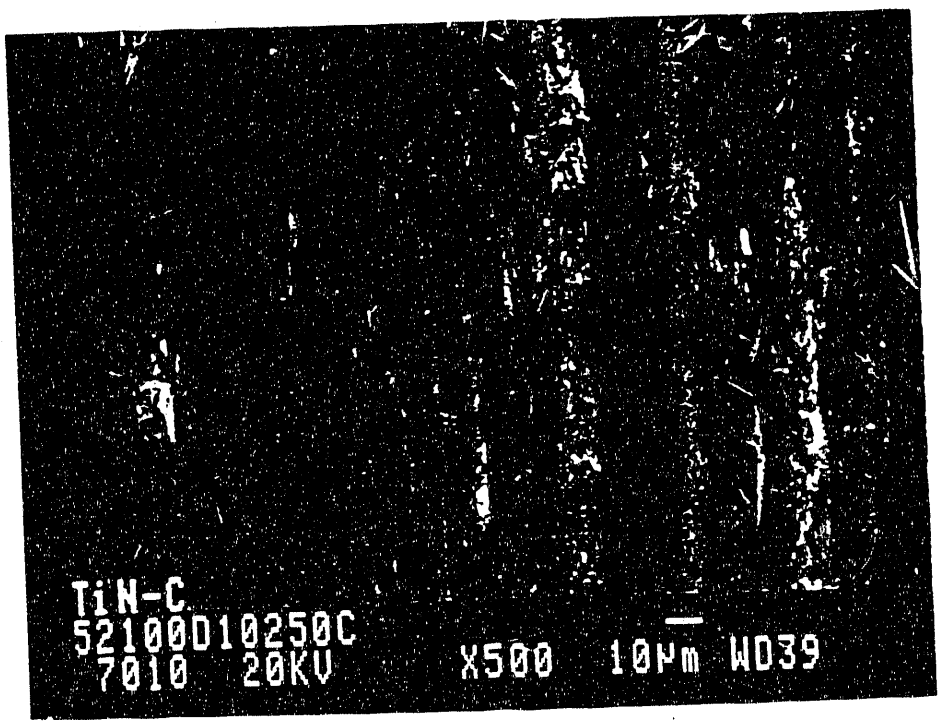
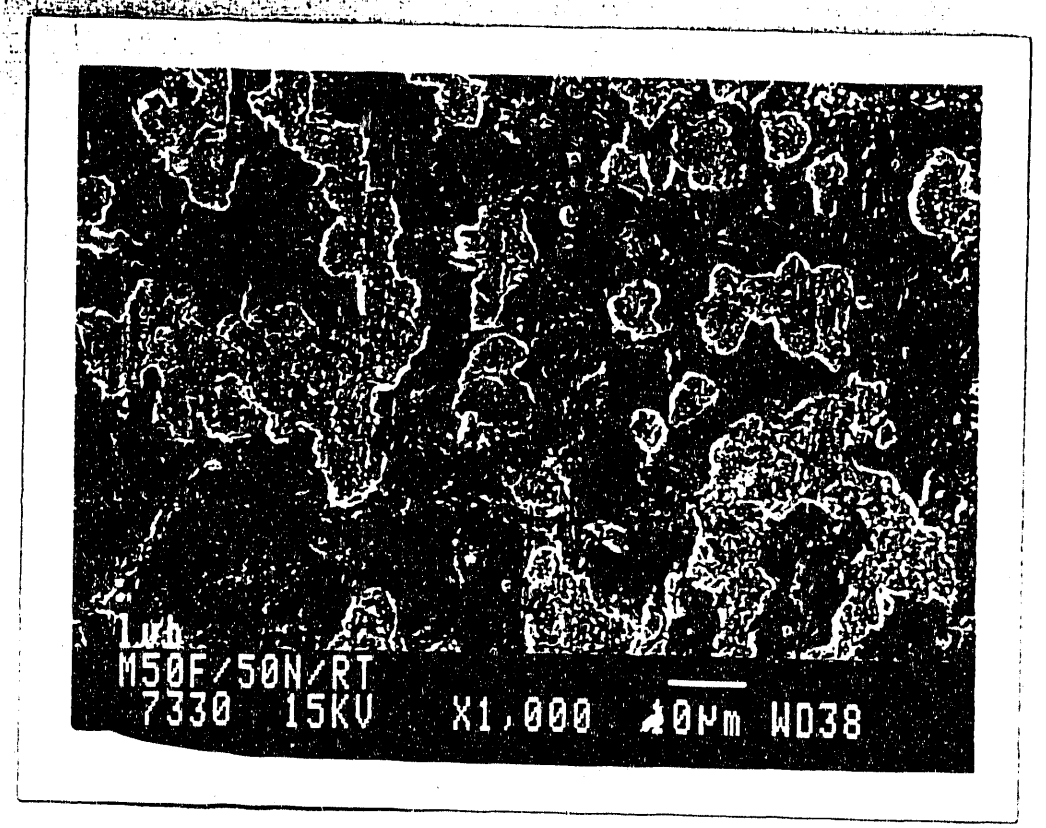
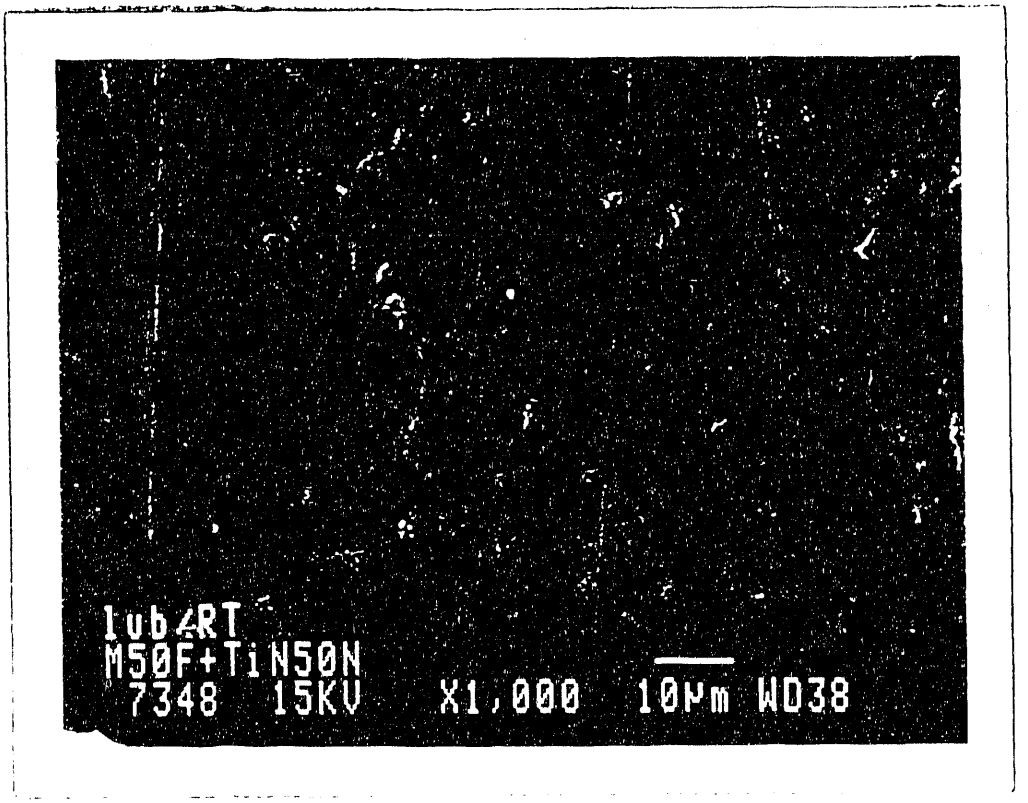


Figure 4


M50F/50N/RT
7330 15KV X1,000 10µm WD38

(a)


lub/RT
M50F+TiN50N
7348 15KV X1,000 10µm WD38

(b)

Figure 5

END

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