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Friction and wear behaviour of ion beam modified ceramics

J. LANKFORD, W. WEI
Southwest Research Institute, San Antonio, Texas 78284, USA
R. KOSSOWSKY
Pennsylvania State University, State College, Pennsylvania 16804, USA

In the present study, the sliding friction coefficients and wear rates of carbide, oxide, and nitride materials for potential use as sliding seals (ring/liner) were measured under temperature, environmental, velocity, and loading conditions representative of a diesel engine. In addition, silicon nitride and partially stabilized zirconia discs were modified by ion mixing with TiNi, nickel, cobalt and chromium, and subsequently run against carbide pins, with the objective of producing reduced friction via solid lubrication at elevated temperature. Unmodified ceramic sliding couples were characterized at all temperatures by friction coefficients of 0.24 and above. However, the coefficient at 800° C in an oxidizing environment was reduced to below 0.1, for certain material combinations, by the ion implantation of TiNi or cobalt. This beneficial effect was found to derive from lubricious titanium, nickel, and cobalt oxides.

1. Introduction

Improvements in low heat rejection engines require components to endure contact at temperatures considerably in excess of those attainable by current metal alloys. The obvious candidate materials for fulfilling these needs are ceramics, but only as part of an integrated ceramic/lubricant system, since conventional liquid lubricants break down at temperatures above a few hundred degrees Centigrade. The objective of this paper is to describe the results of a study aimed at establishing the feasibility of high-temperature, self-lubricating metal ion-implanted ceramics.

In recent years, numerous investigators have explored the friction and wear of ceramic–ceramic sliding pairs [1–3]. Couples exhibiting minimum wear can be identified, with high wear resistance usually being associated with cases where material transfer occurred, leading to the *in situ* formation of lubricating media [3–9]. However, wear is never negligible, and the unlubricated sliding friction coefficient is usually discouragingly high, i.e., $\mu_F \gtrsim 0.2$ [1–15]. It has generally been concluded that ceramic components will not be used unlubricated in sliding contact engine applications at either low [1] or elevated [2] temperatures.

While this is a discouraging conclusion, especially in light of the breakdown of conventional lubricants at elevated temperature, certain other related work suggests alternative approaches to the problem. In particular, the normally observed ceramic wear mechanisms, adhesion [16] and abrasion [17], can be mitigated by environmental factors. As early as 1964, it was observed [18] that adsorbed water, for example, can promote near-surface plasticity, leading to a soft surface layer some 1 to 10 μm thick. This soft surface layer seems to prevent adhesion and to lubricate ceramics as they slide over each other. For alumina sliding against alumina, it was found that the coefficient of friction rose from 0.25 to 0.8 as the temperature of friction experiments was raised from room temperature to 400° C, driving off adsorbed water; introduction of moist air then produced an immediate reduction of μ_F. Adsorbed organic substances also tend to lubricate and soften the surface of ceramics. Myristic acid, for instance, enhances surface plasticity and suppresses cracking during sliding of steel on lithium fluoride [18]. Chemisorbed oxygen appears to reduce the friction coefficient of titanium carbide by one-half [19].

Particularly pertinent to the present study is the work of Shimura and Tsuya [4], who investigated atmospheric and temperature effects on the friction and wear of several ceramics, including tungsten carbide, titanium carbide, chromium carbide, aluminium oxide, and their cermets. It was found that wear rates and coefficients of friction are higher in vacuum than in air. Based on characterization of the wear surfaces, the results were explained in terms of a postulated thin, soft lubricating surface layer which is formed by interaction of the ceramic/cermet with adsorbed moisture.

Similarly, Fischer and Tomizawa [6] studied the friction and wear of hot pressed Si_3N_4 in various environments. The coefficients of friction of Si_3N_4 sliding on itself at room temperature were measured in the range of 0.85 for dry environments, to 0.7 for humid environments. There were, however, marked differences in the wear rates. In dry atmospheres, wear

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rates were measured at $40 \times 10^{-10}\,\mathrm{kg\,Nm^{-1}}$, dropping two orders of magnitude to $0.4 \times 10^{-10}\,\mathrm{kg\,nm^{-1}}$ in wet environments. The high wear rates were associated with surface spallations which were initiated by high contact stresses at asperities. Wear in the humid environments was dominated by tribochemical reactions which produced amorphous, water soluble, lubricating layers. Fischer and Tomizava [6] pointed out also that the amorphous layers were soft, and thus provided for distribution of contact stresses over larger areas.

Likewise, Sutor [7] investigated the sliding wear of stainless steel against hot pressed Si_3N_4 in air at temperatures to 540° C. He observed progressive increases in wear rates of the steel component as load and velocity increased. Wear was associated with spallation and re-oxidation of the metal surface. The author noted concomitant increased transfers of metal (oxide) to the ceramic component, accompanied by noted reductions in the coefficients of friction. The latter were attributed to the formation of a lubricating glaze.

Finally, Kramer [8] studied the friction and wear properties of two forms of SiC and two forms of Si_3N_4 sliding against themselves under unlubricated conditions. Coefficients of friction were correlated with changes in surface chemistries at the sliding interfaces. NC 132 silicon nitride and NC 430 silicon carbide exhibited up to factors of two reductions in the coefficient of friction with increases in the sliding velocity. These correlated with substantial increases in the levels of oxygen measured on the surfaces. Kramer attributed these phenomena to the formation of SiO_2 due to the increased oxidation rates of free silicon present in the materials.

These results suggest that the intentional introduction of lubricious solid films, especially metal oxides, might provide ceramics with the ability to slide efficiently at elevated temperature. Delivery of such films, however, is a problem. Many ceramic coatings tend to fail via adhesion and delamination, so that any beneficial aspects of their presence are short-lived. However, Hirano and Miyake [20] have recently shown that adhesion during sliding contact may be reduced by ion implantation of both metal and inert ions. If such layers were, in addition, of a lubricious nature, they might be induced to provide transfer lubrication [21], i.e. continuous transfer of solid lubricant from one body to another during sliding contact. Ion implantation can, potentially, provide such layers, and by providing an intimate mix of implanted species and substrate, can produce a harder [22], tougher [23] surface, extremely resistant to lateral cracking and adhesive delamination [24]. Further, ion implantation provides a very thin "coating", so that well prepared surfaces maintain their original tolerance and finish.

With this idea in mind, an investigation was conducted on the sliding friction and wear behaviour of ion implanted ceramics. Simple ceramic–ceramic couples nominally compatible with the service requirements for seals (piston rings and cylinder liners) in a low heat rejection diesel engine were run in 3-pin-on-disc tests over a wide temperature range in both inert and simulated diesel exhaust environments, where the pins simulated moving piston rings, and the discs stationary cylinder liners. After evaluation of friction coefficients and wear mechanisms, similar specimens were ion-implanted with carefully chosen metal ion species, tested in the diesel exhaust environment at elevated temperature, and again evaluated.

2. Experimental factors and procedures

2.1. Material selection

The choice of ceramic materials for use in this investigation was based on the results of several studies [3, 25–29] which strongly suggest that certain carbides are the outstanding candidates for high temperature piston rings. In particular, hot-pressed TiC, Ni–Mo bonded TiC cermets [3, 26–29], and sintered SiC [28, 29] have demonstrated favourable wear resistance and (relatively) low friction coefficients. Accordingly, these were selected as pin materials for the present experiments. Similarly, Si_3N_4, SiC, and partially stabilized zirconia (PSZ) seem to be the most likely cylinder liner materials [29]. Because of the extensive body of knowledge available regarding the former, and the attractive mechanical strength properties and minimal thermal conductivity of the latter, hot pressed Si_3N_4 and zirconia were selected as disc materials. Properties of the bulk ceramics selected for testing are given in Table I.

Metal species for ion implantation included chromium, cobalt, nickel and a double layer of nickel and titanium. These choices were based on (i) the results of the unimplanted ceramic–ceramic tests, which led to the selection of TiNi and nickel, and (ii) work reported in the literature [30] which seemed to indicate possible lubricious properties for oxides of cobalt and chromium. As will be shown subsequently, the best results for unimplanted sliding pairs corresponded to the transfer, from pins onto discs, of metal oxides to form discontinuous, but relatively lubricious, films. Unfortunately, this wore the pins significantly and did not produce uniform lubrication. Therefore, it was

TABLE I Material properties

Material	E (GPa)	H (GPa)	K_{IC} ($MNm^{-3/2}$)	Grain size (μm)	Tensile strength (MPa)
Carborundum sintered alpha SiC	380	32	4.4	3–5	345
HP TiC (Ceradyne, Inc.)	450	32	5.0	30	260
TiC–Ni–Mo (K162B) (Kennametal Corp.)	407	12	~ 15	3	1587
Si_3N_4 (NC 132) (Norton Co.)	310	19	4.8	0.5–3	810
Partially stabilized zirconia (Nilsen (Nilcra) TS grade PSZ)	210	10	8–15	60	600

E = Young's modulus; H = hardness; K_{IC} = fracture toughness.

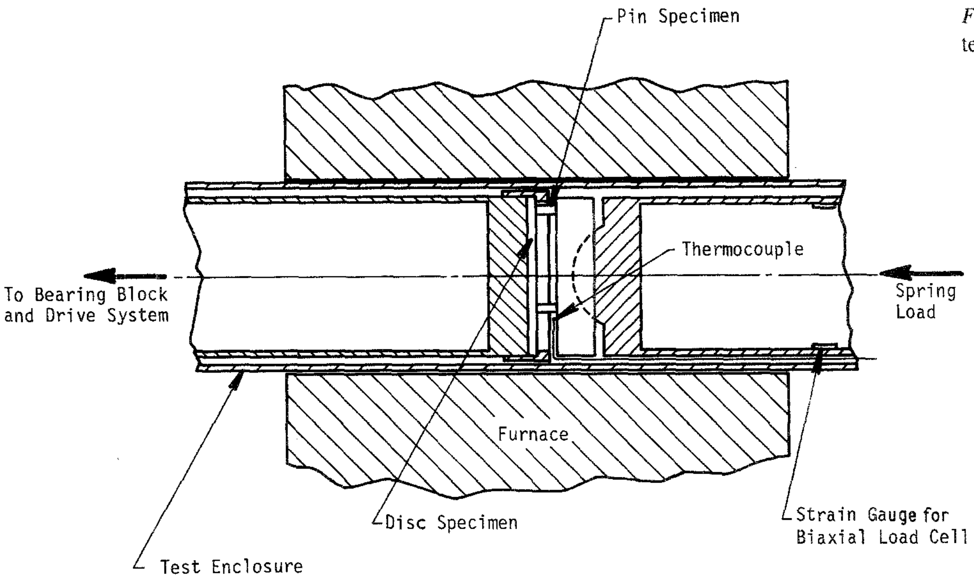

Figure 1 Schematic diagram of test set-up.

decided to preimplant candidate metal oxide films into the discs, in the hope that they might transfer to the pins, while maintaining minimal disc wear.

2.2. Ion implantation

The implantation process used was actually a variant known as ion mixing. In this technique, a thin coating of metal is vapour deposited onto the ceramic substrate, and argon ions are accelerated through a potential field, driving the deposited metal atoms into the substrate. In the case of the TiNi implant, nickel was first deposited, followed by the titanium; both layers were then simultaneously ion mixed.

The argon ions were accelerated using a beam voltage of 140 keV, with a fluence of 1×10^{17} ions cm^{-2}, and a flux of $\sim 10^{12}$ ions cm^{-2} sec^{-1} (Westinghouse R&D Center, Pittsburgh, Pennsylvania). Implant layer thickness was estimated to be on the order of $\gtrsim 400$ nm, based on subsequent Auger analysis.

2.3. Friction and wear testing

A special wear testing machine was constructed to permit testing under conditions that are pertinent to the service environment of sliding seal materials in an adiabatic diesel engine. As sketched in Fig. 1, the basic configuration of the machine was that of a rotating disc against which three flat-ended pins of identical material slide under controlled normal force. The pin and disc assembly were housed within a heated environmental chamber. Since the pins suffered continuous sliding, while any point on the disc surface experienced only periodic passage of a pin, the pin materials operated under conditions similar to those imposed on a sliding seal ring, and the disc material experienced conditions analogous to those of cylinder liner materials.

Testing was performed from 23 to 800° C, in both argon and simulated diesel exhaust environments, although most of the ion-implant tests were carried out at 800° C in the diesel exhaust environment. The latter consisted of 7.8% CO_2, 8.9% O_2, and the balance N_2; both diesel exhaust and argon environments were maintained at 276 kPa pressure. Discs and pins were run-in at temperature for 5 min at the start of each test, following initial evacuation and purging of the system with either diesel exhaust or argon and a minimum 20 min pre-test soak at test temperature. Each test was performed under a static load of 22.5 N, with the disc rotational speed at the point of pin/disc contact being 1 m sec^{-1}. Run time ranged from 40 min to 1 h.

2.4. Wear characterization

Scanning electron microscopy was used to characterize wear mechanisms, while scanning Auger electron spectroscopy (AES), and energy dispersive spectroscopy (EDS) were employed to characterize wear particles and transfer layers. Surface profilometry was used as a measure of relative material loss. Because of problems associated occasionally with chipping and frequently with oxidation, wear rate determination by the measurement of specimen weight change was frequently compromised.

Prior to testing, both pins and discs were carefully polished to a 0.5 μm diamond finish. There was always, however, a slight mismatch in parallelism between pins and disc once a test began, and so on each pin, a small wear flat was produced (tests did not begin until this flat was run-in and basically stable). For subsequent wear characterization, the polished surfaces provided reference "no-damage" regions, while the boundaries of the pin flats constituted regions in which damage development could be observed from its inception to equilibrium wear. Similarly, the width of the annular wear track on each disc slowly expanded with the pin flats, so that again, by tracking across the boundary one passed quickly from no damage through the various stages of damage development leading to equilibrium wear.

The Auger analysis was extensive, and the procedures employed, results, and interpretation are the principal focus of a following companion paper [31]. The present work will invoke those results.

3. Results

3.1. Friction coefficients

Steady-state friction coefficients (μ_F) for a typical ceramic–ceramic pin/disc couple in diesel exhaust

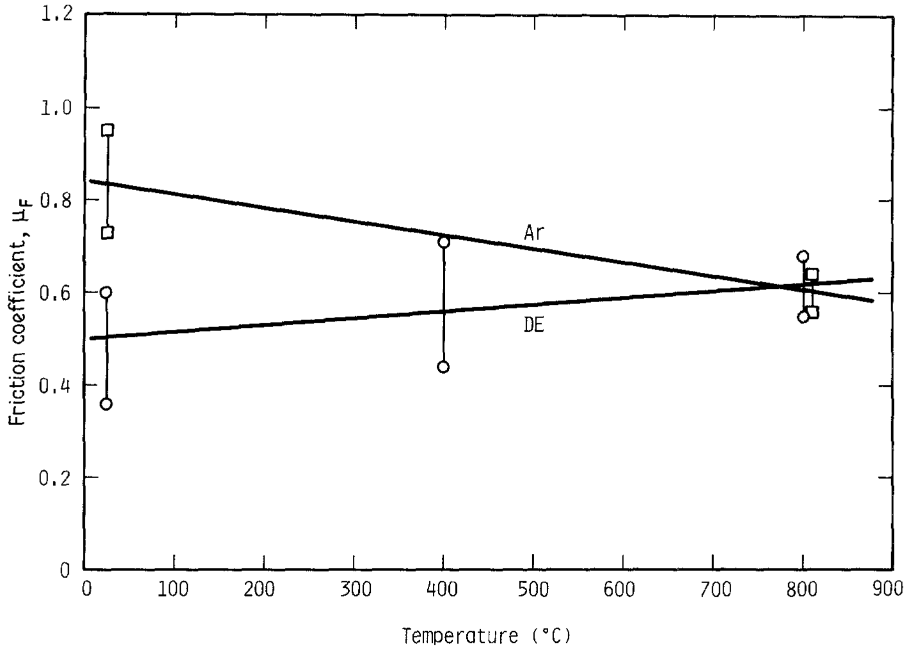

Figure 2 Friction coefficient against temperature for TiC pins/NC 132 Disc in DE and argon.

and argon are shown as a function of temperature in Fig. 2; scatter bands indicated were characteristic of all couples tested. A more effective mode of presentation for subsequent discussion is to combine the curves for all three pin materials sliding on one disc in a given environment as in Figs 3–6. For example μ_F lies between 0.6 and 0.8 for all three pin materials run against zirconia in argon (Fig. 3). For Si_3N_4 discs in the same environment (Fig. 4), the situation is only marginally different; as the temperature increases, μ_F for all pins decreases slightly from ~0.8 to ~0.6. This simple behaviour is altered drastically, however, by running in diesel exhaust environment.

As shown in Fig. 5, for zirconia discs in diesel exhaust μ_F rises rapidly with temperature for SiC and TiC, peaks at ~400° C, and then decreases rapidly with further increase in temperature. For TiC–Ni–Mo pins on the other hand, μ_F decreases monotonically from its initial room temperature value of ~0.9, to a low of ~0.45 at 800° C. For Si_3N_4 discs in diesel exhaust (Fig. 6), the TiC–Ni–Mo material behaves much as it does for zirconia discs in the same environment, dropping from ~0.75 at 23° C to 0.24 at 800°C. However, $\mu_F(T)$ for SiC and TiC increases monotonically; there is no mid-temperature peak, and at 800° C, μ_F is quite high for both.

Since most of the ion-implanted discs were run at a single elevated temperature (800° C), it is most convenient to consider the corresponding friction coefficients as shown in Fig. 7. Clearly, ion-implantation with chromium is no more beneficial to friction behaviour than is the use of unmodified ceramic couples. However, TiC–Ni–Mo pins run against zirconia discs implanted with TiNi and with cobalt have coefficients of friction of 0.09 and 0.06, respectively. Similarly, TiC pins with Si_3N_4 implanted with TiNi exhibit a friction coefficient of 0.09. These are extremely low friction coefficients, lying within the upper end of the range of values normally achieved through conventional liquid lubrication at low (< 300° C) temperatures.

It is interesting to note that for TiC pins run against

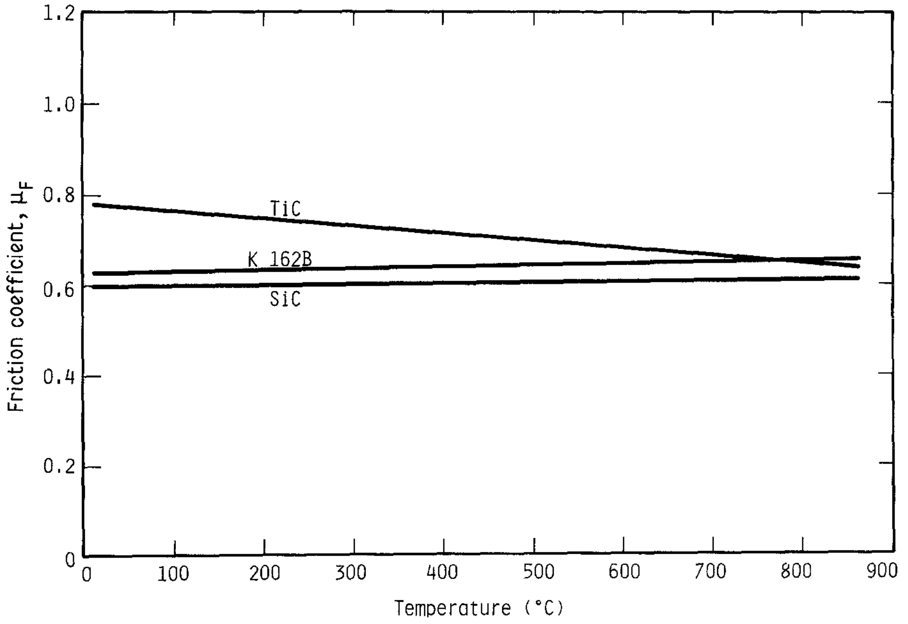

Figure 3 Friction coefficient against temperature for all pin materials on PSZ discs in argon.

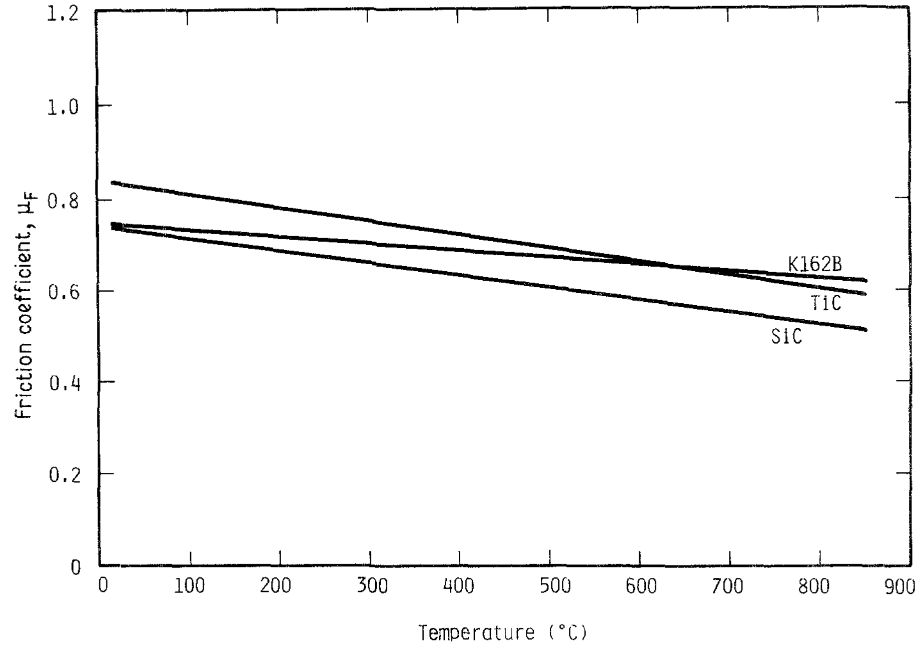

Figure 4 Friction coefficient against temperature for all pin materials on NC 132 discs in argon.

nickel-implanted silicon nitride, μ_F was $\geqslant 0.14$, considerably worse than the value of 0.09 obtained for the TiNi-implanted case. Also, it is obvious that good friction coefficients are very sensitive to the precise nature of the couples. Titanium carbide was much worse than NiMo-bonded TiC against TiNi-implanted zirconia, while for TiNi-implanted Si_3N_4, the NiMo–TiC was superior (lower μ_F) to the TiC.

3.2. Wear characterization

For non ion-implanted ceramic–ceramic couples, by far the most commonly observed wear mechanism was delamination parallel to the sliding direction. Radial cracking/chipping, in fact, was observed in only one instance, i.e. for SiC pins sliding on a Si_3N_4 disc at 23° C in diesel exhaust environments. The cracks penetrated at a fairly steep angle, and interacted to form irregularly-shaped chips (Fig. 8). The Si_3N_4 disc, on the other hand, manifested the much more commonly observed wear mechanism of delamination, the early stages of which are associated with the edges of the wear tracks.

An an example of this process, the development of delamination in Si_3N_4 and zirconia discs can be seen in Fig. 9, which shows several aspects of the process. In Fig. 9a, the delaminations have formed after no more than a few passes. Close study of equilibrium wear regions (Fig. 9b) shows that they are composed of thin sheets in the process of being removed (Fig. 9c). Delaminated sheets of zirconia could often be found lying at random on the discs and on the pins.

Delamination-type disc wear similar to this prevailed in all tests involving SiC pins, and for all tests involving zirconia in argon. In all of the other tests, which tended to correspond to lower friction coefficients, erosion occurred primarily by pin delamination combined with the transfer of pin material, either titanium oxide in the case of TiC pins, or titanium and/or nickel oxide*, in the case of NiMo-bonded TiC (molybdenum never was transferred, since at 800° C it

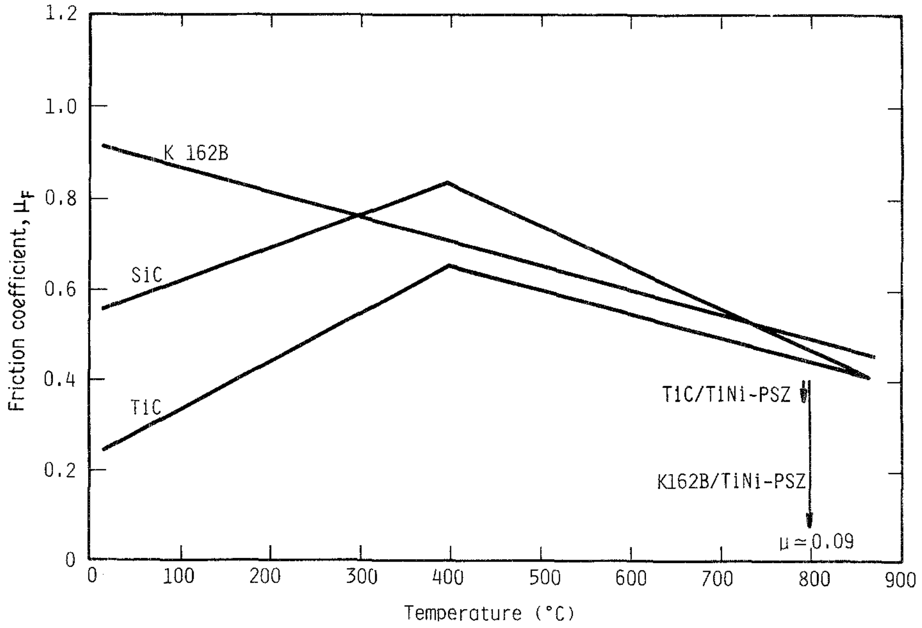

Figure 5 Friction coefficient against temperature for pure PSZ discs in DE.

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*We shall use TiO_2 and NiO to indicate these oxides, although the exact stoichiometry was not determined.

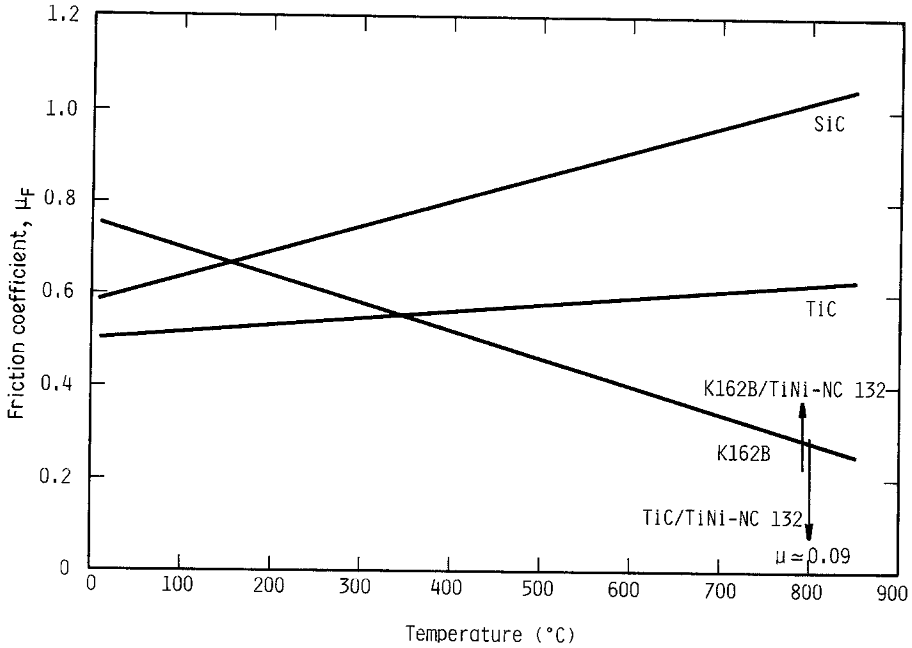


Figure 6 Friction coefficient against temperature for pure NC 132 discs in DE.

rapidly volatilizes as MoO_3). An example of such a film transfer process is shown in Fig. 10, in which TiO_2 has transferred from TiC pins to Si_3N_4 discs at 23 and 800° C in the diesel exhaust environment.

Before discussing the wear behaviour of the ion-implanted material, it is instructive to consider the nature of the implanted "coatings". As shown in Fig. 11a, the titanium and nickel are mixed together reasonably well, but contain relatively little of the underlying Si_3N_4 (indicated by the silicon profile). Some oxygen is present near the surface, predominantly within the titanium-rich portion of the "alloy". However, after a 30 min soak at 800° C in the diesel environment (Fig. 11b), both the titanium and, especially, the nickel have diffused together, oxidized, and mixed with the Si_3N_4. The thickness of the implanted coating is estimated, on the basis of previous sputtering rate work, to be around 0.40 μm. Further discussion of these points is provided elsewhere [31].

During wear tests, the pins oxidize, and then develop small wear zones where they have been in contact with the ion-implanted discs. An example of such a zone is shown in Fig. 12 for a TiC–Ni–Mo pin run against TiNi–zirconia. Based on the sizes of such zones, the contact stress was typically on the order of 3.5 to 7 MPa.

Inspection of the "wear zones" shows that they actually do not represent pin wear; instead, they are thin lubricating films transferred from the ion-implanted discs. Auger spectroscopy [31] indicates that the films are composed of TiO_2 and NiO. Similar results were found for the cobalt-implanted zirconia discs tested against TiC–Ni–Mo pins, i.e., CoO was transferred from disc to pin.

4. Discussion

When disc (and heavy pin) wear was observed, it usually was in the form of delamination. The latter is a form of adhesive wear, in which intermittent contact nucleates shallow subsurface cracks which eventually

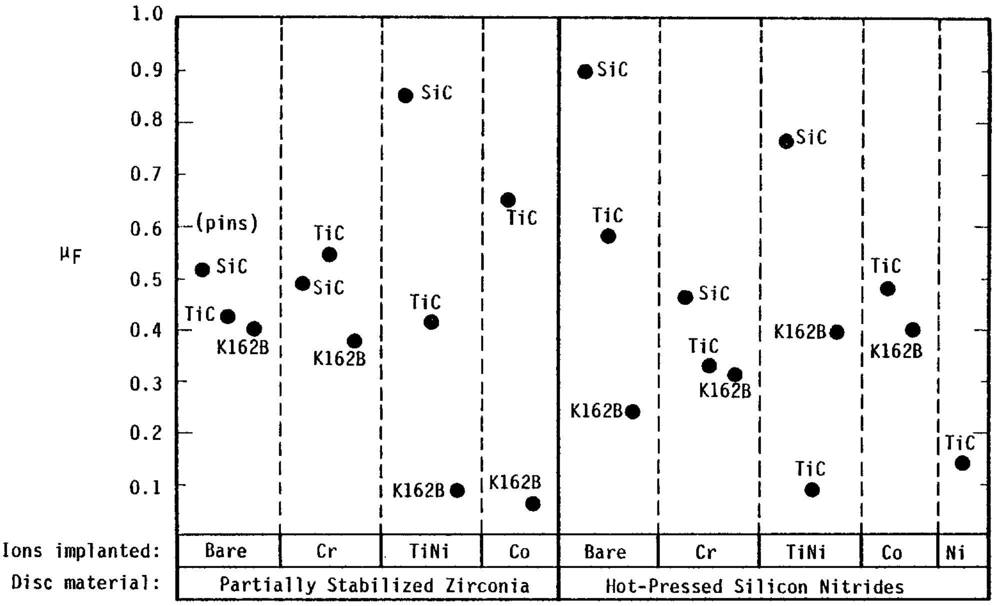


Figure 7 Steady-state coefficient of friction at 800° C in DE.

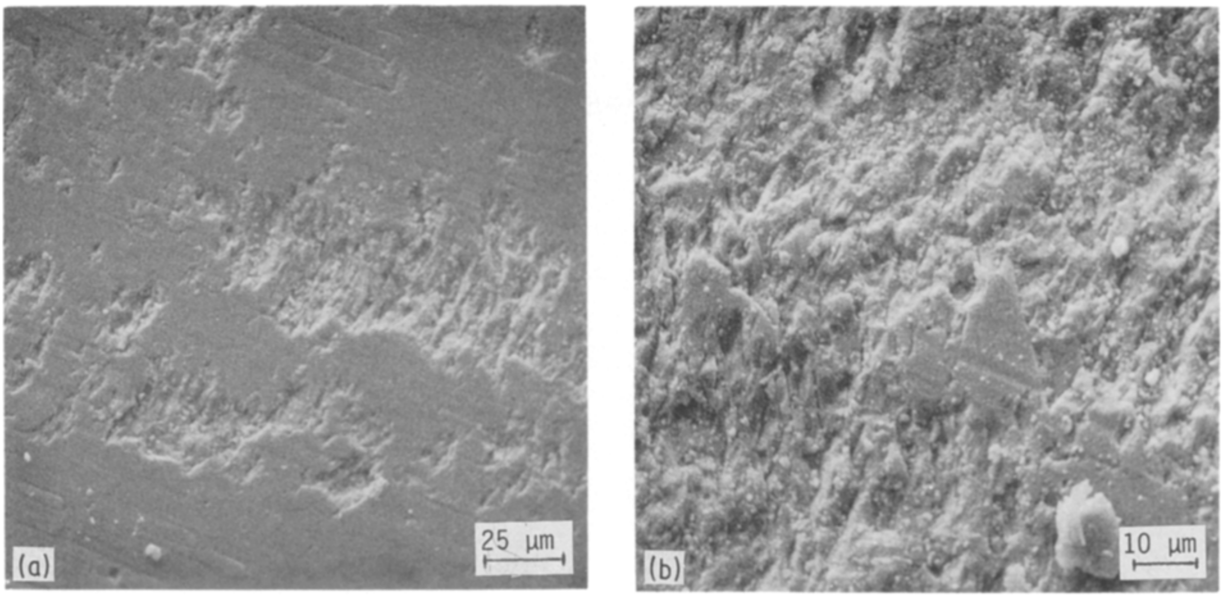
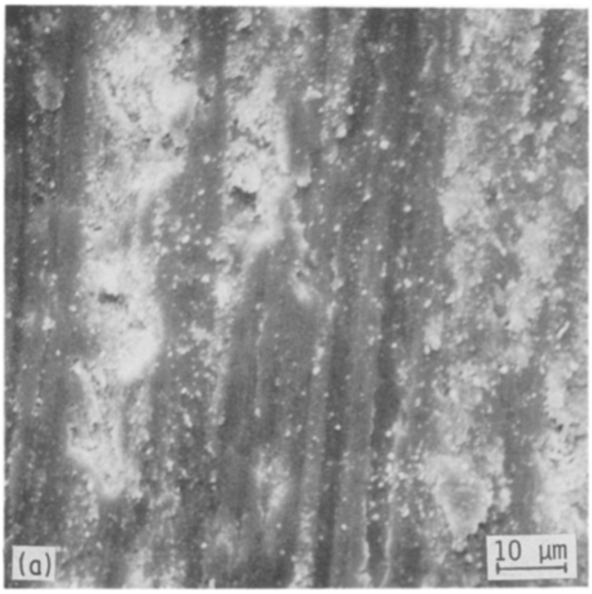


Figure 8 Radial crack wear particle formation in SiC pin run against NC 132 at 23° C in DE. (a) Initial wear, (b) equilibrium wear.

grow to the surface, creating a thin delaminated sheet of material. This is similar to delamination which has been observed in metals [32] and in ceramics [1], and modelled based on a theory [33, 34] of cyclic crack growth. However, in the present case, delaminate sheets often form within only a few, possibly even one, pass (cycle). This is just as observed by Yamamoto and Buckley [35] for 304 stainless steel wear sheets produced by sliding against Al_2O_3. In fact, the photomicrographs of wear layers in the steel alloy studied by the latter look almost identical to those observed (Fig. 9) in the present ceramics. Prevention of this process must involve either (i) provision of sufficient lubrication to inhibit ceramic–ceramic adhesion,



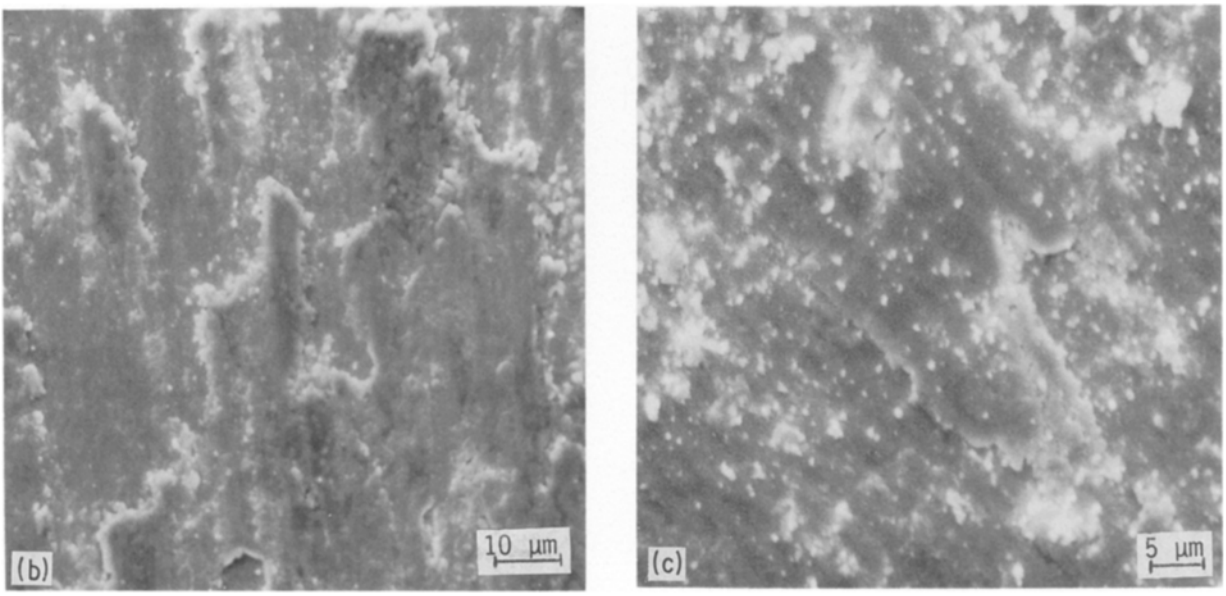


Figure 9 Examples of delamination in NC 132 and PSZ discs, 23° C in DE. (a) NC 132 disc (SiC pins), (b) PSZ disc (SiC pins), (c) PSZ disc (TiC pins).

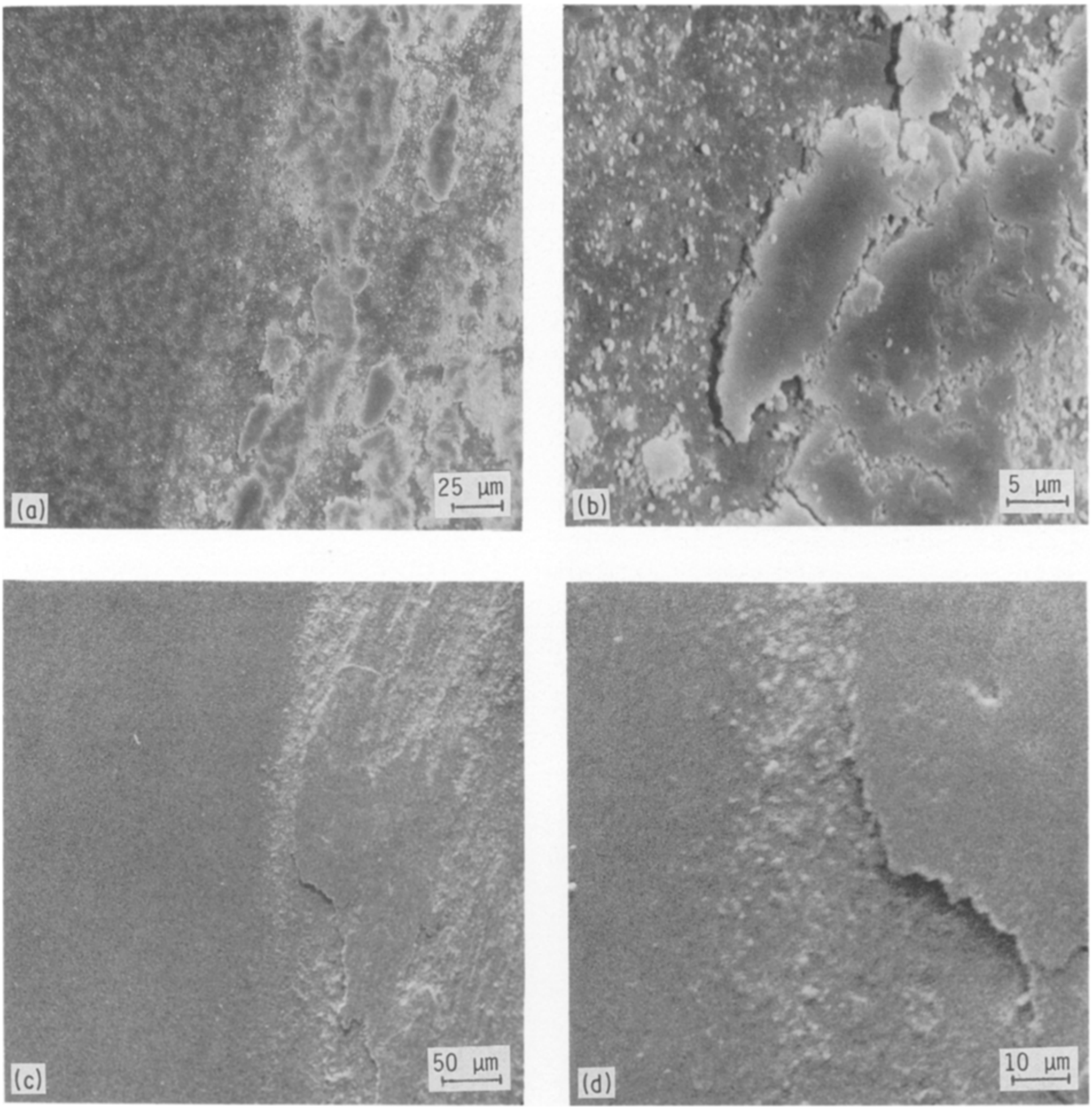


Figure 10 TiO_2 deposits on NC 132 discs run against TiC pins in DE. (a) TiO_2 deposit, disc, 23° C, (b) Details TiO_2 deposit, disc, 23° C, (c) TiO_2 deposit, disc, 800° C, (d) Details TiO_2 deposit, disc, 800° C.

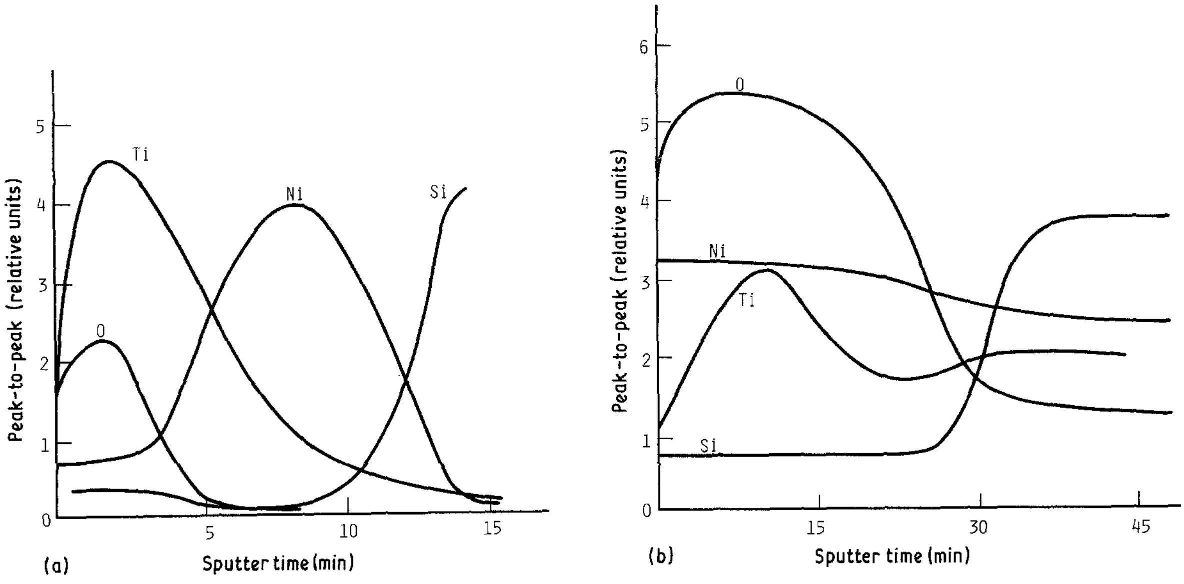


Figure 11 Scanning Auger analysis of TiNi ion-implantation in NC 132. (a) As-implanted elemental distribution, (b) elemental distribution after 30 min at 800° C in DE.

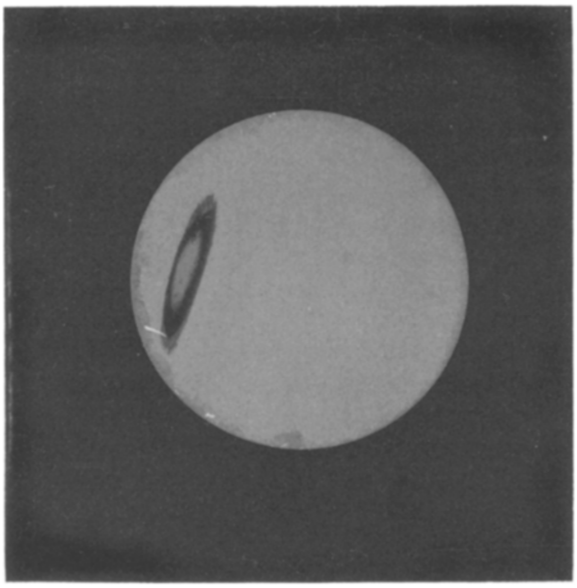


Figure 12 Wear zone (deposits) on K162B pin run against TiNi-PSZ.

and/or (ii) increase in the near-surface crack growth resistance of the delaminating material.

Although the friction coefficients of all of the unmodified couples exceeded 0.2, there were encouraging results regarding wear resistance. For those cases in which TiC or TiC–Ni–Mo material was transferred to discs in the form of titanium oxide films, disc wear was essentially non-existent (unmeasurable via surface profilometry), and pin wear did not appear to be high once the initial sacrificial material transfer was accomplished. Apparently these films, which can form from room temperature to 800° C, are lubricious at higher temperatures.

The true potential of the films, of course, seems to have been realized by first ion-implanting and then oxidizing the discs. Under these circumstances, titanium oxide, nickel oxide, and cobalt oxide transfer selectively, i.e. from certain discs to certain pins (Figs 13 and 14), and serve as liquid-like lubricants, with correspondingly low friction coefficients ($\mu_F < 0.1$) at the operational temperature. Based on the results of Breznak *et al.* [1], it would be reasonable to suspect that the temperature rise at the sliding interface was at least 300° C, hence the "lubricant" temperature probably was at least 1100° C. This should be sufficient to enhance significantly the viscosity of a ternary or quaternary oxide alloy. In this regard, it is interesting to note that the friction coefficient of nickel-implanted Si_3N_4 run against TiC in diesel exhaust was 0.14 at 800° C, but rose to 0.4 when testing was performed at room temperature. Evidently the NiO, which was observed to transfer during sliding, was not sufficiently hot at 23° C (plus ~300° C due to sliding contact) to serve as an effective lubricant.

It should be borne in mind that work to date has emphasized relatively short term tests designed to screen the best pin–disc–ion implant combinations. It will be very important to evaluate the ability of the self-lubricating systems which have been identified to maintain their desirable traits over extended operating times.

5. Conclusions

Friction and wear tests have been conducted on a variety of ceramic–ceramic couples in the unmodified state, and with their surfaces modified by ion implantation, in both argon and an environment typical of high temperature adiabatic diesel engines. It has been found that:

1. Lowest friction coefficients of unmodified ceramic couples correspond to the formation of lubricious oxide films.
2. Oxide film formation depends on disc/pin combination, temperature, and environment.
3. The most common and destructive mode of wear is delamination.
4. For unmodified ceramic pin/disc combinations, the lowest friction coefficient obtained was 0.24.
5. Ion implantation of discs with TiNi or cobalt reduces coefficients of friction at 800° C in simulated diesel exhaust environment to 0.06–0.09, for certain specific pin–disc combinations.
6. The latter combinations appear to be attractive piston ring/liner candidates for the adiabatic diesel engine.

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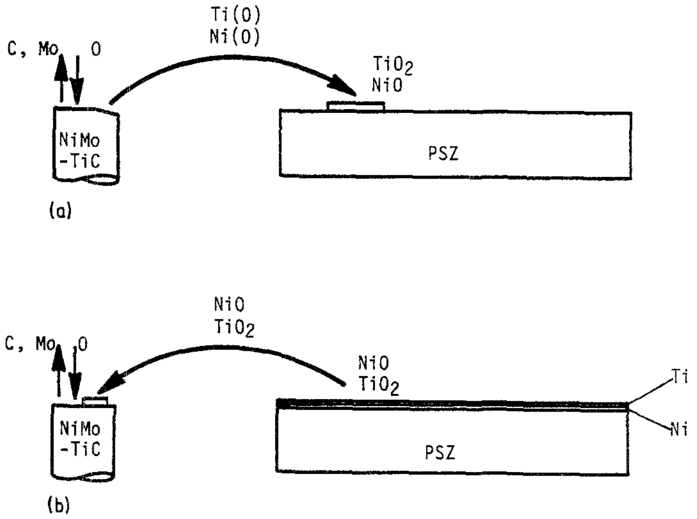

Figure 13 Film-lubricant transfer in the NiMo–TiC/PSZ systems. (a) Pure PSZ, (b) TiNi-implanted PSZ.

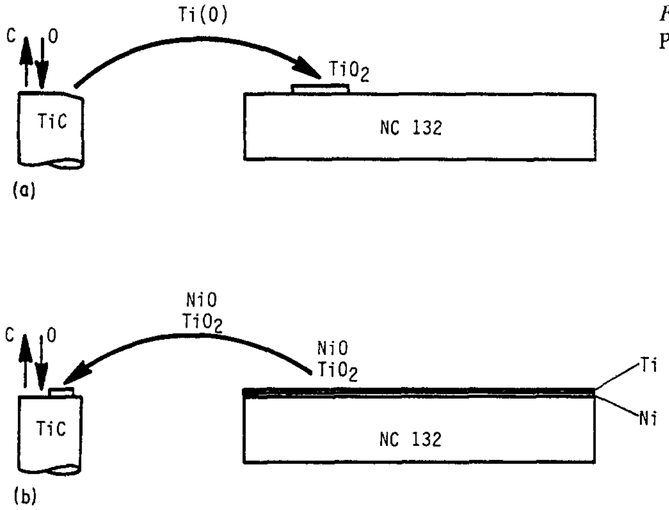

Figure 14 Film-lubricant transfer in the TiC/NC 132 systems. (a) Pure (b) TiNi-implanted NC132.

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