

Wear performance and mechanisms of EN coating under reciprocating sliding conditions

C. M. LI*, K. N. TANDON

Metallurgical Sciences Lab/EITC-AIM Initiative, Department of Mechanical and Industrial Engineering, University of Manitoba, Winnipeg, Manitoba, Canada R3T 2N2

Electroless nickel (EN) coatings are commonly used as an engineering coating for protecting components from corrosion and wear. In the present work, EN was applied to a mild steel substrate and its wear performance under dry as well as lubricated conditions was evaluated for reciprocating sliding wear. A sliding pin made from nodular cast iron was used as the mating surface. In order to assess the wear mechanisms involved in the process, both optical and scanning electron microscopy were utilized to study the wear surface and the debris. The results indicate that the EN coating performed very well under lubricated conditions. In dry tests, extensive adhesion and material transfer was observed. Scanning electron micrographs have been interpreted to analyse various wear mechanisms operating under different loading conditions.

1. Introduction

Electroless nickel (EN) coating is deposited by an autocatalytic reaction without the use of an external electrical current. Of the three main types of coatings (nickel–phosphorus, nickel–boron and pure nickel), nickel–phosphorus has been developed and used extensively in commercial applications [1, 2]. The main advantages of using EN coating are its excellent resistance to wear and corrosion as well as its capacity to deposit a uniform coating thickness. Research has shown that various EN coatings offer excellent lubricity and wear resistance under certain conditions [3–8], and resistance to various chemical environments [9–11].

The two most important properties that have generated so much interest in Ni–P coatings are (a) the high hardness of the deposit, and (b) the uniformity of the coating. The as-plated hardness of the coating is around 500 H_v which can be increased to 900–1000 H_v after suitable heat treatments. This increase in hardness is attributed to the precipitation of nickel phosphide which is well documented and researched in the literature [12–14]. Furthermore, the phosphorus content of the coating can be varied from 5%–12% to achieve desired properties in terms of coating strength [15].

This research was undertaken as part of a major project, the objective of which is to evaluate alternative coatings for locomotive diesel engine liners where hard chrome is a conventional coating material. The wear conditions in this application are essentially reciprocating and sliding under mild lubricated conditions. Wear is complicated in nature because it is not an intrinsic property of the material, but rather the

response of the material system to the relative motion under certain specified conditions. It therefore limits the comparability of wear data from different tests and necessitates the use of proper mating surfaces under simulated conditions.

It was with this purpose in mind that the present research focused on using cast iron, a common piston-ring material, as a mating surface for the simulated testing of the coating.

The wear behaviour of the EN coating has been evaluated mostly under dry non-lubricated conditions, from the several mechanisms established for the metal to metal wear [16, 17]. Essentially, there are two mechanisms involved in the failure of the EN coating under wear: adhesion and abrasion [7]. However, the relationship between the two is still not clear. The wear characteristics of EN coating against modular cast iron has not been investigated, and such a study is very useful for the application stipulated above. Moreover, a detailed microscopical analysis of the wear process, debris and its effect on the EN coating loss for the reciprocating sliding wear is still lacking.

2. Experimental procedure

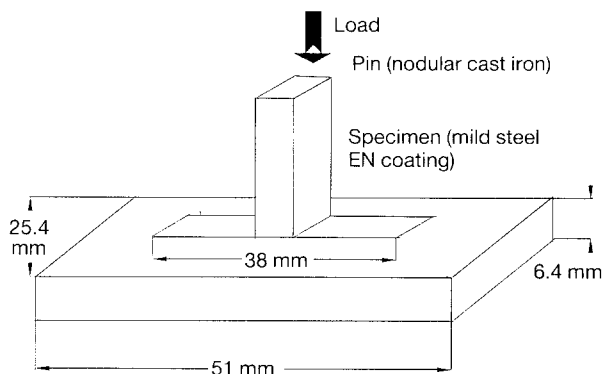
A special wear-testing apparatus was designed and fabricated to simulate the wear conditions of the diesel engine liner, and details of the machine have been described elsewhere [18]. The dimensions of the pin and the coated flat specimen are shown in Fig. 1. The pins were machined from nodular cast iron piston rings used in the diesel engines. The hardened bainite matrix of the pin had a hardness of 550 H_v.

* Permanent address: Department of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics, Beijing 100083, People's Republic of China.

EN plating was applied using a Shipley bath formula NIPOSIT Electroless Nickel NL-63 giving a phosphorus content of 7%. The EN coating thickness was 0.075 mm (3 mil) on both sides of the flat specimen (Fig. 2), with a roughness of $R_a = 2.56 \mu\text{m}$ (Fig. 3). Wear test under lubrication (see below) showed that this roughness was acceptable for direct use on cylinder liners without further machining. The roughness on different sides varied somewhat: there were more coarse nickel particles on one side than on the other, presumably because of the improper circulation of the (bath) solution. The EN coating was, however, uniform (Fig. 2), and was heat treated to a hardness of $H_V = 1080$ (200 g).

Wear tests were conducted under loads of 0.5, 1.0, 1.5 and 2.0 kg for unlubricated conditions, and under loads of 2, 3, 5, 8 kg for lubricated conditions. The tests were carried out for different numbers of cycles under each load. The reciprocating frequency was $414 \text{ cycle min}^{-1}$, or $828 \text{ stroke min}^{-1}$. The track length was 38 mm, indicating an average relative motion of 525 mm s^{-1} . Motor oil 10W30 was used as a lubricant with a supply of 4 ml hr^{-1} which maintained a constant oil cover on the wear track.

The wear of the electroless nickel coating was determined by the loss method, using a balance with a sensitivity of 10^{-4} g . Specimens were cleaned ultra-



Reciprocating motion at $414 \text{ cycle min}^{-1}$, $828 \text{ stroke min}^{-1}$
Average speed of the relative motion, $= 525 \text{ mm s}^{-1}$

Figure 1 Schematic diagram of the reciprocating pin-on-flat wear tester.

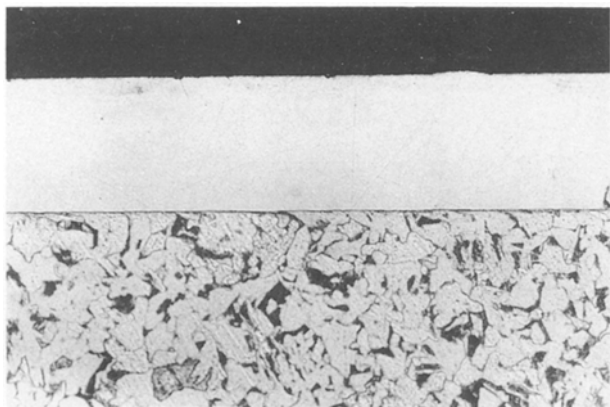


Figure 2 Micrograph showing the uniformity of the EN coating and the mild steel substrate, $\times 300$, 2% Nital etch.

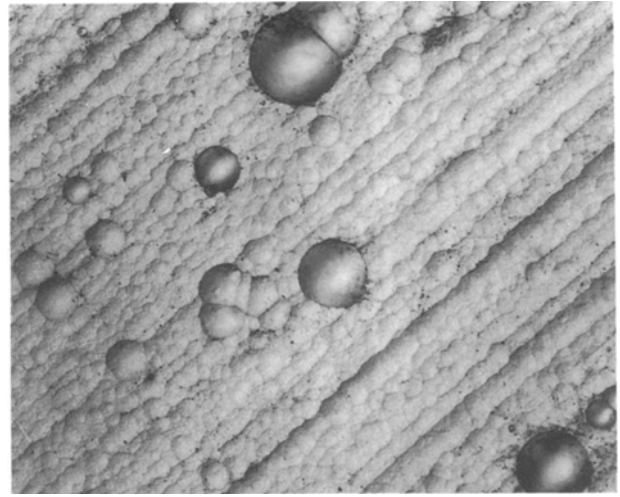


Figure 3 As-deposited EN surface, $\times 400$.

sonically and weighed before wear testing, and were cleaned and weighed again after being tested.

Optical microscopes and a scanning electron microscope (with EDAX facility) were used for the wear track and debris examination and the wear mechanism investigation.

3. Results

3.1. Wear performance of EN coating

Fig. 4 shows the weight loss of the EN coating versus the cycles of wear testing with (Fig. 4a) and without (Fig. 4b) lubrication. The load on the pin was varied from 0.5–8.0 kg. The data for these tests are given in Table I.

The following features can easily be seen from Fig. 4.

1. The weight loss increases linearly with the number of cycles, which is consistent with the general trend for sliding wear [16, 17].
2. The running-in stage is not very pronounced.
3. Lubrication dramatically improves the wear performance of the EN coating (discussed later, Section 4.2).

Fig. 5a shows the variation in weight loss of the EN coating as a function of the applied load for a dry test period of 10 h, whereas Fig. 5b shows the weight loss as a function of applied load for lubricated tests. It can be seen from Fig. 5 that the effect of load on the weight loss is normal for an unlubricated test (weight loss is proportional to load), but is a bit "abnormal" in the case of lubricated tests (Fig. 5b) as there is a minimum on the curve. A tentative explanation is given in Section 3.2.

Observation of the wear tracks, especially from dry (unlubricated) tests, revealed that the wear was not uniform. The severest wear took place at the two ends of the track and the lightest wear took place in the middle of the track. This is, of course, very important in engine design and the selection of materials and processing. The test machine ran at $24840 \text{ cycle h}^{-1}$ ($414 \text{ cycle min}^{-1}$), which is not as fast as a diesel engine (typically 1100 r.p.m.), but the data are still of some value for reference.

TABLE I Wear data of EN coating

Lubrication condition	Load (kg)	Weight loss (mg) (duration of test (h))							
Unlubricated	0.5	3.6(3)	8.6(6)	10.6(9)	15.9(12)	24.2(19)	31.5(25)	41.1(31)	56.6(41)
	1.0	17.2(3)	30.7(6)	43.4(9.5)	55.0(12.5)	86.0(19.5)			
	1.5	36.5(3)	50.2(5)	71.8(7)	114.7(10.5)				
	2.0	7.2(1)	23.1(2)	44.6(3.25)	56.8(4.25)	72.3(5.28)	137.7(8.25)		
Lubricated	2.0	1.4(12)	1.8(36)	1.8(111.5)					
	3.0	0.1(22)	0.8(44)	1.1(67.5)	1.6(139.5)				
	5.0	-0.1(24)	0.9(48)	1.1(72)	1.2(167)				
	8.0	0.0(24)	1.6(45)						
	8.0	0.6(24)	1.6(48)	3.0(115)					

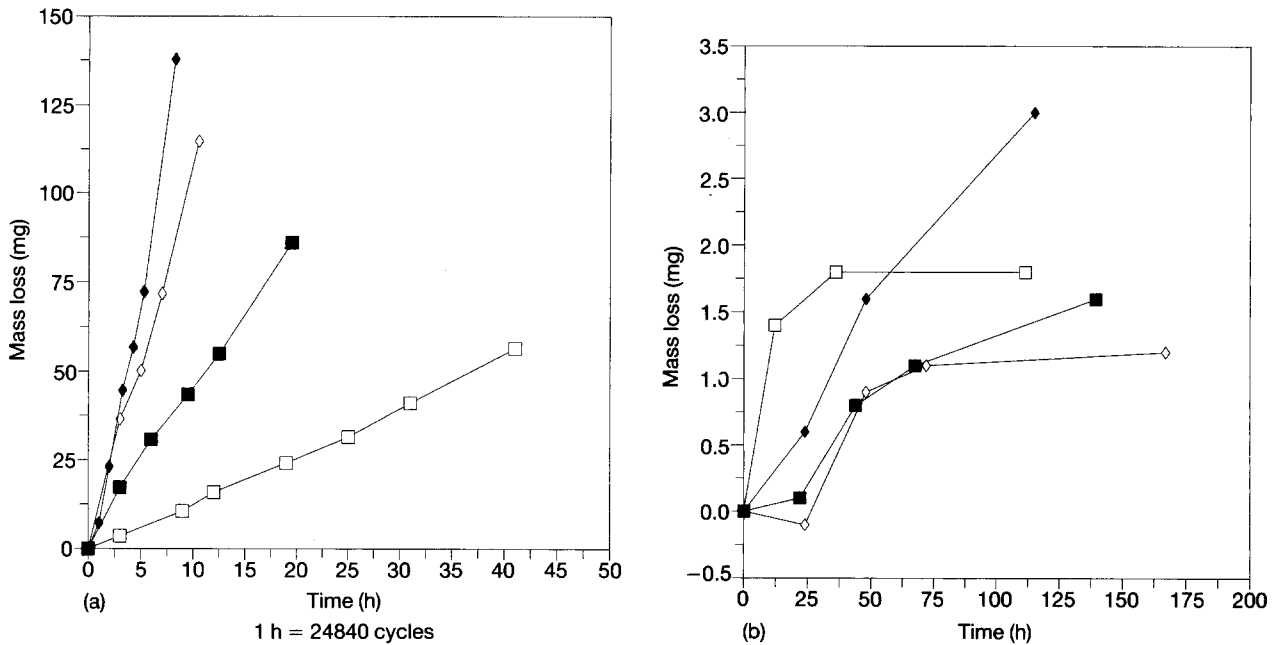


Figure 4 Mass loss of the coating against time for various loads (a) dry tests; (b) lubricated tests. (a) (□) 0.5 kg, (■) 1 kg, (◇) 1.5 kg, (◆) 2 kg. (b) (□) 2 kg, (■) 3 kg, (◇) 5 kg, (◆) 8 kg.

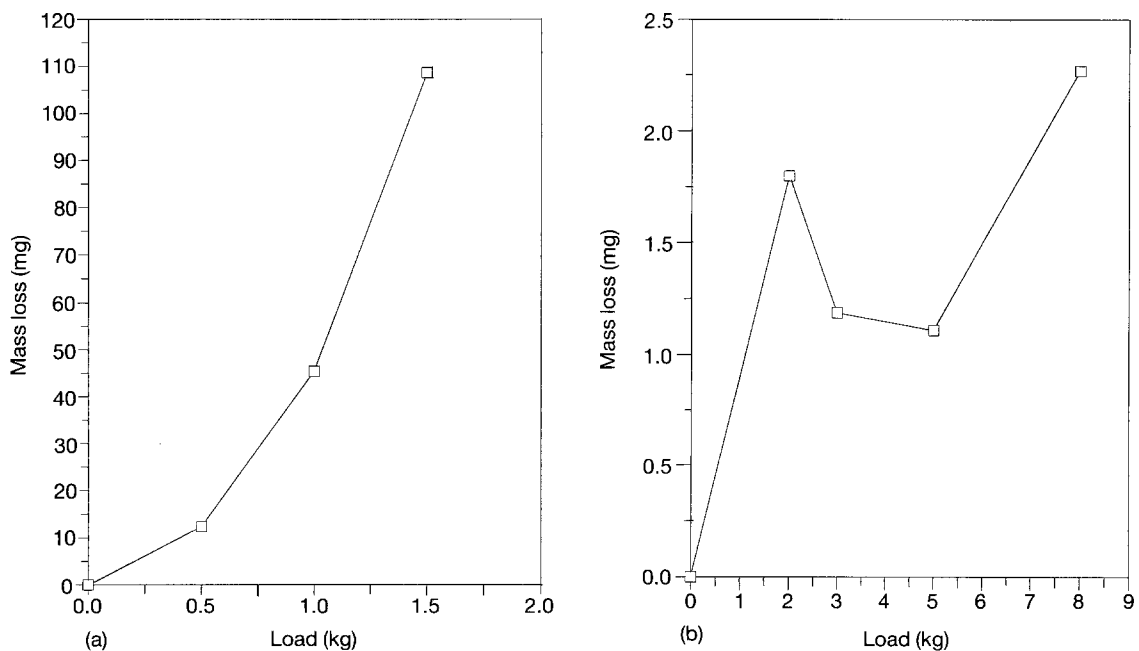


Figure 5 Loss in the coating mass as function of load for (a) dry tests (10h), and (b) lubricated tests (80h).

3.2. Observation of the worn surfaces and debris

3.2.1. Dry wear tests

Fig. 6a is an optical micrograph taken from a specimen dry wear tested under 1.5 kg load for 1.5 h, showing the early stage of wear. It is evident that the dominant mechanism of wear was abrasion caused by the asperities of the pin, although there was some

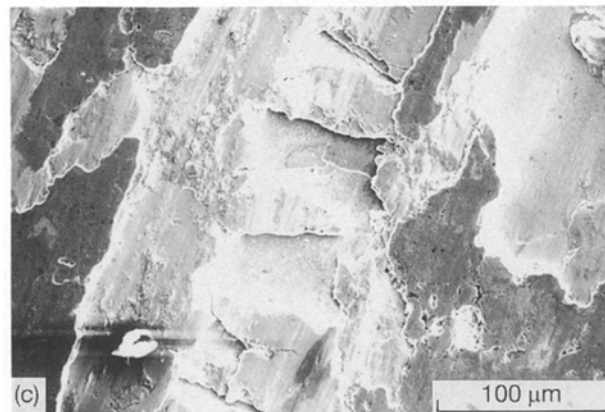
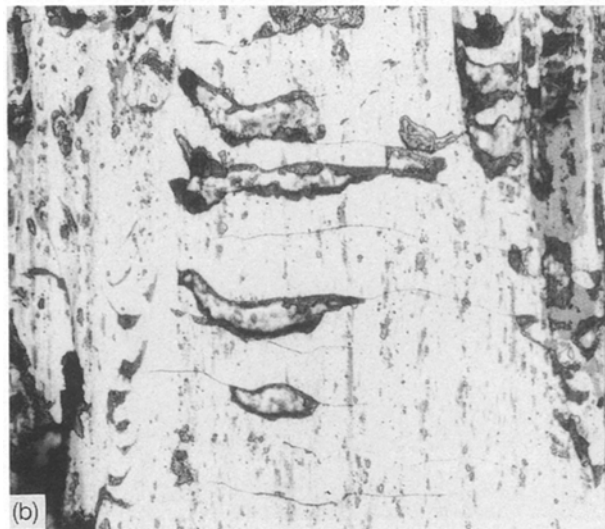
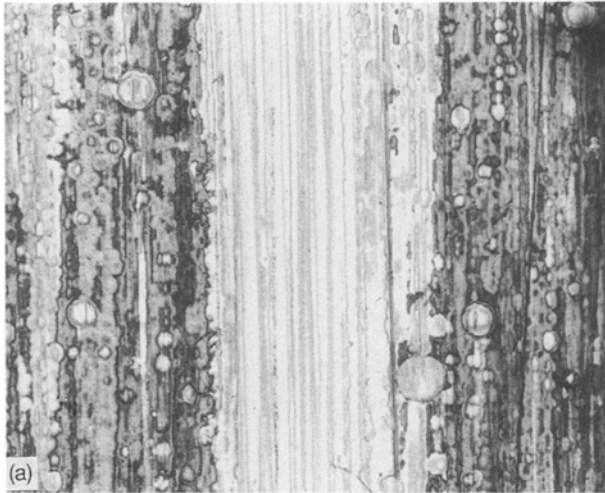


Figure 6(a) Optical micrograph of the wear track for a specimen dry tested for 4 h under 1.5 kg load, $\times 400$. (b) Optical micrograph of the wear track showing cracks perpendicular to the sliding direction. Specimen dry wear tested: load 0.5 kg, time 19 h, $\times 400$ (c) Scanning electron micrograph showing the delamination in the wear track. Specimen dry tested: load 2.0 kg, time 11.0 h.

adhesion observed. Many cracks can be seen at the bottom from the coating of some grooves, Fig. 6b. It can also be seen from Fig. 6b that some fragments have spalled off. The scanning electron micrograph in Fig. 6c shows clearly that the cracking and spalling is actually by a delamination mechanism, which is evidently one of the main mechanisms contributing to the weight loss of the specimen.

Ploughing or cutting is another wear mechanism, evident in Fig. 7. The micrograph in Fig. 7 was taken from the same specimen as in Fig. 6c but at a different location in the worn area. It is worth noting that the bulk hardness of the pin is much lower than that of the flat coating and yet ploughing of the coating has still occurred. In general there is usually some ploughing or cutting occurring on the harder part of a wear pair and work hardening of either the softer material itself or the debris is most likely responsible for this.

Fig. 8 a and b are scanning electron micrographs which were taken from a specimen dry wear-tested under a load of 0.5 kg for 41 h, showing that the dominant mechanism of weight loss is adhesive wear. It can be seen from these micrographs that the essential features of the adhesion surface are that the overall topography is irregular, with thin pieces of material torn away, leaving behind shallow, flat caves in different sizes and shapes. Usually the bottoms of the caves are flat (with or without small particles) sometimes with scratches in the direction of relative motion, according to the tearing condition. To confirm that the surroundings were not attached debris, EDS point analyses were conducted. The EDS data for different points in Fig. 8b are given in Table II.

Redeposition of the debris can be considered another form of adhesion. Although it may add some

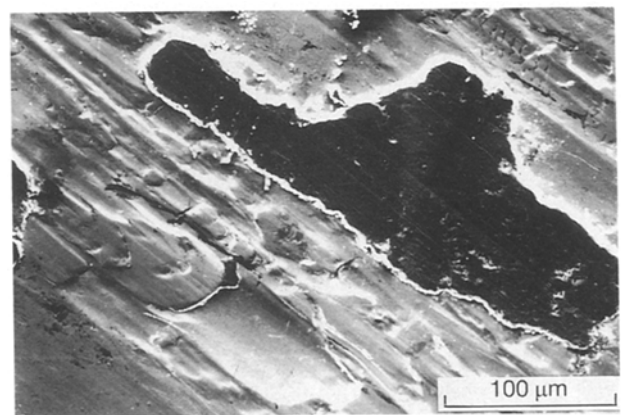


Figure 7 Scanning electron micrograph showing the ploughing and cutting of the coating dry tested under 2.0 kg load for 11.0 h.

TABLE II Data of point analysis of points in for Fig. 8b

Point number	Content (wt%)			
	Fe	Ni	Si	P
1	0.00	92.93	0.10	6.98
2	0.09	91.90	0.00	8.00
3	0.45	91.95	0.12	7.47
4	0.29	91.80	0.09	7.82

weight initially, later it will result in further weight loss because of the redetachment of the deposited debris. Fig. 9a shows a micrograph taken from a specimen wear tested under a load of 2 kg for 11.2 h. The dark area in Fig. 9a redeposited debris from the cast iron pin being rolled in to form a thin layer. An X-ray map of the same view field, Fig. 9b, shows that the main constituent of the dark layer in Fig. 9a is iron from the cast iron pin, and that the rest of the area is nickel and phosphorus, i.e. the EN coating. The topography of

the middle of Fig. 9a shows the features of adhesion, while the bottom left corner and top right corner, show features of abrasion. Point analysis was conducted on another specimen tested under a load of 1 kg for 26.5 h. Fig. 9c shows these points, and the data obtained are given in Table III. Ploughing and cutting of the hard coating is evident in Fig. 9d.

Checking of the worn pin surface also revealed redeposition of debris from the Ni-P coating. Fig. 10 shows a scanning electron micrograph of the selected

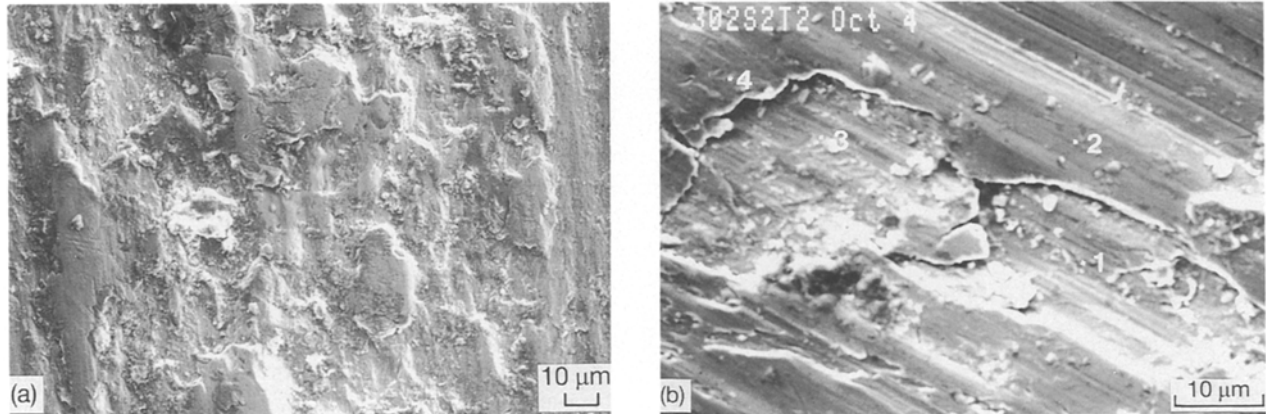


Figure 8(a,b) Scanning electron micrographs showing the features of adhesively worn surface of the coating dry wear tested under 0.5 kg load for 41.0h

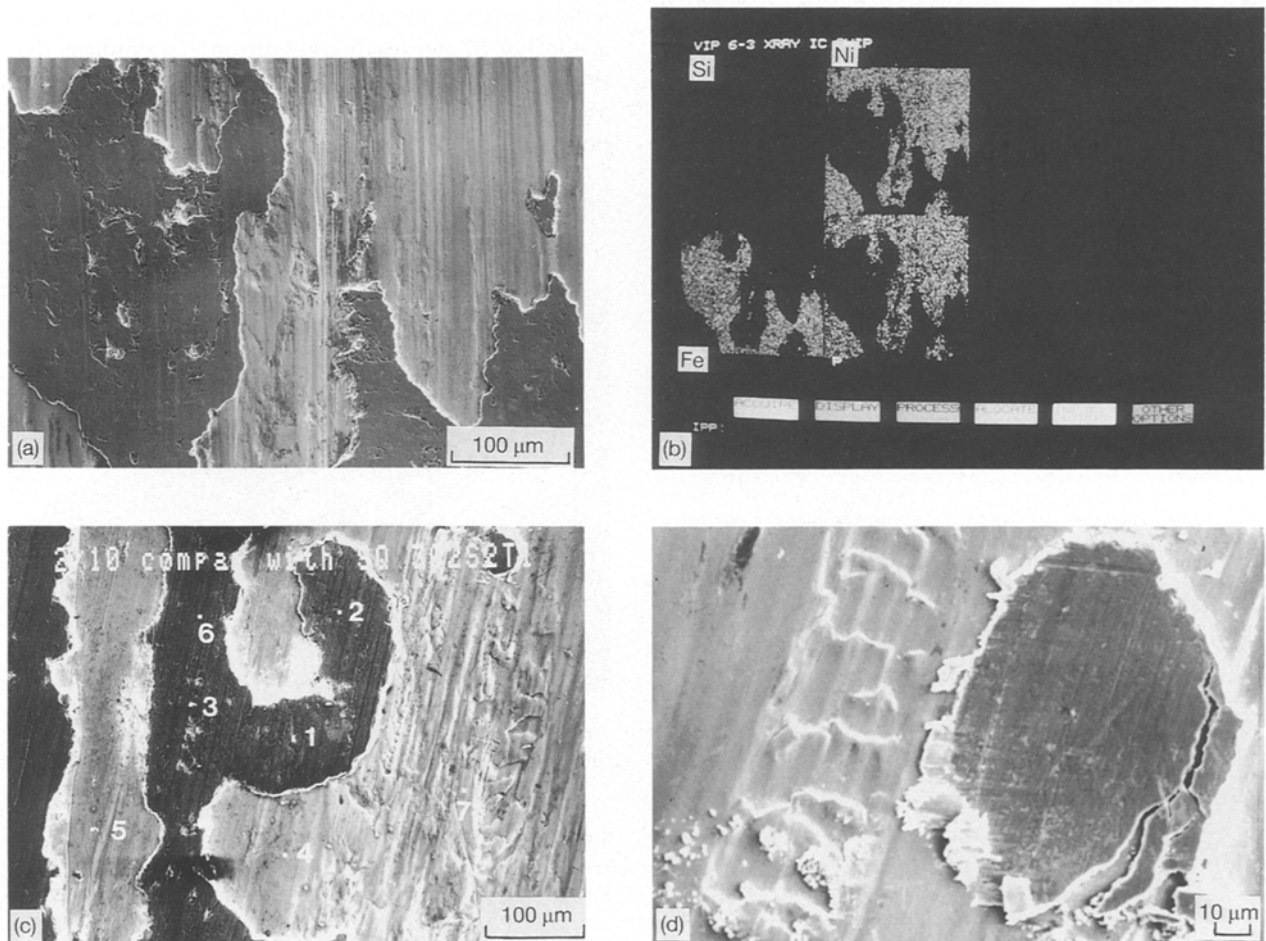


Figure 9(a) Scanning electron micrograph showing the redeposition and rolling-in of the debris on the coating surface, taken from a specimen dry wear tested under a load of 2.0 kg for 11.2 h. (b) An EDS X-ray map for silicon, nickel, iron and phosphorus showing the deposits as Iron. (c) Scanning electron micrograph of the deposited iron layer from a specimen dry tested under a load 21.0 kg for 26.5 h. Points marked indicate the locations of EDS analysis shown in Table III. Ploughing of the coating is also visible. (d) Deposited debris layer showing cracking and separation.

TABLE III Data of point analysis for Fig. 9

Point number	Content (wt%)					
	Fe	Ni	Cr	Mn	Si	P
1	91.10	7.35	0.11	0.76	0.50	0.18
2	91.79	6.74	0.00	0.60	0.52	0.35
3	90.73	7.41	0.12	0.77	0.61	0.38
4	0.36	92.01	0.00	0.00	0.12	7.51
5	0.82	91.50	0.06	0.00	0.07	7.56
6	91.61	6.79	0.07	0.49	0.66	0.37
7	1.26	90.93	0.00	0.00	0.10	7.70

TABLE IV Point analysis data for Fig. 10

Point number	Composition (wt%)				
	Fe	Ni	Ni	Mn	P
1	56.90	0.45	38.93	0.43	3.29
2	53.84	0.22	42.16	0.28	3.50
3	53.32	0.28	42.29	0.37	3.74
4	88.61	1.39	9.20	0.31	0.48
5	90.83	1.47	6.70	0.73	0.26
6	83.691	1.29	13.80	0.65	0.56
7	86.23	1.63	11.06	0.53	0.56
8	79.78	1.16	17.35	0.56	1.14
9	64.69	0.66	31.59	0.50	2.57

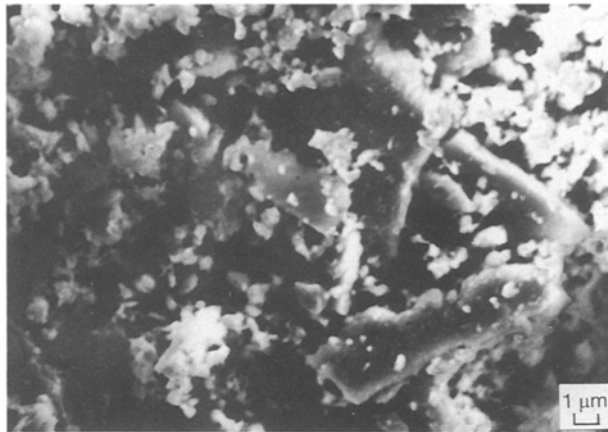


Figure 10 Scanning electron micrograph showing the surface of the cast iron pin, dry wear tested, 1.0 kg load, 26.5 h. The points indicate the location of EDS analysis (Table IV).

area of the worn surface of a pin which was used for the dry wear test. It can be seen from Fig. 10 that the dominant mechanism of wear in the area is adhesion. Several deposits from the counterface (EN-coated specimen) or mixed debris are attached to the pin surface. From the spot EDS analysis it is evident that the redeposited flakes of cast iron abraded more severely with the Ni-P coating and extensive transfer occurred (points 1, 2, 3, 8 and 9), whereas the low points (4, 5, 6 and 7) had smaller debris of Ni-P. Furthermore, large flakes or pieces of Ni-P are absent as well. It should be noted that the redeposited flakes of iron have undergone severe fatigue and work hardening.

Fig. 11 is a scanning electron micrograph showing the structure of the debris which was collected from

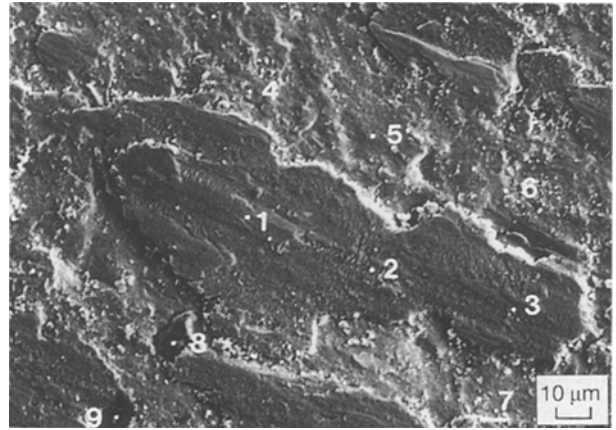


Figure 11 SEM image of the debris collected after 25 h test under a load of 0.5 kg.

the wear testing under a load of 0.5 kg for 25–31 h. It is obvious that the debris varies widely in size, from 0.1 μm to ~20 μm, and varies widely in shape. Most of them are small, about 1 μm nearly equiaxed, probably from both the pin and the flat Ni-P coating specimen during the mild abrasion. Several larger pieces seemed to come from the heavier ploughing of the EN coating.

3.2.2. Lubricated wear tests

When the wear tests were conducted under lubrication, the weight losses were drastically reduced, as shown in Table I and Fig. 5a. The corresponding features of the worn surfaces were also changed significantly.

Fig. 12a–c give a series of micrographs showing the topology of worn lubricated surfaces tested under different loads. (The common feature is that they were all in their first stage of the wear sequence—mild abrasive wear stage). Polishing of the coating has occurred. Many-sided lips of each groove show that the EN coating experienced some plastic deformation instead of material removal, and therefore kept the weight loss very low. Some evidence of adhesive wear and material removal can be seen in Fig. 12c. Observations of the worn surface of the pin used for wear testing under lubrication revealed that the surface was generally much smoother compared to the pin after the dry test. Fig. 13a shows the pin surface after wear testing under a load of 8 kg for 160 h. In the areas where the wear was heavy, adhesive wear can be found (Fig. 13b), and the removal of the material by a plastic tearing mechanism is visible. The reason for this difference is still unclear.

Lubrication also changes the attachment condition of the debris from the coating to the pin. Very little debris was found on either surface. Only a few spots on the pin surface could be seen where debris from the EN coating was visible (Fig. 14a,b). The X-ray mapping in Fig. 14b showed that this piece of debris was cut (or ploughed) from the EN coating of the flat specimen.

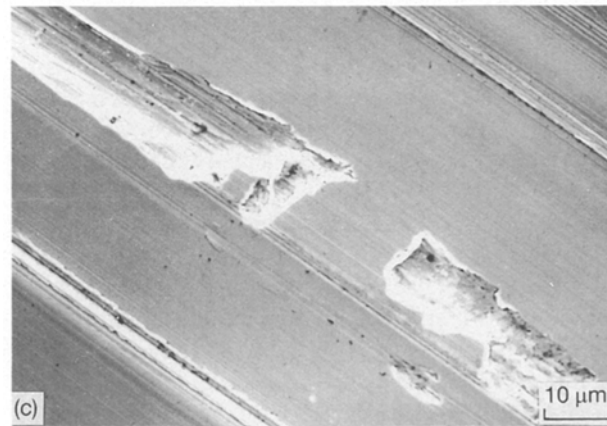
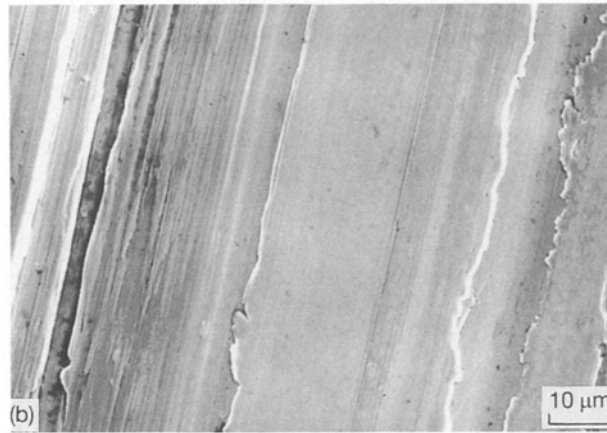
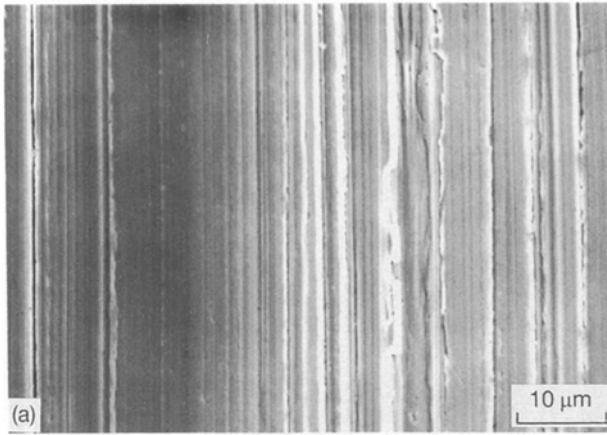


Figure 12 Scanning electron micrographs of the worn coating surface under lubricating conditions: (a) 2.0 kg, 87 h; (b) 8.0 kg, 45 h; (c) 8.0 kg, 115 h.

4. Discussion

4.1. Wear performance of EN coating

The wear performance has been evaluated between two counterfaces: EN, which has been given a heat treatment for maximum hardness, and a comparatively softer matrix of cast iron with graphite nodules. This is a desired combination for application where lubricated metal to metal wear is encountered, such as in diesel engine liners.

The experimental conditions for wear testing in this work may not have been exactly the same as real conditions for the cylinder liner of an engine. However, the main features such as reciprocating movement, applied load and lubrication have been main-

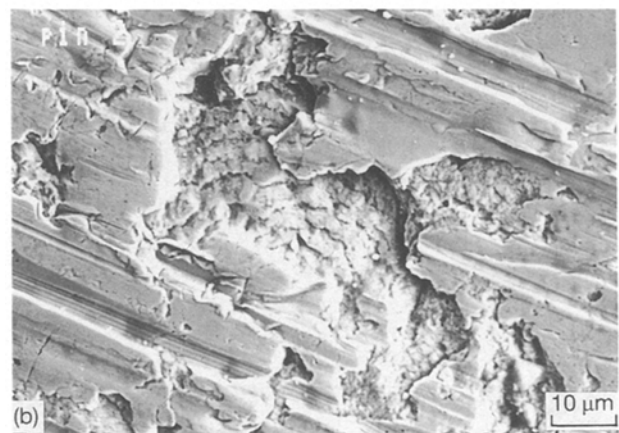
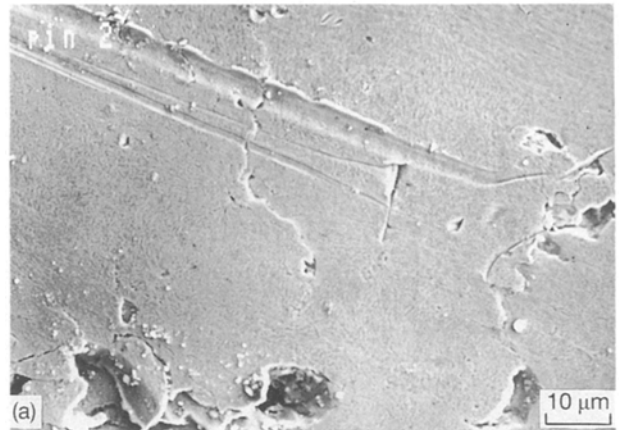


Figure 13(a) Scanning electron micrograph of the worn surface of the pin used for lubricated wear test under 8.0 kg load for 160 h. Delamination and tearing is evident. (b) An area of heavy wear on the pin surface. Plastic tearing and delamination is present.

tained. The results show that the EN coating is not suitable for use under unlubricated conditions when the counterface is iron. The weight loss of the coating under various loads showed a linear relation with time (Fig. 4(a), or cycles of distance tested) similar to the published results [5, 7, 16]. The rate of removal, as shown in Fig. 15 progressively increased with load, indicating a power-law relationship between load and the material removal rate. In sliding wear, where adhesive wear is playing a predominant note, the loss of material volume is expressed as [16] $V = kPs$, where P is the load, s is the sliding distance and k is a constant. This indicates that the wear is proportional to the applied load, P , and total sliding distance, s . It also demonstrates that for the same velocity, ds/dt , the removal rate, dV/dt should be directly proportional to the applied load, P . However, the present results do not indicate so, and it appears that applied load does affect the rate of material removal. Perhaps under high load conditions, worn debris experiences severe work hardening, thus causing pronounced adhesive wear [16, 17]. As far as the mechanism of wear is concerned, it has been established that adhesive wear occurs throughout the test regime. No such observation was made in the past for the dry tests under the load range of 0.5–2.0 kg, instead a straight line for each test was noted. This is probably because either the wear rate by

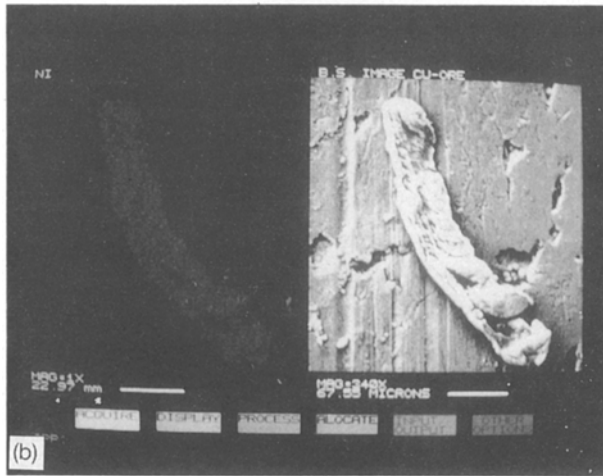


Figure 14(a) Deposition of Ni-P flake on to the pin surface. (b) An EDS X-ray map of nickel for the flake.

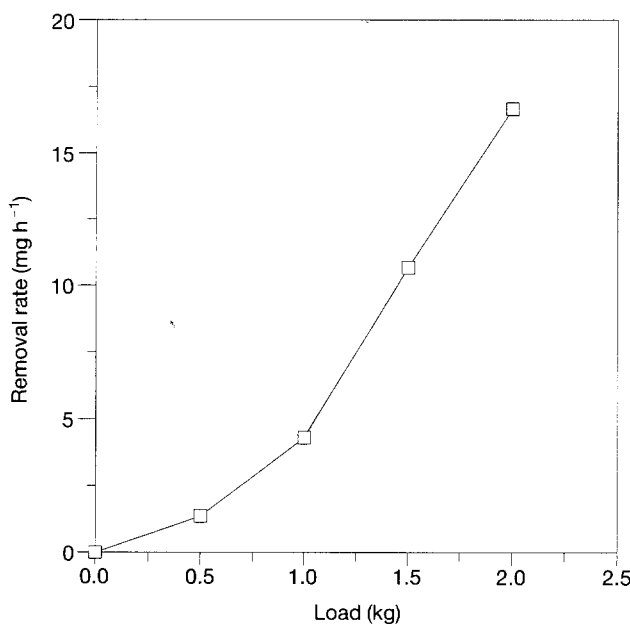


Figure 15 Removal rate of the coating versus the applied load for the unlubricated tests.

adhesion after running-in is still relatively high, or the wear rate during the running-in period is low.

Under lubricated testing conditions there is very little weight loss in the EN coating after long tests. It has been shown that the EN coating exhibits some

ductility under a compressive stress condition. Instead of material removal, polishing and plastic deformation takes place, as shown in several micrographs (Fig. 12a-c). However, the contact pressure has to be high enough to cause this flattening or polishing of the EN coating. The reason why the specimen has more weight loss under lighter load (2.0 kg in this case) is probably that the contact pressure was not high enough, did not cause enough plastic deformation, and instead merely cut off material from those asperities which were high above the surface (Fig. 4b). More work may have to be carefully done to confirm this phenomenon.

4.2. The mechanism of wear of EN coating

There are two main mechanisms operating in the process of wear of the EN coating: abrasive wear (abrasion) and adhesive wear (adhesion). Abrasion, characterized by scratching or ploughing in the direction of relative motion operates first (Figs 6a, 7a, 12) and throughout the test. Plastic deformation takes place during abrasion, appearing either as wear track lips (Fig. 12) or as plastic cutting debris (Fig. 14a). It is noteworthy that (1) the EN coating is able to experience plastic deformation (flattening) or to exhibit good ductility despite its high hardness under certain load conditions, and (2) the EN coating can be abrasively worn even with a softer counterface material.

As an accompanying phenomenon, cracks appear at the bottom of some grooves in the EN coating when the surface rubbing has proceeded for some time, before spalling starts (Fig. 6b, c). This is somewhat different from Tomlinson's work [17], in which it was shown that "after heat treatment the electroless nickel showed no signs of cracking or delamination". The contradiction may come from the differences in load (1500 g in this work compared to 50 g), in speed (0.524 m s^{-1} compared to 0.28 m s^{-1}), or in counterface material (nodular cast iron versus nitrided steel), but may be due mainly to the pattern of relative motion of the two contacting surfaces, i.e. reciprocating in this work and unidirectional in [17]. Therefore, it can be derived that in the present case, the cracks arose mainly from the fatigue of the surface or sub-surface layers due to the reciprocating action under high stress.

It does not appear that abrasion is the dominant mechanism responsible for the weight loss and the surface roughening in this work, because all the worn out surfaces showed the characteristic pattern of adhesive wear. Adhesive wear (see Fig. 8a and b) results in more weight loss, as can be seen in Fig. 8a. But the topography of the surface and the tearing-off process still needs to be scrutinized. Generally speaking, the first step of any adhesive wear is adherence of two asperities between the two contacting surfaces by a sub-process of frictional welding. Then, during the subsequent sliding stroke, the welded pieces break near the contact surface leaving a piece of material on the counterface. Obviously the breaking or the tearing-off of the small piece takes place at a high speed, something like an impact shearing, which makes the

tearing a brittle one. The smooth, more or less flat surfaces on the bottom of the caves shown in Fig. 8a and c support the above mechanism. There are particles stuck to the bottom of the adhesion-damaged caves in Fig. 8a–c. It is still unclear where they came from and when.

The main differences in morphology between adhesion caves and surface fatigue spalling (called delamination in [7, 8]) are that (1) the edge of the former is irregular but the latter shows a straight or flat edge, and (2) the latter is always connected with some cracks while the former is not (compare Fig. 8a–c with Fig. 6b and c).

As mentioned before, redeposition of debris from either surface is evidence of adhesion, and is also an auxiliary mechanism of adhesion. The redeposit will eventually deform and break away, causing additional weight loss, and will probably adhere to one of the surfaces and break away again.

4.3. The wear sequence

It was found through sequential observations of the worn surfaces after different cycles of wear testing, that the wear sequences under the conditions of this work are: (1) initial abrasive wear; (2) adhesive wear starts later when the fresh surface areas in contact, owing to the roughening by abrasive wear, are high enough to cause adhesion; (3) redeposition of debris starts even later when there is sufficient debris. This is quite consistent with the existing theories of metal to metal wear [16, 17]; however, the “rate of wear” versus the applied load appears to follow the power law. Redeposition of debris and subsequent redetachment of the redeposited debris can be considered as an auxiliary form of adhesive wear. A dynamic equilibrium between redeposition/redetachment may build up after a period of running time; (4) abrasive wear operates throughout the whole wear process whenever the conditions are suitable.

It would seem reasonable that there is no pure adhesive wear existing: whenever two surfaces slide relative to each other, the first form of wear that takes place would be abrasion, and adhesion will take place afterwards when the adhering of some asperities on the two surfaces is possible. This is especially true when reciprocating motion is occurring. On the contrary, pure abrasive wear is possible in many cases.

4.4. The function of lubrication

It can easily be seen by comparing Fig. 12 with Figs 6 and 8, that all the specimens tested under lubrication still remained in the abrasive wear stage, although the loads used were much higher and the time span tested was much longer than that used in unlubricated testing. This indicates that lubrication, by forming a thin oil film separating the two surfaces, makes it very difficult for adhesive wear to take place, and postpones adhesive wear, thereby reducing the weight loss drastically. Limited by the time span used for the experiment (longest time 167 h), it is very hard to predict when the adhesive wear would start, if at all.

Maintaining a continuous oil film is very important in ensuring the essential function of surface separation.

5. Conclusion

EN coating shows excellent wear performance under lubrication, which makes it a promising candidate coating for railway engine liners. The mechanisms involved, in sequence, for the whole process of wear of EN coatings are: abrasive wear first, adhesive wear second, redeposition/redetachment third, and abrasive wear possibly operating throughout the whole wear process. The two stages of adhesive wear (and tearing/breaking), take place sequentially in one stroke (the speed of relative motion is high at 0.5 m s^{-1}), hence the tearing has the feature of brittle fracture with the bottom surface of the adhesion caves being more or less flat. The rate of wear is also found to be non-linear with the applied load.

The function of the lubricant is to separate the two surfaces which are originally in contact and to protect them from adhesion. Abrasive wear would not cause much weight loss because the EN coating exhibits ductility under multi-axial pressure and compressive stresses cause the coating to flatten rather than plough it off the substrate.

Acknowledgements

The authors thank the Canadian International Development Agency for supporting the research, Mr John Doerksen, Manager, Cadorth Plating Ltd, Winnipeg, MB, for providing the plated specimens and providing valuable technical guidance, Dr M. C. Chaturvedi for his encouragement on this project, and Mr J. Vandorp and Mr D. Mardis for their help in the experimental work.

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Received 9 September 1992
and accepted 8 June 1993