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HYDRODYNAMIC AIR LUBRICATED COMPLIANT SURFACE BEARING FOR AN AUTOMOTIVE GAS TURBINE ENGINE

II - MATERIALS AND COATINGS

Bharat Bhushan, David Ruscitto, and Stanley Gray
Mechanical Technology Incorporated
Latham, New York 12110

July 1978

Prepared for the
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center
Cleveland, Ohio 44135

Contract NAS 3-19427

As a part of the
U.S. DEPARTMENT OF ENERGY
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13

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16. Abstract
The objective of the program was to select and develop material coatings good to service temperatures of either 540° C (1000° F) or 650° C (1200° F) for an air-lubricated, compliant journal bearing for an automotive gas turbine engine. The selected combinations have been exposed to service test temperatures of 540° C or 650° C for 300 hours, and to 10 temperature cycles from room temperature to the service test temperatures. Selected coatings were put on journal and partial-arc foils and tested in start-stop cycle tests at 14 kPa (2 psi) loading for 2000 cycles. Half of the test cycles were performed at a test chamber service temperature of 540° C (1000° F) or 650° C (1200° F); the other half were performed at room temperature. Based on test results, the following combinations and their service temperature limitations were recommended: HL-800^{TM} (CdO and Graphite) on foil versus Chrome Carbide on Journal up to 370° C (700° F); NASA PS 120 (Tribaloy 400, Silver and CaF_2) on journal versus uncoated foil up to 540° C (1000° F); and Kaman DES on journal and foil up to 640° C (1200° F). Kaman DES coating system has been further tested successfully at 35 kPa (5 psi) loading for 2000 start-stop cycles.

17. Key Words (Suggested by Author(s))
Hydresil^{TM} bearing; Compliant foil bearing; Journal; Automotive gas turbine engine; Friction; Wear; Surface coatings; High temperature

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SUMMARY

The objective of the program was to develop material coatings for an air-lubricated compliant journal bearing for an automotive gas-turbine engine.

Based on past experience, A-286 was selected as the journal base material and Inconel X-750 was selected as the foil base material. Hard, wear-resistant coatings and soft, low shear strength coatings were selected. The coatings were expected to function in either 540°C (1000°F) or 650°C (1200°F) ambient. Soft lubricant coatings are generally limited in temperature. Therefore, most of the emphasis has been on the hard, wear-resistant coatings.

The coatings on the journal coupons were applied by sputtering, plasma spraying, detonation gun process, electroplating, fusion process, diffusion process and other proprietary processes. For the foils, since they are thin, flame spraying and diffusion techniques could not be used, but all other processes were used. Based on previous experience, a variety of coatings were selected for the tests. The coating materials covered were: TiC, B_4C, Cr_3C_2, WC, SiC, CrB_2, TiB_2, Cr_2O_3, Al_2O_3, Si_3N_4, Tribaloy 800, CaF_2, CaF_2-BaF_2 eutectic, Ni-Co, Ag, CdO and Graphite, and other proprietary compounds.

The coatings on test coupons were subjected to static oven screening tests. The tests were conducted to screen out these coatings which could not stand the extended thermal exposure and thermal cycling. The tests consisted of exposure of material samples in an oven for 300 hours duration at service temperatures of 540°C or 650°C, and exposure to 10 temperature cycles from room temperature of the service temperatures.

After the oven tests, the specimens were examined using the following techniques: flex bending test, tape test, scratch test, superficial hardness test, weight change and metallurgical examinations. The surface morphology of coatings was examined in the Scanning Electron Microscope (SEM). Some coated specimens were sectioned and examined in the SEM for interfacial bond. An X-Ray Energy Dispersive Analyzer (X-REDA) was utilized to determine the elemental composition of specific particles or structures on the samples. Analysis by reflection electron diffraction was made to determine any change in the chemical composition of the coatings during the oven tests.

Based on the specimen examinations, the following coatings were recommended for the start-stop cycle tests: TiC (sputtered), Cr_2O_3 (sputtered), Si_3N_4 (sputtered), CdO and Graphite (fused) - HL-800, Kaman DES - proprietary coating, CrB_2 (plasma sprayed), Cr_3C_2 (detonation gun) and NASA PS 106 (plasma sprayed).

The start-stop cycle tests consisted of testing partial-arc bearings at 14 kPa (2 psi) loading based on bearing projected area for 1000 start-stop cycles at room temperature and 1000 start-stop cycles at the service temperatures. Static and dynamic breakaway torques were recorded during the tests. Based on torque data, visual examinations and metallurgical examinations of the bearing surfaces, the following combinations were recommended: HL-800™ (CdO and Graphite) on foil versus Linde Ni-Cr bonded Cr_3C_2 on journal up to 370°C (700°F); Plasma sprayed NASA PS 120 (60% T400, 20% Ag and 20% CaF_2) with Ni-Aluminide undercoat on journal versus uncoated heat-treated foil up to 540°C (1000°F); and Kaman DES on journal and foil up to 650°C (1200°F).

The most promising combination (Kaman DES versus itself) was tested at 35 kPa (5 psi) loading in partial-arc bearing form. This coating combination successfully completed 2000 start-stop cycles. Some of the coating was worn under the loaded area. There was some coating left on the grain boundaries, and the remaining area was oxidized during exposure to higher temperature.

This combination was further tested at 14 kPa (2 psi) loading as a complete bearing. It completed 1000 start-stop cycles. A considerable amount of loose wear debris collected at the interface and, apparently, could not easily escape. As a result, it damaged the surfaces.

I. INTRODUCTION

This report describes Part II of a technology program performed for the NASA Lewis Research Center for the development of hydrodynamic air lubricated journal bearings for an automotive gas turbine engine. Part I of the program (Reference 1) had focused on advancing compliant surface journal bearing technology by providing design information through an experimental and analytical effort. Part II of the program has investigated and tested materials and coatings for compliant surface bearings and journals good to either 540°C (1000°F) or 650°C (1200°F) environment.

BACKGROUND

The compliant foil air bearing offers the ideal solution to the problem of a support bearing for a high-speed, high-performance, vehicular, gas-turbine engine rotor. This is particularly so at the turbine end where the temperatures are at such a level as to preclude the use of liquid lubricants because of short-term vaporization, longer term coking of oil seals and bearings and the possibility of fire hazards. The use of a foil air bearing also offers reduced power losses due to lower frictional drag at high speed.

In the recent tasks of the subject technology program, the MTI Hydresil^{TM} compliant hydrodynamic journal foil bearing was extensively evaluated to achieve a greater understanding of the characteristics of the foil type of air bearing. This was done from dynamic measurements of film thickness in the gas film around the bearing and the determination of maximum load capacity at elevated temperatures.

Equally important to the success of the hydrodynamic foil bearing, particularly when having to operate over a temperature range from normal ambient up to 650°C (1200°F) and to speeds up to 60,000 rpm, is the selection of the journal and foil substrate materials and of wear resistant and lubricant coatings. The materials and coatings must be suitable for the range of environmental temperatures, be capable of surviving the start and stop sliding contact cycles prior to rotor lift-off and at touchdown, and survive occasional short-time high-speed rubs under representative loading of the engine, rotor weight at the bearings and in this case loading was 14 kPa (2 psi) based on bearing projected area.

Limited evaluations of materials and coatings for foil bearings at temperatures up to 540°C (1000°F) were made at MTI (Reference 2).

In the present program, a more intensive study has been undertaken in selecting a number of candidate materials and coatings, in screening, dynamic rig testing at temperatures up to 650°C (1200°F) and in the determination of specimen performance. In this work the dynamic testing consisted of the simulation of start and stop sliding contacts. The largest variable has been the selection of wear resistant and lubricant coatings.

PROGRAM OBJECTIVE

The objective of the program for the development of an air-lubricated, compliant journal bearing for an automotive gas-turbine engine was to achieve improved materials and surface coatings capable of meeting the start-stop sliding duties at turbine end temperature and loading conditions. This objective has been accomplished. A number of candidate materials and coatings for the bearing foils and journal were selected, subjected to static oven screening tests, and dynamically tested in a modified test rig under simulated engine operating conditions in order to arrive at optimum solutions.

PROGRAM APPROACH

In the continuation tasks of the program for the development of an air-lubricated, compliant journal bearing for an automotive gas turbine engine, an experimental study has been performed to achieve improved materials and surface coatings capable of start-stop sliding operation under temperatures and loadings representative of the turbine end of the engine.

The program has accomplished the following significant activities on materials and coatings suitable for bearing foils and journals:

- Select and prepare some 30 specimen combinations and statically oven test them to temperatures of 540°C (1000°F) and 650°C (1200°F).
- Modify the MTI journal foil bearing and material tester used in the earlier tasks of this program, for endurance testing of partial-arc foil bearing specimens under start-stop cycles to temperatures of 540°C (1000°F) and 650°C (1200°F).
- Dynamically test some 20 material and coating specimen combinations selected from the static screening tests, under start-stop cycles at 14 kPa (2 psi) loading and at 540°C (1000°F) and 650°C (1200°F) temperature levels.
- Perform additional start-stop cycle tests with the most promising candidate at 35 kPa (5 psi) loading and elevated temperatures.
- Build the most promising candidate into complete bearings and conduct elevated temperature start-stop cycle tests to ensure that no coating migration occurs which could cause jamming in the bearing clearance.

Extremely important in the proposed program has been the effort to ensure the quality of specimens, care in screening, planning of the test matrices and the quality of specimen analysis.

The authors wish to express their appreciation to S. Frank Murray, Research Engineer, Institute for Wear Control Research, RPI, for his invaluable guidance in choosing the matrix of coatings to be investigated.

II. MATERIAL SELECTION AND PREPARATION

MATERIAL SURVEY

An extensive literature search was conducted to select the possible candidate materials for journal and foil substrates and coatings. The selection was based on the literature search, past experience and consultations with personnel at RPI and NASA, and included some new promising materials, previously not explored. To keep the number of variables to a minimum, one journal substrate and one foil substrate were selected; but a large number of coatings to be applied on foil and journal substrates were included.

The selected materials and coatings have to meet a wide range of exacting requirements over the operating temperature range, these include:

- Good mechanical properties (hardness, yield strength, ductility and rupture strength) at elevated temperatures
- Dimensional stability
- Corrosion and oxidation resistance
- Compatibility between foil and journal surface coatings
- Shock resistance; thermal and impact
- Compatibility between substrate and surface coatings
- Good machining and formability characteristics
- High thermal conductivity to rapidly dissipate frictional heat from the interface
- Thermal properties compatible with the coatings in order to minimize interface stresses
- Good anti-galling characteristics should the coatings be penetrated
- Good spring properties in case of foils
- Formation of soft, protective oxide films
- Low surface friction and longer wear life
- Availability at acceptable cost

Some of the criteria mentioned previously need some clarification. Although hardness, per se, is a poor criterion (since many hard coatings are subjected to excessive abrasive wear), experience has shown that for a given material, the higher the hardness, the smaller the true area of contact and, therefore, the less likelihood of gross interactions between the surfaces. In addition, it should be noted that low shear strength solid lubricant films are usually more effective on hard substrates.

There is considerable evidence to show that coating materials having hexagonal crystal structures and layered lattice structures tend to wear-in smoothly with much less tendency for surface damage than those materials which fall into other crystal classifications. While a hexagonal structure and layered lattice structure are no guarantee of sliding compatibility, it has been established that the most promising gas-bearing materials either have these structures or contain some constituent with these structures.

Certain naturally-occurring oxide films can provide protection for metallic surfaces sliding in contact. In addition, many ceramic combinations can interact to form eutectic compounds which behave as low shear strength solid films. In most cases, such preferential interactions are limited to high-temperature or high-energy input conditions, but the possibility of deriving some benefit from this type of reaction should not be overlooked since this effect would be most evident during very severe operating conditions such as a high speed rub at high ambient temperatures.

SELECTION OF MATERIALS

Foil Substrate Material Selection

The commercially available superalloys for high-temperature applications which can be used for foil substrate are Inconel X-750, Inconel 718, 17-7 PH (TH 1050), Hastelloy B, Rene 41, and Haynes 25. Their relevant mechanical and thermal properties, and chemical compositions are shown in Tables II.1 and II.2, and Figures II.1 to II.4. To date, Inconel X-750 has been used as the preferred high-temperature foil material because of its excellent physical properties at temperatures up to at least 650°C (1200°F), ease of heat treatment, good spring properties, availability and cost. However, this alloy is difficult to coat

TABLE II.1

NOMINAL CHEMICAL COMPOSITION OF CANDIDATE SUBSTRATE MATERIALS

Alloy Designation	Nominal Chemical Composition, Wt. %													
	Ni	Co	C	Mn	Si	Cr	Mo	W	Cb	Fe	Ti	Al	B	S	Other
Inconel X-750	Bal		.04	.70	.30	15.0			.90	6.8	2.5	.80			
Inconel 718	Bal		.04	.20	.30	18.6	3.1		5.0	18.5	.90	.40			
Hastelloy B	Bal	2.5	.05	1.0	1.0	1.0	26.0-30.0			4.0-7.0				.03	.04P .2 - .6V
A-286*	26.0		.05	1.35	.50	15.0	1.3			Bal	2.0	0.2	.015		
17-4 PH*	3.0-5.0		.07	1.0	1.0	15.5-17.5				Bal				.03	.04P, 3.0-5.0 Cu, .15-.45, Ta &Cb
17-7 PH	6.5-7.75		.09	1.0	1.0	16.0-18.0				Bal		.75-1.5		.03	.04P
Rene 41	Bal	11.0	.09			19.0	10.0				3.1	1.5	.005		
Haynes 25	9.0-11.0	Bal	.05-.15	1.0-2.0	1.0	19.0-21.0		14.0 16.0		3.0					

*Suitable only for Journal Material

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TABLE II.2

MECHANICAL AND THERMAL PROPERTIES OF CANDIDATE SUBSTRATE MATERIALS

Alloy Designation	Data Presented		Heat Condition	Temperature °C(°F)	1000 Hour Rupture Strength MPa(ksi)	Modulus of Elasticity GPa (10^6 psi)	Coeff. of Thermal Expansion 21°C to Temp. x 10^{-6}/°C (70°F to Temp. x 10^{-6}/°F)	Thermal Conductivity W/(m.K) (BTU/ft^2/hr/°F/in.)
	Sheet	Bar						
Inconel X-750	X		Cold Rolled + 705°C/20 Hours/AC	RT(21°C) 650(1200) 760(1400)	- 365(53) 145(21)			
		X	1150/2/AC + 845/24/AC + 705/20/AC	RT 650(1200) 760(1400)	- 469(68) 207(30)	214(31.0) 176(25.5) 166(24.0)	12.53(6.96) 95°C 15.14(8.41) 15.91(8.84)	11.95(83) 20.6(143) 22.2(154)
Inconel X-718	X		925/1/AC + 720/8/FC 55°/hr to 620/AC	RT 650(1200) 760(1400)	- 586(85) 172(25)			
		X	980/1/AC + 720/8/FC 55°/hr to 1150/AC	RT 650(1200) 760(1400)	- 592(86) 172(25)	200(29.0) 164(23.7) 154(22.3)	12.78(7.10) 95°C 15.12(8.40) 16.02(8.90)	11.2 (78) 21.3(148) 23.2(161)
Rene 41	X		1065/½/AC + 760/16/AC	RT 650(1200) 760(1400)	- 551(80) 386(56)			
		X	1065/4/AC + 760/16/AC	RT 650(1200) 760(1400)	- 689(100) 510(74)	220(31.9) 182(26.4) 173(25.1)	11.94(6.63) 95°C 14.00(7.80) 14.76(8.20)	8.9 (62) 19.6(136) 21.3(148)
Haynes 25	X		1175/1/RAC Cold Workable	RT 650(1200) 760(1400)	- - 121(17.5) 815°C	225(32.6) 174(25.2) 163(23.7)	12.29(6.83) 95°C 14.76(8.20) 15.48(8.60)	9.4 (65) 21.5(149) 23.6(164)
		X	1175/1/RAC Cold Workable	RT 650(1200) 760(1400)	- - 208(30.2) 730°C	200(29.0) 159(23.0) 540°C		
17-4 PH (H 1150)	X		620/4/AC	RT 425(800) 480(900)	- 689(100) 483(71)	200(29.0) 159(23.0) 540°C	11.90(6.60) 13.00(7.20) 13.00(7.20)	16.3(113) 22.2(154) 23.0(160)
17-7 PH (TH 1050)		X	565/1½/AC	RT 425(800)	- 359(52) 480°C	196(28.5) 179(26.0) 315°C	10.10(5.60) 11.90(6.60)	14.7(102) 20.7(144)
Hastelloy B	X		1150/2/RAC Cold Workable	RT 650(1200) 760(1400)	- 593(86) 172(25)	203(29.5) 190(27.5) 161(23.4)	11.54(6.41) 95°C 12.00(6.66) 14.00(7.78)	12.2 (85) 16.4(114)
A-286		X	980/1/OQ + 720/16/AC	RT 650(1200) 760(1400)	- 317(46) 104(15)	200(29.1) 153(22.2) 142(20.6)	16.51(9.17) 17.78(9.88) 18.58(10.32)	12.7 (88) 24.8(172)

AC - Air Cooled
RAC - Rapid Air Cooled
OQ - Oil Quenched

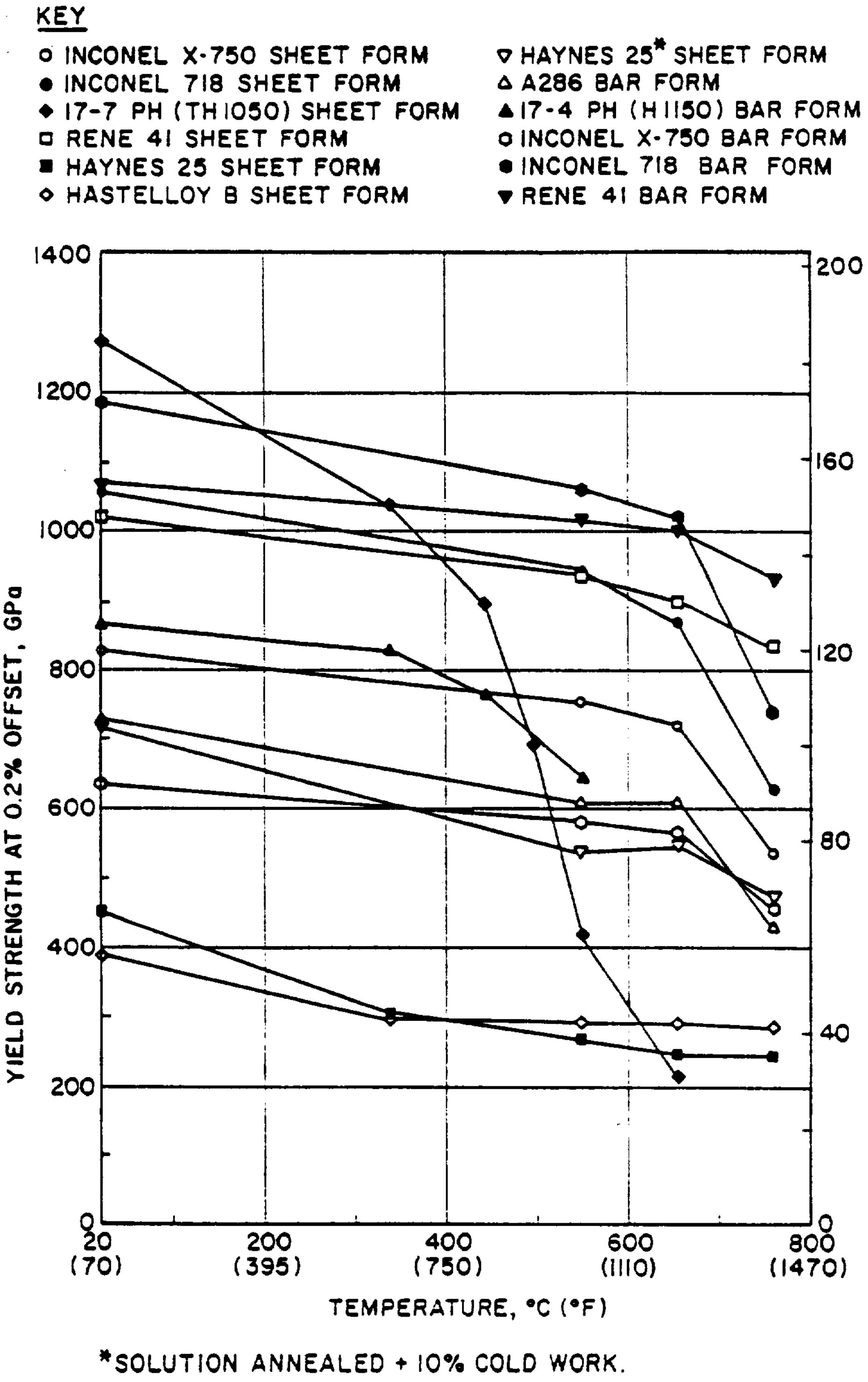
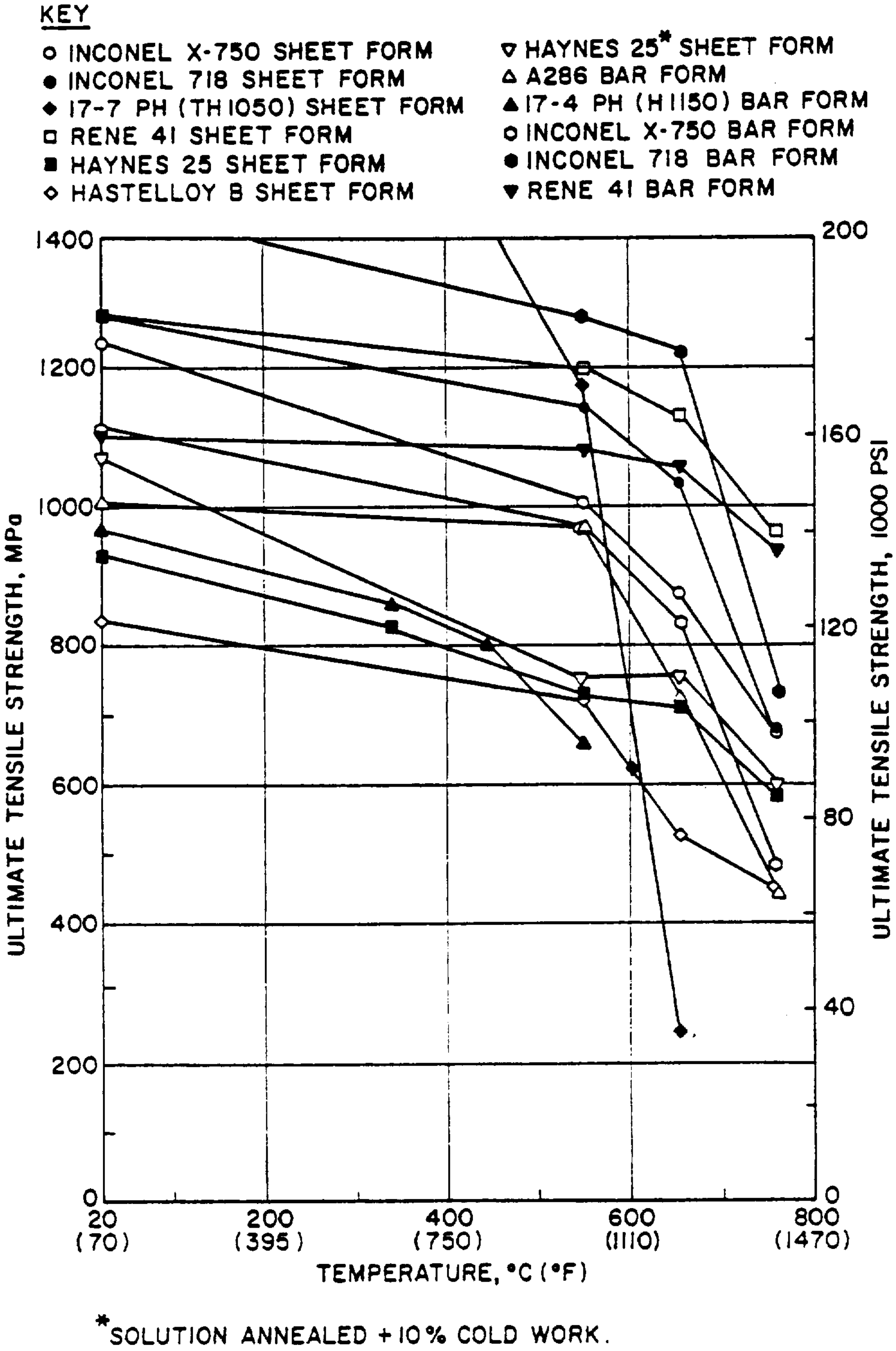


Fig. II.1 Yield Strength at 0.2 Percent Offset Versus Temperature



*SOLUTION ANNEALED + 10% COLD WORK.

Fig. II.2 Ultimate Tensile Strength Versus Temperature

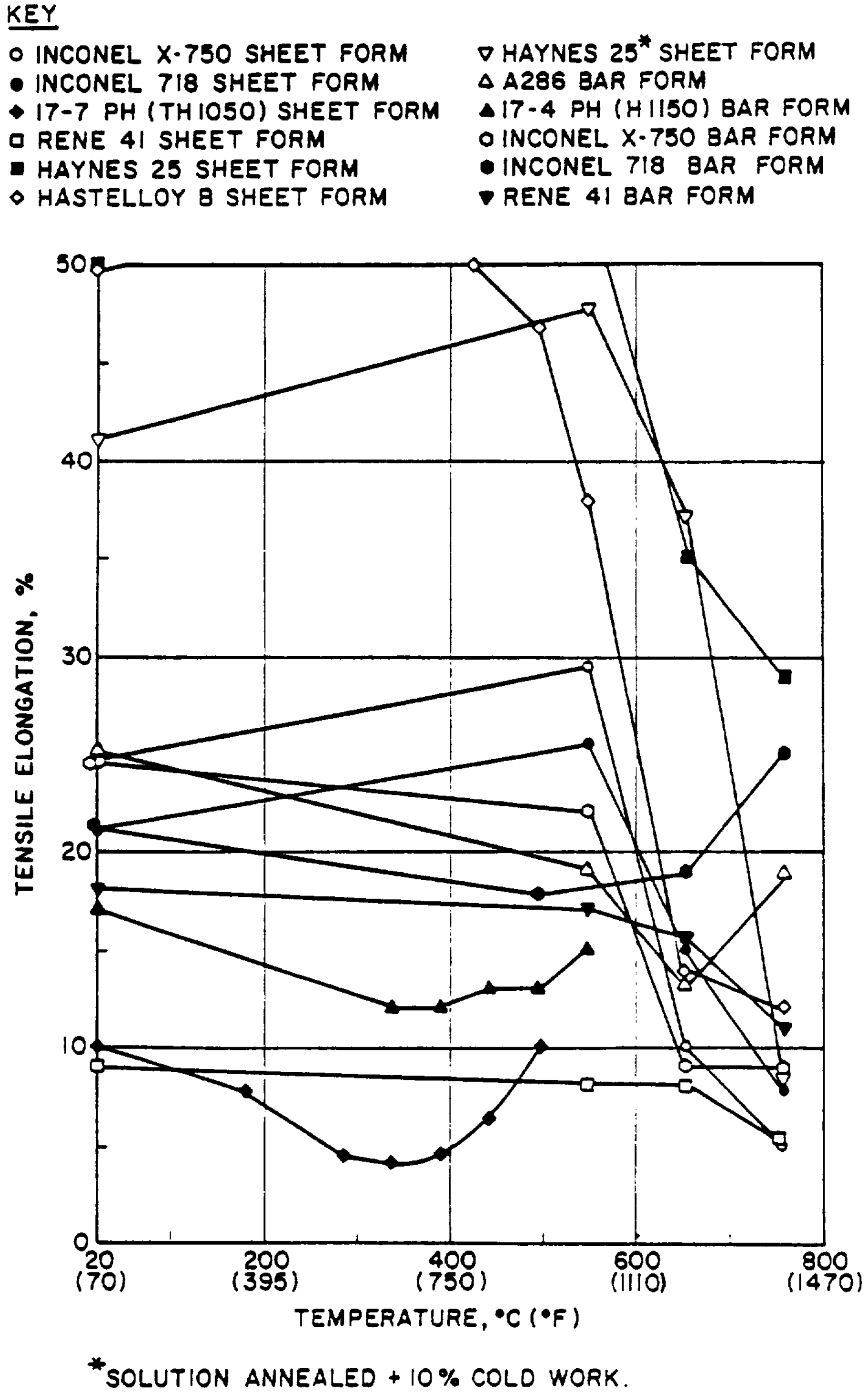


Fig. II.3 Elongation Versus Temperature

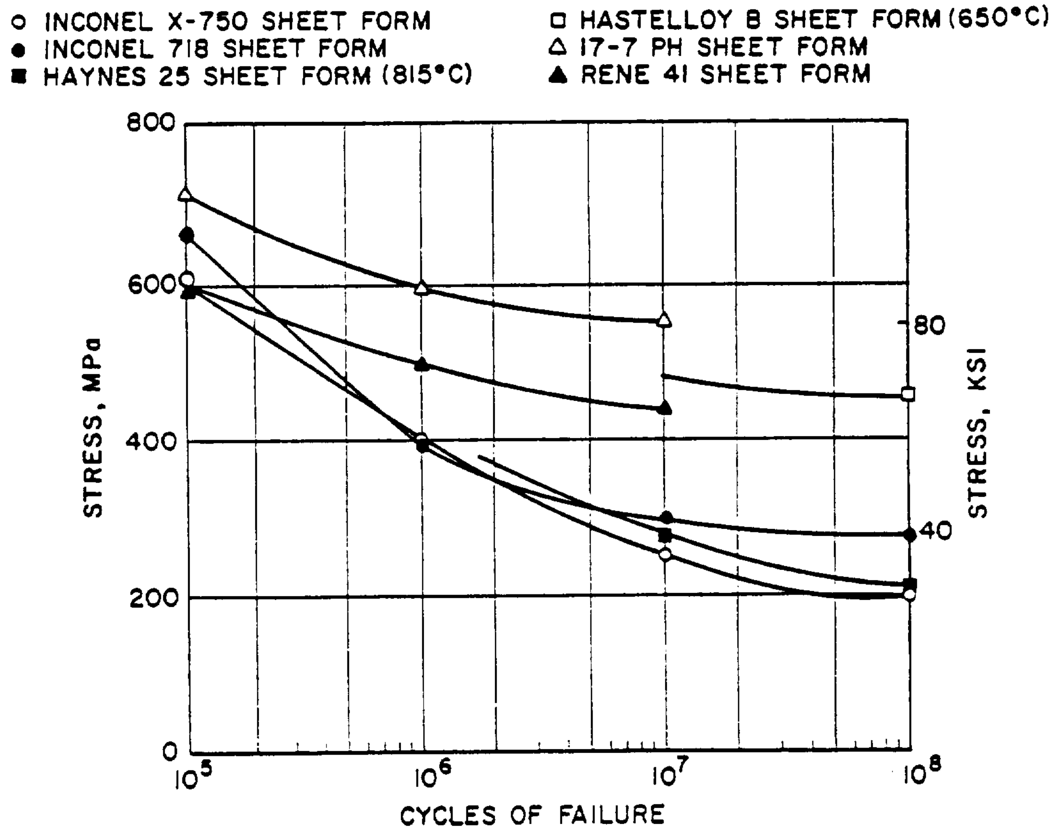


Fig. II.4 Fatigue Strength of Materials

effectively with solid lubricant or hard wear-resistant materials because of its poor amendability to chemical and mechanical pretreatment. In addition, nickel-base alloys usually experience surface damage (galling) as soon as any part of the coating is penetrated.

Previous experience has shown that alloys which contain molybdenum and/or cobalt as a major alloying elements have superior sliding characteristics, and do not gall as much as nickel-base alloys. This is probably because of the formation of beneficial oxide films. Alloys high in molybdenum, such as Hastelloy B and cobalt-base alloy Haynes 25, have a somewhat better sliding characteristic and probably can survive until a substantial percentage of coating has been worn away.

However, from Figure II.1, it is observed that the yield strengths of Hastelloy B and Haynes 25 are low. A simple analysis using the continuous beam theory (Appendix A) has shown that, assuming an average load of 210kPa (30 psi) on the top foil resulting from high speed dynamic loading, the stresses developed in the top foil are approximately 280 MPa (40,000 psi). Therefore, from a strength point of view, Hastelloy B and Haynes 25 would be marginal. Fatigue properties of both materials are acceptable (Figure II.4). Neither of these materials can be age hardened. However, the tensile properties can be increased by prior cold work. In the case of Haynes 25 with 10 percent cold work, there is appreciable increase in the yield and tensile strengths (Figures II.1 and II.2) but there is appreciable loss of ductility (Figure II.3). A compromise has to be made between desired ductility and tensile properties. Also special cold working procedures make availability and cost a problem. Therefore, it is not recommended as a suitable material. After cold working, Hastelloy B experiences an increase in yield strength but there is a considerable loss of ductility at high temperature. However aging of this material for a long time at high temperature increases its yield strength but reduces ductility; and hence it is not a good high temperature candidate. Hastelloy S and Hastelloy X (nickel base), although not considered here, have the same problem.

The precipitation hardening treatment of 17-7 PH material is carried out in the range of 480°C (900°F) to 570°C (1050°F). Therefore, 17-7 PH (TH 1050) would lose its strength at temperatures above 570°C (1050°F) and consequently would not be acceptable (for a similar reason 17-4 PH would not be suitable as a journal material). As far as mechanical strength is concerned, Inconel X-750, Inconel 718 and Rene 41 would be acceptable. Rene 41 has low ductility (Figure II.3) but the fatigue properties are quite good (Figure II.4). Fatigue properties of Inconel X-750 and 718 are marginal. Of these three materials, Inconel X-750 was selected and recommended because of prior experience with the material in foil bearings, acceptable mechanical and thermal properties, ease of heat treatment, formability, availability in foil form and cost. Dependence has to be placed on the performance of the coatings to overcome the lower galling resistance of the Inconel material.

Journal Substrate Material Selection

The candidate journal materials, based on design strength considerations, are Inconel X-750, Inconel 718, A-286, Rene 41, and 17-4 PH (H 1150). As mentioned previously, 17-4 PH would be unsuitable from the strength point of view at 650°C (1200°F). The coefficient of thermal expansion of Inconel X-750, Inconel 718 or Rene 41 is lower than A-286. Since the journals will be coated with materials having low coefficients of thermal expansion, it is advisable to choose a journal material having a low coefficient of thermal expansion to minimize interface stresses. Because of machinability, availability, and cost A-286 is commonly used in high temperature gas turbine applications and while there has to be a trade-off between these factors and the better high temperature strengths of the other materials, it is believed that A-286 has the necessary thermal and mechanical properties at 650°C (1200°F), and consequently it was selected as the journal material in the program.

Selection of Surface Coatings and Treatments

Surface coatings and treatments basically fall into two categories: hard wear-resistant coatings; and soft, low shear strength coatings. Coating techniques and treatments are reviewed in Figure II.5. It is recognized that coating effectiveness (bond strength, wear resistance and life) may depend on the coating technique. Therefore, it is important that potential coating materials should be put on by several techniques.

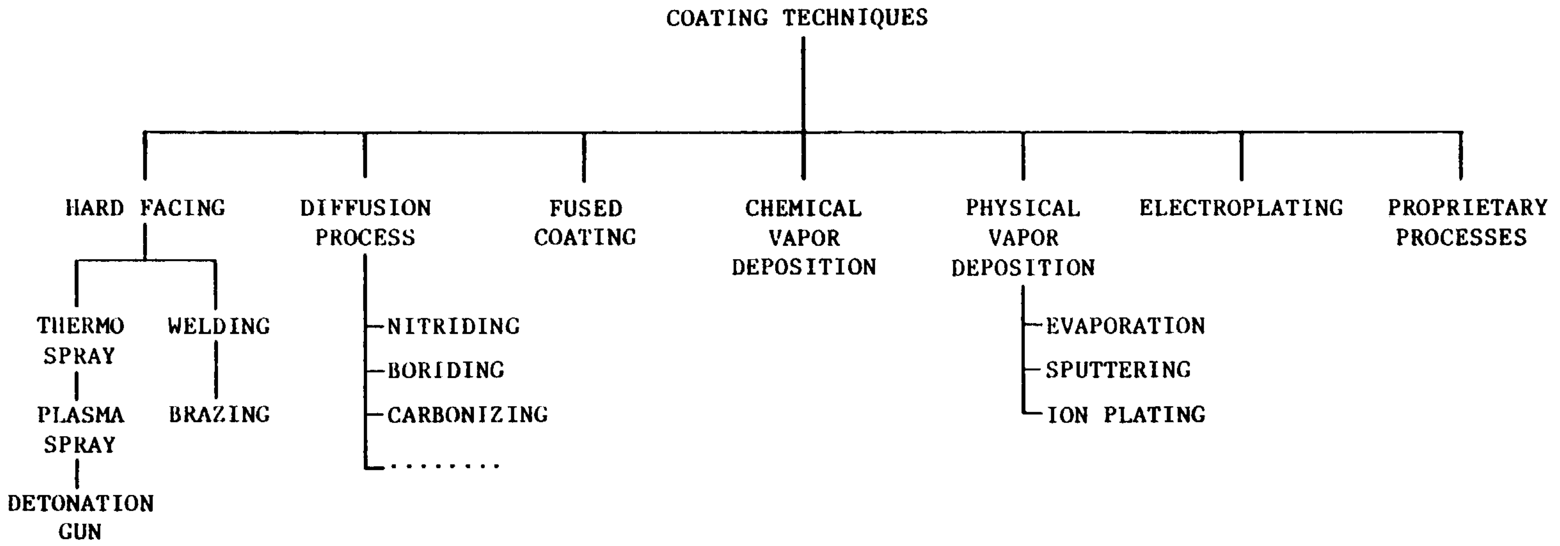


Fig. II.5 Coating Techniques

Depending on the coating technique and substrate, special substrate material preparation and undercoating may be required. Nickel-base alloys (e.g. Inconel 718 and Inconel X-750) are oxidation resistant and do not require undercoating. But iron-base alloys (e.g. A286) may oxidize, and the coating may therefore become less tenacious so that undercoat may be useful. Another use of undercoating is to match the thermal expansion of the mating surfaces. Nichrome (80 percent Ni and 20 percent Cr) and Ni-Aluminide (5 percent Al and 95 percent Ni) are commonly used as undercoating materials. Ni-Aluminide is less corrosion resistant in water but has better thermal shock resistance and about 20 percent higher bond strength than Nichrome. Temperature capabilities of Ni-Aluminide are up to 760°C (1400°F) while Nichrome can be used above 760°C (1400°F). In the case of very hard coating materials, Nichrome is usually added as a binder to improve the ductility.

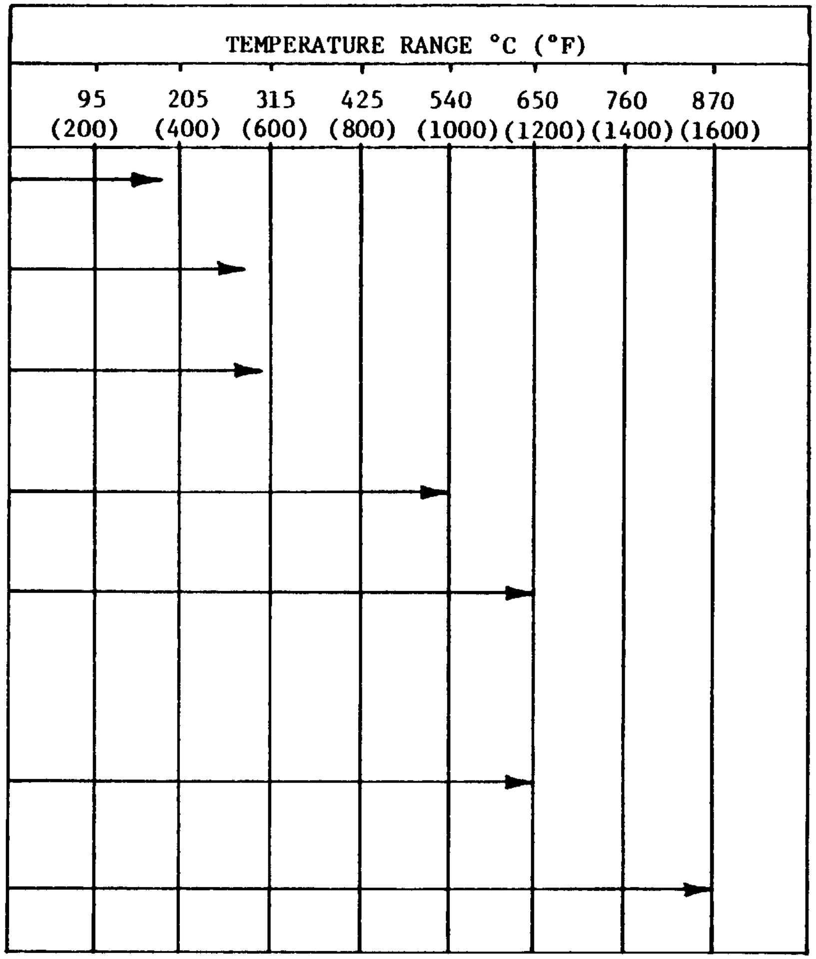
Considerable work has been done to select suitable coatings to improve the wear of bearing surfaces (References 2 to 12). Soft lubricant films have been successfully used up to 315-370°C (600-700°F) temperature range (References 5 to 10). Their status for foil bearing application at the start of the program is reviewed in Table II.3. The commonly available lubricants cannot be used at high temperatures for extended periods. Mostly one has to depend on hard-hard combinations. In marginally lubricated conditions, heavy emphasis is placed on hard coatings (References 2 to 4, 11, and 12). The status of foil bearing materials developmental work at the start of the program is reviewed in Table II.4.

Based on an extensive literature search and past experience at MTI, a large number of coating candidates were selected.

The initial selection of coating combinations for the study program is given in Table II.5. Coupons of foil and journal substrate materials were treated with the coatings and subjected to the static oven screening tests prior to further grading for start-stop testing. Some of the solid lubricant combinations and some of the hard coating combinations (the ones which oxidize at higher temperatures and as a result lubricate better) were believed to work better at higher interface temperatures and to have relatively higher coefficients of friction at low temperatures. It should be noted that the interface temperature depends on relative sliding speed and load. If the speeds are high, the interface

TABLE II.3

CANDIDATE SOLID LUBRICANT FILMS WHICH HAVE BEEN EVALUATED FOR FOIL BEARING APPLICATIONS AT THE START OF THE PROGRAM



Candidate	Status
Modified Teflon	Extensively Evaluated
Teflon S	Extensively Evaluated
Resin-Bonded MoS_2 (Several Compositions)	Extensively Evaluated
Silicate-Bonded MoS_2-Graphite-Gold (MLF-5)	Evaluated at 540°C for Short Period
$PbO.SiO_2$	Has Not Been Evaluated (Good at 500°C to 650°C and Abrasive at Lower Temperatures)
CaF_2-BaF_2-Eutectic + $Al_2(PO_4)_3$ (AFSL-28)	Evaluated at 540°C (Not Too Promising)
CaF_2 - BaF_2	Very Limited Evaluation

TABLE II.4

STATUS OF FOIL BEARING MATERIALS DEVELOPMENT WORK AT THE START OF THE PROGRAM

Foil Substrate	Journal Surface	Foil Coating	Temperature Limit	Experience
Inconel X-750	Hard chrome plate	Teflon S	290-315°C (550-600°F)	Used commercially.
Inconel X-750 Haynes 25	Hard chrome plate	Polyphenylene sulfide + aromatic polyester + Teflon + polyimide	370°C (700°F)	Used in full scale bearing tests. Limitations not established.
Inconel X-750	Plasma-sprayed Ni-Cr bonded chrome carbide	Sodium silicate bonded MoS_2-graphite-gold (MLF-5)	540°C (1000°F)	Completed 1000 starts and stops at 28-35 kPa (4-5 psi) stress level and 90 high speed rubs at 35,000 rpm, 540°C (1000°F).
Inconel X-750	Plasma-sprayed Ni-Cr bonded chrome carbide	CaF_2/BaF_2 + $Al_2(PO_4)_3$ (AFSL-28)	650°C (1200°F)	Completed 1000 starts and stops at 14-21 kPa (2-4 psi) stress level and 90 high speed rubs at 35,000 rpm, 540°C (1000°F).
Inconel X-750	Steel	Teflon S	290-315°C (550-600°F)	Completed 800 starts and stops at 28 kPa (4 psi). Coating wearing through.
Inconel X-750	Steel	Sputtered MoS_2	370°C (700°F)	Similar to Teflon S performance.
Inconel X-750	Steel	Hard, filled and bonded MoS_2	315°C (600°F)	Very difficult to run-in. Would not carry more than 14 kPa (2 psi).
Inconel X-750	Steel	Softer, resin-bonded MoS_2	290°C (550°F)	Life similar to Teflon S.
Inconel X-750	Steel	Modified Teflon coating	180°C (350°F)	Excellent performance at stress levels up to 40 kPa (6 psi). Wear life double that of other coatings.

TABLE II.5

SELECTED COATING COMBINATIONS FOR STATIC SCREENING

FLAT (SHAFT) A-286 →		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
FOIL INCONEL X-750 ↓		TiC (Sputtered)	B_4C (Sputtered)	CrB_2 (Sputtered)	Ni-Cr Bonded CrB_2 (Plasma Sprayed)	Ni-Cr Bonded Cr_2O_3 (Plasma Sprayed)	Borided A-286	Tribaloy 800 Ni-Aluminide Undercoat (Plasma Sprayed)	Nichrome Bonded Metco Cr_3C_2 (Plasma Sprayed)	Linde Cr_3C_2 with Nichrome (Det. Gun)	Cr_2O_3 (Sputtered)	WC (Sputtered)	NASA PS-101 or NASA PS-106, Ni-Aluminide Undercoat (Plasma Sprayed)	Cr_3C_2 (Sputtered)	Electrolyzed A-286	Silicon Nitride (Sputtered)	Al_2O_3 (Sputtered)	Al_2O_3 (Plasma Sprayed)	CdO and Graphite (HL-800) (Fused Coating)	Ni-Co (Electroplated)	Nitrided or Tufftrided A-286	NASA PS-100	Silicon Carbide Suspended in Electroless Nickel (NYE-CARB)	Kaman DES (Chemical Adherent Coating)
A	B_4C (Sputtered)	①	③																	㉖ 540°C				
B	TiC (Sputtered)		②														㉓							
C	CrB_2 (Sputtered)			④	⑥		⑨	⑩					⑯											
D	TiB_2 (Sputtered)				⑤															㉗ 540°C				
E	Ag (Sputtered)					⑦					⑳				㉑							㉚		
F	Cr_2O_3 (Sputtered)			⑧				⑲			⑬										㉘ 540°C			
G	Al_2O_3 (Sputtered)								⑪	⑫			⑮											
H	WC (Sputtered)											⑭ 540°C												
I	NLF-5 (Fused Coating)													⑱ 540°C										
J	Silicon Nitride (Sputtered)															㉒							㉛ 540°C	
K	AFSL-28 (Fused Coating)									㉔								㉕						
L	Cr_3C_2 (Sputtered)																				㉙ 540°C			
M	CdO and Graphite (HL-800) (Fused Coating)																		㉜ 540°C					
N	Kaman DES (Chemical Adherent Coating)							㉝																㉞
O	Uncoated Foil												⑰											

temperature can be several hundred degrees even though the ambient can be at room temperature (see Appendix B).

It will be appreciated that depending on the vehicle operating circumstance the start-stop contacts in the bearing can occur at any temperature level, but the majority of contacts would occur at elevated temperatures. Since the drive gas turbine has sufficient power at start up, the main criteria should be to choose coatings which will have long wear life even though they may not have the best lubricating properties in terms of friction at low temperature.

An optimum coating would of course have good lubrication properties over the whole of the working temperature range.

In soft-hard combinations, the hard coating was put on the journal to minimize the possibility of any damage since the cost of repairing and balancing a journal may be pretty high. It is also noted that low shear strength solid lubricant films are usually more effective on a hard substrate. An additional consideration when the foil and the journal are coated with two different materials, e.g. putting coating A on the foil and coating B on the journal, is that reversing these coatings may have a significant effect on the results. The underlying cause of this difference is thermal in nature. When the journal is rubbing in contact with the foil, the contact area on the foil is fixed and all of the frictional heat is concentrated in one area, while the contact on the journal is continuously changing with shaft rotation and the temperature rise is much more gradual.

Thus, material transfer and frictional behavior are markedly different with certain combinations of materials, depending on their relative positions on the foil or on the journal. This effect would be particularly important in the case where a soft metal film, such as silver or gold, was being used to protect one of the surfaces.

The possibility of coating the Inconel foil by a flame spraying process was explored. Surface roughening to 60-120 rms by grit blasting is required for plasma sprayed coatings. Grit blasting wrinkled the foil badly and so it was not possible to plasma spray. The detonation gun process doesn't require surface pretreatment, so this process was tried to put a coating of Cr_3C_2 on the foil. Parameters in the process were varied and substrate was continuously cooled to keep the distortion of the specimen at a minimum due to heat and mechanical forces. In spite of all the precautions, the foil wrinkled badly during coating. It was felt at that time that Inconel foil used in the program was not thick enough to withstand heat and mechanical forces during flame spraying processes.

All combinations selected in Table II.5 were subjected to static tests. Some reasons for the selection of each combination are given below. The first of the two coatings given below is on the shaft and the second is on the foil.

Comments on Initial Combination Selection. (Reference Table II.5)

1. TiC (sputtered) - B_4C (sputtered)

Both of the coating materials will form oxides at high interface temperatures. The oxides will interact to form a lubricating film and should have better characteristics. At low temperatures, this combination may have higher coefficient of friction. TiC and B_4C have cubic structures.

2. B_4C (sputtered) - TiC (sputtered)

Sometimes a coating may adhere better to one substrate than another. TiC coating may adhere better on an Inconel foil than on an A-286 shaft and vice versa. Therefore the previous combination was switched.

3. B_4C (sputtered) - B_4C (sputtered)

This provides an extremely hard material coating combination.

4. CrB_2 (sputtered) - CrB_2 (sputtered)

CrB_2 has a hexagonal crystal structure. It is known that materials having hexagonal crystal structure sometimes have superior low frictional and wear properties. Some friction and wear tests at room temperature on hot pressed CrB_2 against itself were made by Murray (Reference 13). It was found that this combination had low friction and essentially no wear. After running the tests for an extended period of time the surfaces were polished. The coating should be good for temperatures up to 980°C (1800°F).

5. Ni-Cr bonded CrB_2 (plasma sprayed) - TiB_2 (sputtered)

A plasma sprayed coating of CrB_2 tends to spall at 510°C (950°F). For the coating to adhere well at 650°C (1200°F), Ni-Cr was added as a binder. TiB_2 has a hexagonal structure.

6. Ni-Cr bonded CrB_2 (plasma sprayed) - CrB_2 (sputtered)

In combination (4), CrB_2 was applied on the shaft using the sputtering technique. Here CrB_2 is applied using the plasma spraying technique. Usually the coating technique has some effect on the adherence and mechanical properties of the coatings.

7. Ni-Cr bonded Cr_2O_3 (plasma sprayed) - Silver (sputtered)

Cr_2O_3 has been used by MTI previously on gas bearing shafts. Cr_2O_3 spalled at about 510°C (950°F). Ni-Cr was added as a binder; the amount was varied to prevent spalling at 650°C (1200°F). Cr_2O_3 has a hexagonal crystalline structure. Silver is soft and a low shear strength material at high temperatures. It provides a strong coating at low temperatures. It should provide a good coating provided the substrate does not oxidize. Silver has cubic structure.

8. CrB_2 (sputtered) - Cr_2O_3 (sputtered)

CrB_2 and Cr_2O_3 have hexagonal crystalline structure. This new combination was selected.

9. Borided A-286 - CrB_2 (sputtered)

Boriding of a shaft introduces CrB_2. Hardness of the surface from this process is about Rc 70. This process is cheaper than coating by sputtering or plasma spraying technique. This combination was selected for comparison with the results of combination 4.

10. Tribaloy 800 (plasma sprayed) - CrB_2 (sputtered)

Tribaloy 800 (made by Dupont; Co 52%, Mo 28%, Cr 17% and Si 3%) can withstand 870-980°C (1600-1800°F) due to its high chromium content. This may be a good combination to try. Tribaloy 800's laves phase is hexagonal.

11. Metco Cr_3C_2 (with a binder) - Al_2O_3 (sputtered)

Chrome carbide coatings have been used by MTI in past against MLF-5. A new combination of hard surfaces was tried here. Cr_3O_2 coating contains hexagonal structure.

12. Linde Cr_3C_2 (with a binder) - Al_2O_3 (sputtered)

This combination was selected to compare the Cr_3O_2 coatings made by Metco and Linde. Al_2O_3 has a hexagonal structure.

13. Cr_2O_3 (sputtered) - Cr_2O_3 (sputtered)

Cr_2O_3 has a hexagonal crystalline structure. Ni-Cr binder used in plasma spraying may not be necessary. Thin films made by sputtering may not spall.

14. WC (sputtered) - WC (sputtered)

WC is a very hard material and is known to be good in sliding. There may be an oxidation problem over 540°C (1000°F). WC has hexagonal crystalline structure.

15. NASA PS 101 (plasma sprayed) - Al_2O_3 (sputtered)

NASA PS 101 contains silver, nichrome, calcium fluoride, and an oxidation protective glass. It has been shown that it is good up to 1040°C (1900°F). Alumina is good at high temperatures. PS 101 was tried against Alumina.

NASA PS 106 is the same as PS 101 without the glass. Glass is added for oxidation protection at the temperatures above 650°C. For our application, we may not need glass. Moreover the absence of glass may improve the wear properties and there may be better matching of the thermal expansion between coating and substrate.

16. NASA PS 101 (plasma sprayed) - CrB_2 (sputtered)

CrB_2 is hexagonal in structure. A new combination will be tried.

17. NASA PS 101 (plasma sprayed) - Uncoated Foil

NASA PS 101 has been successfully used against the nickel base alloy, Rene 41, in an oscillating bearing application (high loads and low speeds).

18. Cr_3C_2 (sputtered) - MLF-5 (fused coating)

MLF-5 is sodium silicate bonded MoS_2 - graphite - gold. This combination has been used by MTI and is found satisfactory at 540°C (1000°F) for short test periods. It was felt that MoS_2 will oxidize to MoO_3 after long term exposure at high temperature. However it was selected as a base line. In the previous combinations, Cr_3C_2 was plasma sprayed. Here Cr_3C_2 was intended to be sputtered. This may give better adherence.

19. Tribaloy 800 (plasma sprayed) - Cr_2O_3 (sputtered)

Tribaloy 800 and Cr_2O_3 are good at high temperatures. This new combination was considered.

20. Cr_2O_3 (sputtered) - Silver (sputtered)

It is believed that low shear strength silver coating may be beneficial at high temperatures. This new combination was considered.

21. Electrolyzed A-286 - Silver (sputtered)

This new combination was considered.

22. Silicon Nitride (sputtered) - Silicon Nitride (sputtered)

Silicon Nitride has hexagonal crystaline structure and good sliding characteristics.

23. Al_2O_3 (sputtered)- TiC (sputtered)

This new combination was considered.

24. Linde Cr_3C_2 with Nichrome (detonation gun process) AFSL-28 (fused coating)

AFSL-28 consists of BaF_2/CaF_2 eutectic and $Al_2(PO_4)_3$. MTI has used this coating in past at 650°C (1200°F). This combination was further investigated.

25. Al_2O_3 (plasma sprayed) - AFSL-28 (fused coating)

Al_2O_3 is an extremely hard material and has been used in rigid bearing application at high temperatures. This new combination was considered.

26. Ni-Co (electroplated) - B_4C (sputtered)

Ni-Co (electroplated) was used by Koepsel [12].

27. Ni-Co (electroplated) - TiB_2 (sputtered)

Titanium diboride has hexagonal crystalline structure and was considered a good sliding candidate.

28. Nitrided A-286 - Cr_2O_3 (sputtered)

Cr_2O_3 has hexagonal crystalline structure. The oxidation of nitride may take place over 540°C (1000°F).

29. Nitrided A-286 - Cr_3C_2 (sputtered)

Cr_3C_2 was tried against nitrided A-286.

30. NASA PS-100 (plasma sprayed) - Ag (sputtered)

NASA PS-100 contains nichrome, dispersed glass and CaF_2 and has no silver as in PS 101. During sliding at high interface temperatures, PS-100 may interact with silver and produce properties the same as or better than those of PS 101.

31. Silicon Carbide suspended in electroless nickel (NYE-CARB) - Silicon Nitride (sputtered)

Coating of Silicon Carbide suspended in electroless nickel is applied by Electro-Coating, Inc. Benton Harbor, Michigan. This coating was tried in bearing applications. The shaft coating is limited to 540°C (1000°F).

32. CdO and graphite with a binder (fused coating) - CdO and graphite a binder (fused coating)

Peterson and Johnson (Reference 8) found that CdO and graphite mixture has low coefficient of friction in the entire temperature range up to 540°C (1000°F). The mixture cannot be plasma sprayed as CdO and graphite might reduce at high temperatures. CdO and graphite was tried fused to the surface with a binder. CdO has cubic structure.

33. Tribaloy 800 (plasma sprayed) - Kaman DES (chemically adherent coating)

Kaman DES is a proprietary coating put on by Kaman Sciences Corp., Colorado Springs. This coating has shown promise in some tests (Reference 12).

34. Kaman DES - Kaman DES

A combination of thin Cr_2O_3 coating vs. itself was tried.

Late Entries of Promising Candidate Materials

In the program, provisions were made to introduce new promising coating materials as the work proceeded. Constant effort was made to look into all high temperature and wear applications to find coatings which should be considered. Some promising coatings were identified through the static oven testing.

Calcium zirconate (calcium oxide - 31% and zirconium oxide-balance) and magnesium zirconate (magnesium oxide - 24% and zirconium oxide-balance) are used in aircraft engine combustors. These coatings are put on by plasma spraying and have performed well in relative sliding at high temperatures. These coatings look promising for this program.

Norton Abrasive has its Rokide series of coatings for high temperature application. These coatings are put on by thermo spraying. The coatings so obtained are more porous than those obtained by plasma spraying or detonation gun. The porosity of the coatings by thermo spraying is typically 2 percent. Some porosity provides room for expansion and contraction of the coating during thermal cycling. As a result, these coatings should have better thermal shock properties. However, in gas bearing applications, porosity or higher surface roughness (desired roughness typically 6 rms) is detrimental since the air film thickness in the bearing at high speeds may be as low as 2.5 μm (100 μ in). Any porosity would reduce the effective film thickness (gap between peaks on the shaft and the surface of the foil) and the peaks may start rubbing. Therefore, it is conjectured that the porosity is desirable from thermal shock point of view but may increase the bearing surface wear.

However, a compromise may always be reached. It is hoped that Rokide coating may provide such a compromise. Rokide C (Cr_2O_3) coating is most wear resistant and was selected for oven tests.

Union Carbide has recently developed a new cobalt base alloy coating designated as L 103. The coating consists of 51 percent Co, 20 percent Cr, 19.5 percent W, 5 percent Ni, 4 percent Cr_2O_3. According to metallurgists at Union Carbide, this coating has excellent wear resistance up to 980°C (1800°F) and is believed to be better than other coatings such as LC 1 (Cr_3C_2). The coating is put on by the detonation gun process. The past experience has shown that cobalt base alloys have better anti-galling properties. Therefore, L 103 coating may be superior to other Union Carbide coatings in our application.

An overcoating of an oxidation resistant, softer material may provide some lubrication, at least in the run-in period. The overcoatings of sputtered silver and gold were evaluated on corrosion-resistant undercoatings; TiC (sputtered) and Cr_2O_3 (sputtered). It was hoped that the solid lubricant, silver and gold, would provide backup in case previously selected soft lubricants were not completely effective.

MATERIALS PROCUREMENT

Most of the coatings were applied by outside vendors. Only CdO and Graphite coating (HL 800)* was developed and put on at MTI (for details see Appendix C). The actual vendors and their specifications for the coatings put on shaft and foil coupons are listed in Tables II.6 and II.7.

Foil specimens had to be pretreated before Kaman DES and HL-800 coatings could be put on with adequate bond strength. The surfaces were electro-etched to remove the oxide layer formed during heat treatment and to roughen the surfaces somewhat (for details see Bhushan [14]).

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* MTI CdO and Graphite with a binder coating has been given an identification number HL-800, where HL indicates hydresil™ lubricant and 800 indicates maximum use temperature.

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TABLE II.6

VENDORS AND SPECIFICATIONS OF COATINGS ON TEST COUPONS

S. No.	Coating	Vendor	Base	Specifications
1.	B_4C (sputtered)	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
2.	TiC (sputtered)	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
3.	Si_3N_4 (sputtered)	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
4.	Cr_3C_2 (sputtered)	Millis Research Corp., Millis, Mass	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
5.	Ag (sputtered)	Millis Research Corp., Millis, Mass.	Foil	5000A thick; RF sputtered and water cooled substrate.
6.	Cr_2O_3 (sputtered)	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
7.	Al_2O_3	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
8.	WC (sputtered)	Millis Research Corp., Millis, Mass.	Journal and Foil	5000A thick; RF sputtered and water cooled substrate.
9.	CrB_2 (sputtered)	Hohman Plating and Manuf. Dayton, Ohio	Journal and Foil	5000A thick; RF type 300 watts; Argon pressure, 7.6 microns; substrate temp. 220°C; Target-Substrate distance=45 mm
10.	TiB_2 (sputtered)	Hohman Plating and Manuf. Dayton, Ohio	Foil	6630A thick; RF type and heated substrate; Ti=68.6%, B=31.05%, O=0.17%, N=0.1%, C=.04%, H=.005%
11.	NASA PS 100 (Plasma Sprayed)	Hohman Plating and Manuf. Dayton, Ohio	Journal	0.15-0.2 mm (6-8 mils) thick; 45% Ni-Cr, 35% CaF_2 and 20% glass.
12.	Ni-Cr Bonded CrB_2 (Plasma Sprayed)	Coating Systems and Tech. No. Babylon, N.Y.	Journal	0.15-0.2mm thick; 25% Ni-Cr (80% Ni-20%Cr) and 75% CrB_2 (70.10% Cr, 29.37%, C=.04%, O=0.25%, N=.04%,-325 mesh).
13.	Ni-Cr Bonded Cr_2O_3	Coating Systems and Tech. No. Babylon, N.Y.	Journal	0.15-0.2mm thick; 25%Ni-Cr and 75% Cr_2O_3 (98% pure and -45+15 microns).
14.	Ni-Cr Bonded Cr_3C_2	Coating Systems and Tech. No. Babylon, N.Y.	Journal	0.15-0.2mm thick; 25% Ni-Cr (-53+10 microns) and 75% Cr_3C_2 (99% Pure and -45+5 microns)
15.	Al_2O_3 (Plasma Sprayed)	Coating Systems and Tech. No. Babylon, N.Y.	Journal	0.15-0.2mm thick; 98% Pure Al_2O_3 (-25+5 microns)

TABLE II.6 (CONT'D)

VENDORS AND SPECIFICATIONS OF COATINGS ON TEST COUPONS

S. No.	Coating	Vendor	Base	Specifications
16.	NASA PS 101 (Plasma Sprayed) Ni-Aluminide undercoat	NASA, Cleveland	Journal	50-75μm (2-3mils) undercoat, Ni-Aluminide; 150-200μm topcoat, 30% Ag, 30% Ni-Cr, 25% CaF_2 and 15% Sodium free glass
17.	NASA PS 106 (Plasma sprayed) Ni-Aluminide undercoat	NASA, Cleveland	Journal	50-75μm thick undercoat; Ni-Aluminide 150-200μm topcoat; 35% Ni-Cr, 30% CaF_2 and 35% Ag.
18.	Tribaloy 800 Ni-Aluminide Undercoat (Plasma Sprayed)	Quantom Inc. Wallingford, Conn.	Journal	50-75μm thick undercoat, 450 NS, (4-5.5% Al, 2.5% Max. Impurities and Remainder Ni); 150-200 μm top coat T-800 (52%Co, 28%Mo, 17%Cr and 3%Si)
19.	Ni-Cr Bonded Cr_3C_2 (Detonation Gun)	Linde Div., Union Carbide Corp, North Haven	Journal	150-200 μm thick; 20% Ni-Cr (80% Ni-20% Cr) and 80% Cr_3C_2.
20.	MLF-5	Midwest Research Inst. Kansas City, Mo.	Foil	12-18 μm (0.5-0.7 mils) thick; MoS_2, graphite, gold, sodium sillicate and water
21.	AFSL-28 (Fused Coating)	Midwest Research Inst. Kansas City, Mo.	Foil	12-18 μm thick; calcium fluoride, barium fluoride and aluminum phosphate.
22.	Ni-Co (Electroplated)	Metal Surfaces Inc. Bell Gardens, Cal.	Journal	25 μm (1 mil) thick; Proprietary Process (60 to 80% Co).
23.	Nitrided A286 and Tufftrided A286	Lindberg Heat Treating Rochester, N.Y.	Journal	Nitriding-immersion at 565°C in molten salt for 36 hours and furnace cool Tufftriding-immersion at 570°C for 1.5 hours and WQ.
24.	Borided A286	Lindberg Heat Treating Boston, Mass.	Journal	Surface Core converted to CrB_2 compound.
25.	Kaman DES (chemical adherent coating)	Kaman Sciences Corp. Colorado Springs	Journal and Foil	Foil- (1.3-2.5μm) (50 to 100 μ in.) Journal- 7.5-12.5 μm (300 to 500 μ in.) Proprietary process (primarily Cr_2O_3).
26.	Silicone Carbide Suspended in Electroless Nickel	Electrocoating Inc. Benton Harbor, Michigan	Journal	50 μm (2 mils) thick; NYE-CARB, 20-30% Silicone Carbide particles by volume 10-15 microns in a matrix of alloy, 90-93% Ni, 7-10% Phosphorous
27.	Electrolyzed A286	Electrolyzing Corp.	Journal	3-13 μm thick; Proprietary Process, chromium alloy coating.
28.	HL-800	MTI	Journal and Foil	25 μm (1 mil) thick; CdO, graphite, sodium sillicate, water and wetting agent.

TABLE II.7

VENDORS AND SPECIFICATIONS OF COATINGS SELECTED LATER IN THE PROGRAM*

S.No.	Coating	Vendor	Base	Specifications
1	Magnesium Zirconate (Plasma Sprayed) Ni-Aluminide Undercoat	Coating Systems & Tech.	Journal	150 - 200 µm thick 24% MgO and Zirconium Oxide Balance
2	Calcium Zirconate (Plasma Sprayed) Ni-Aluminide Undercoat	Coating Systems & Tech.	Journal	150 - 200 µm thick 31% CaO and Zirconium Oxide balance
3	Rokide C (Thermo Sprayed) Ni-Aluminide Undercoat	Hard Face Welding and Machining, Buffalo	Journal	50 - 75 µm Ni-Aluminide, 150 - 200 µm Rokide C (Cr_2O_3)
4	L103 (Detonation Gun)	Unide Carbide Corp. Indianapolis, Ind.	Journal	150 - 200 µm 51% Co, 20% Cr, 19.5% W, 5% Ni, 4% Cr_2O_3
5	TiC (sputtered) and Ag (sputtered)	Millis Research Corp. Millis, Ma	Foil	5000 A thick TiC 5000 A thick Ag RF sputtered, substrate water cooled
6	TiC (sputtered) and Au (sputtered)	Millis Research Corp. Millis, Ma	Foil	5000 A thick TiC 5000 A thick Au RF sputtered, substrate water cooled
7	Cr_2O_3 (sputtered) and Ag (sputtered)	Millis Research Corp. Millis, Ma	Foil	5000 A thick Cr_2O_3 5000 A thick Ag RF sputtered and substrate water cooled
8	Cr_2O_3(sputtered) and Au (sputtered)	Millis Research Corp. Millis, Ma	Foil	5000 A thick Cr_2O_3 5000 A thick Au RF sputtered and substrate water cooled

*These coatings were only screened through static oven tests.

III. STATIC MATERIAL SCREENING TESTS

SAMPLE PREPARATIONS

The selected specimens were in the form of small foil samples approximately 50 mm x 50 mm (2" x 2") square and 100 μm (0.004 in.) thick and small journal samples approximately 50 mm x 50 mm (2" x 2") square and 750 μm (0.030 in.) thick each with the appropriate coatings as described in the previous chapter. Thin sheet coupons of journal were selected so that weight change could be detected more accurately. Two sets of each coating were prepared. One set was to go through the oven test and the other set was retained for comparisons. The photographs of microstructures of A286 and Inco X-750 substrates before and after oven tests were taken for reference (Figure III.1). All pertinent manufacturing data, quality control and pre-test examination results were recorded.

OVEN SCREENING TESTS

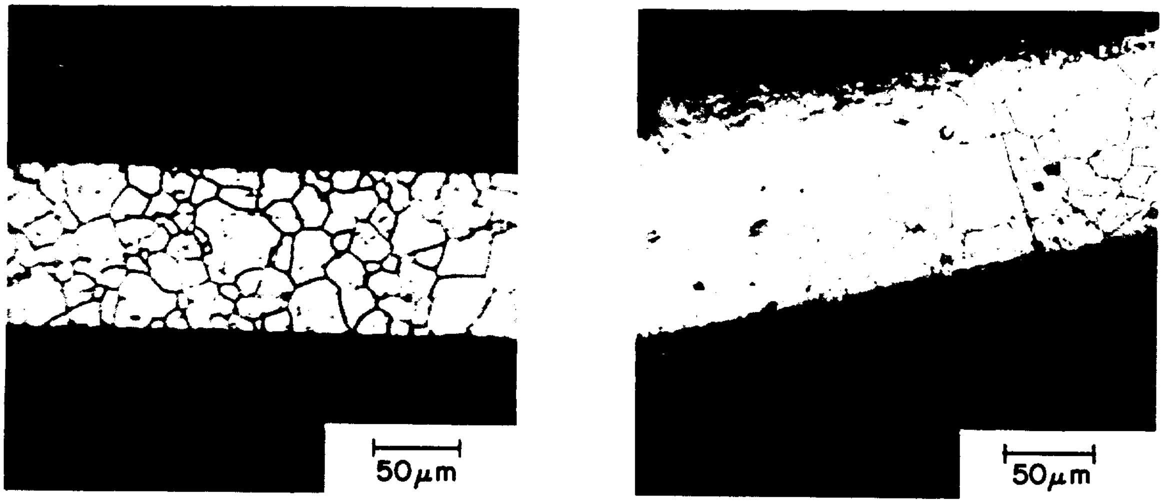
Static screening tests were conducted on material coating combinations suitable for operation over two temperature ranges:

1. Room temperature to 540°C (1000°F)
2. Room temperature to 650°C (1200°F)

The test consisted of exposure of material samples in an oven (oxidizing environment) for 300 hours at the maximum service temperature (either at 540°C or 650°C) and 10 temperature cycles from room temperature to the maximum service temperature. The specimens were cooled from maximum service temperature to close to room temperature in about 20 minutes.

SPECIMEN ANALYSIS

The specimens were subjected to standard physical and metallurgical surface examinations prior to and after test for further screening. The foil coupons were tested under flex bending. All of the shaft and foil coupons went through a surface adhesion tape test, and a scratch test under microscope examination. Selected specimens were examined using scanning electron microscope and electron diffraction techniques. Superficial Rockwell hardness tests were performed on the thicker coatings. The weight and the



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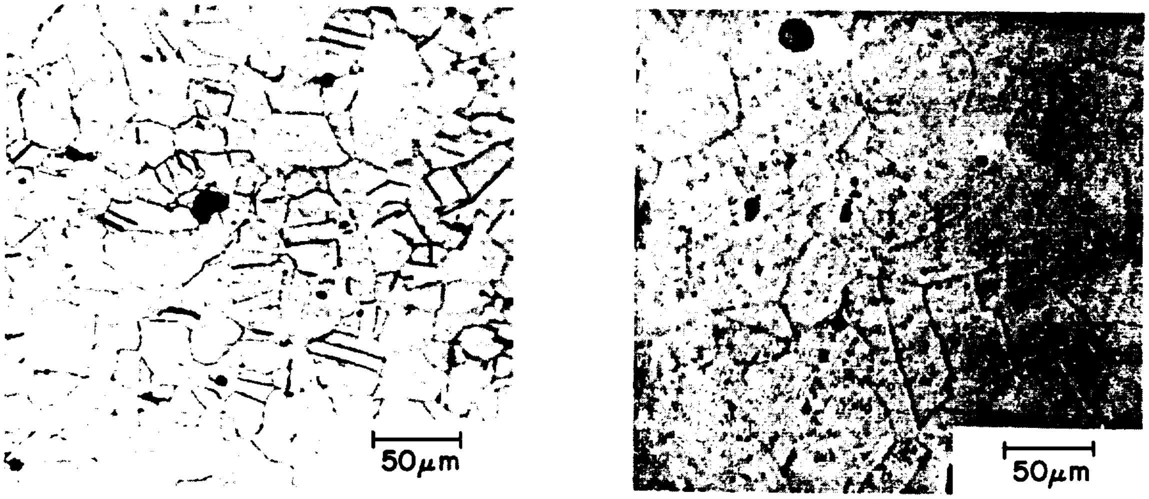
(ACID ETCHED)

INCONEL X-750



AS RECEIVED
(ACID ETCHED)

HEAT TREATED
(ACID ETCHED)

A-286

Fig. III.1 Microstructures of Substrate Cross Sections Perpendicular to Rolling Direction thickness of the coupons, before and after the oven tests, were recorded (See Appendix D for results). The coupons before and after oven tests were mounted side by side for visual comparisons. Photographs are shown in Figures III.2 to III.7.

Flex Bending Test

The foils in the bearing go through flexing during operation. It was suspected that some of the hard ceramic coatings might crack during flexing. The coated coupons were flexed (140 μm max. at 30 cps) for two hours and then examined under the microscope. None of the coatings showed any effect at all from the flexing.

Tape Test

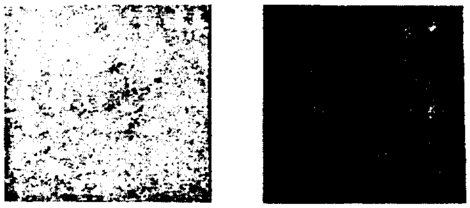
A surface adhesion test was carried out on the coatings to get a qualitative measure of the adhesion between the coating and the substrate. Scotch^R brand magic transparent tape No. 810 was pressed on the coating surface by finger load and was pulled by hand. In some cases coatings which had gone through the oven test showed loss of adhesion in the tape test.

Scratch Test

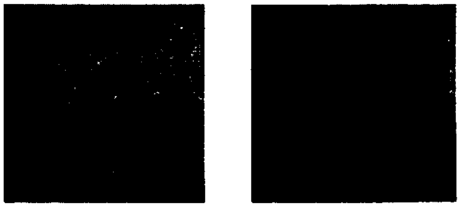
A scriber was used to make a scratch on the coatings before and after oven test, while looking through an optical microscope. The reason for this test was twofold. The test would give a semi-quantitative comparison between the hardness of the coating before and after the oven test, and any significant softening of the coating in extended thermal exposure could be detected. Furthermore, the examination of the scratch would show if the coating was ductile or brittle. If small branch cracks develop on the sides of the scratch, it is believed that the coating may be brittle and vice versa.

Optical Microscope Examination

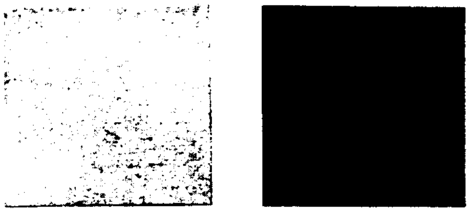
A general microscope examination was carried out to study any change in surface texture. Microscope examination was carried out to detect, in sputtered coatings, if any coating was left after the oven test.



BEFORE OVEN AFTER OVEN
TiC (Sputtered)



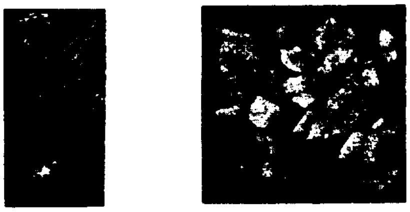
BEFORE OVEN AFTER OVEN
B_4C (Sputtered)



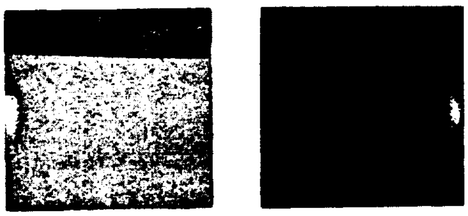
BEFORE OVEN AFTER OVEN
CrB_2 (Sputtered)

BEFORE OVEN AFTER OVEN
Ni-Cr Bonded CrB_2
(Plasma Sprayed)

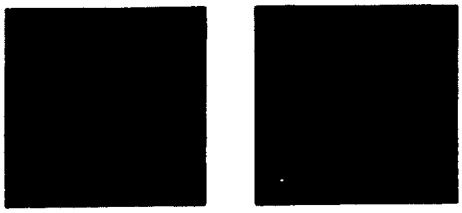
BEFORE OVEN AFTER OVEN
Ni-Cr Bonded Cr_2O_3
(Plasma Sprayed)



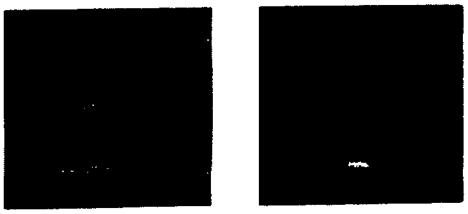
BEFORE OVEN AFTER OVEN
Borided A-286



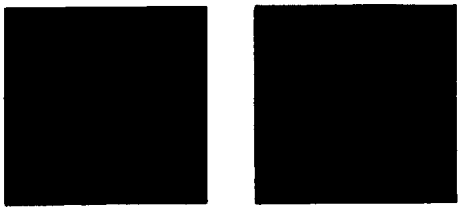
BEFORE OVEN AFTER OVEN
Tribaloy 800 (Plasma Sprayed)



BEFORE OVEN AFTER OVEN
Metco Cr_3C_2 (Plasma Sprayed)



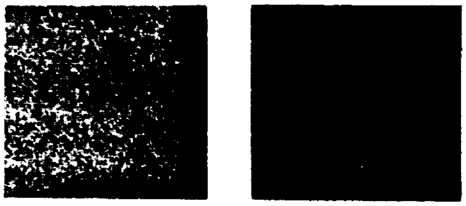
BEFORE OVEN AFTER OVEN
Linde Cr_3C_2 (Detonation Gun)



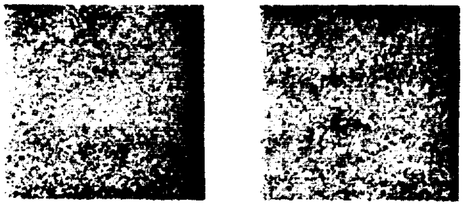
BEFORE OVEN AFTER OVEN
Cr_2O_3 (Sputtered)

A-286 COUPONS (650°C)

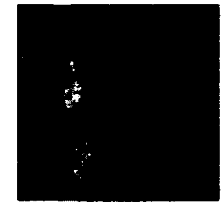
Fig. III.2 Photograph of Coatings Before and After Oven Test



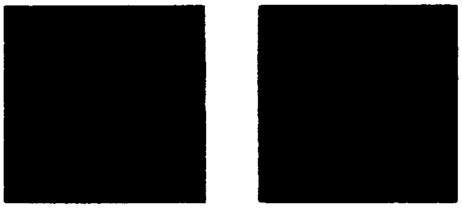
BEFORE OVEN AFTER OVEN
NASA PS-101 (Plasma Sprayed)



BEFORE OVEN AFTER OVEN
NASA PS-106 (Plasma Sprayed)

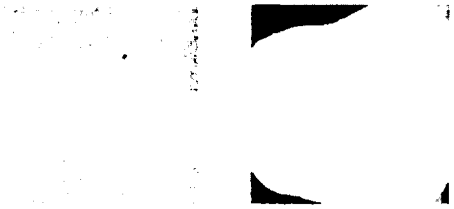


BEFORE OVEN AFTER OVEN
Electrolyzed A-286

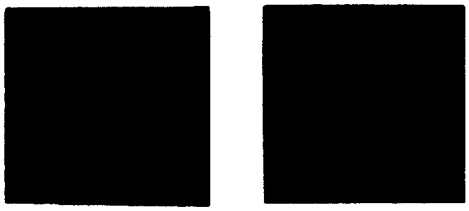


BEFORE OVEN AFTER OVEN
Silicon Nitride (Sputtered)

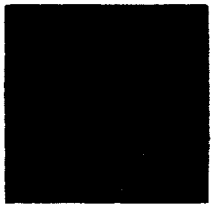
BEFORE OVEN AFTER OVEN
Al_2O_3 (Sputtered)



BEFORE OVEN AFTER OVEN
Al_2O_3 (Plasma Sprayed)

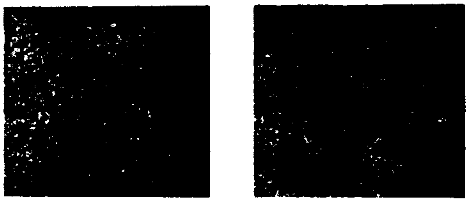


BEFORE OVEN AFTER OVEN
CdO and Graphite (Fused Coating)

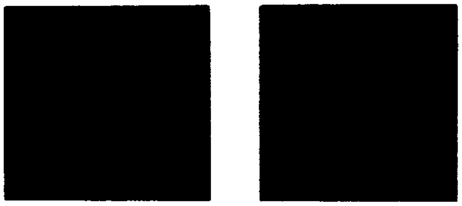


AFTER OVEN
Nitrided A-286

AFTER OVEN
Tufftrided A-286



BEFORE OVEN AFTER OVEN
NASA PS-100



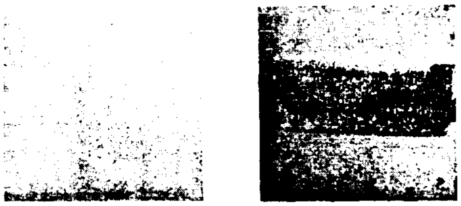
BEFORE OVEN AFTER OVEN
Kamen Dex (Chemical Adherent Coating)

A-286 COUPONS (650°C)

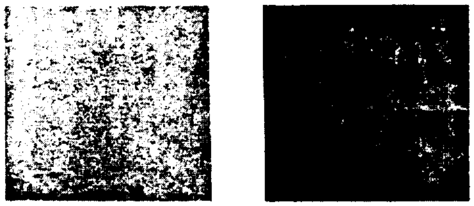
Fig. III.3 Photograph of Coatings Before and After Oven Test

ORIGINAL PAGE IS
OF POOR QUALITY

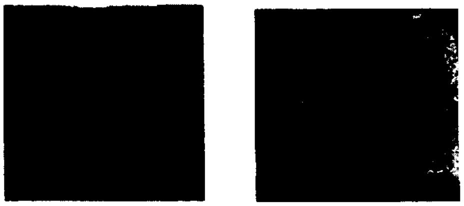
BEFORE OVEN AFTER OVEN
Borided A-286



BEFORE OVEN AFTER OVEN
WC (Sputtered)

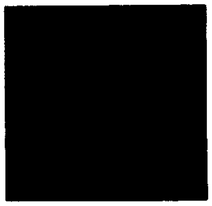


BEFORE OVEN AFTER OVEN
Cr_3C_2 (Sputtered)

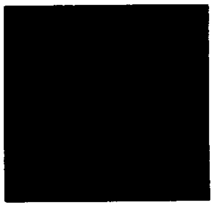


BEFORE OVEN AFTER OVEN
CdO and Graphite (Fused Coating)

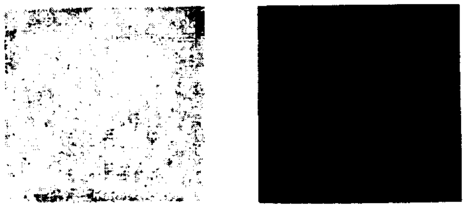
BEFORE OVEN AFTER OVEN
Ni-Co Electroplated



AFTER OVEN
Nitrided A-286

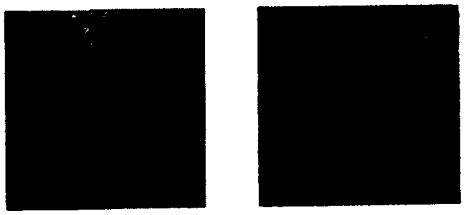


AFTER OVEN
Tufftrided A-286



BEFORE OVEN AFTER OVEN
Silicone Carbide in Electroless Nickel

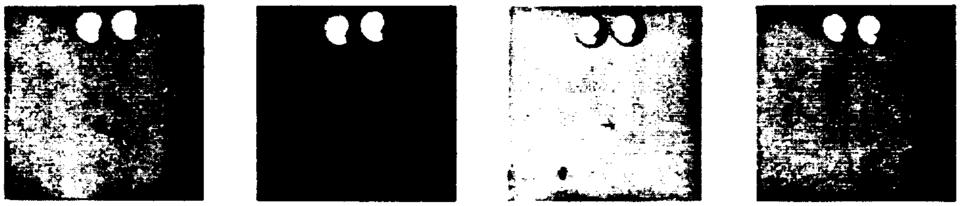
A-286 COUPONS (540°C)



BEFORE OVEN AFTER OVEN
Uncoated Coupon

A-286 COUPONS (650°C)

Fig. III.4 Photograph of Coatings Before and After Oven Test



BEFORE OVEN AFTER OVEN
B_4C (Sputtered)

BEFORE OVEN AFTER OVEN
TiC (Sputtered)

BEFORE OVEN AFTER OVEN
CrB_2 (Sputtered)

BEFORE OVEN AFTER OVEN
TiB_2 (Sputtered)

BEFORE OVEN AFTER OVEN
Ag (Sputtered)

BEFORE OVEN AFTER OVEN
Cr_2O_3 (Sputtered)

BEFORE OVEN AFTER OVEN
Al_2O_3 (Sputtered)

BEFORE OVEN AFTER OVEN
Silicon Nitride (Sputtered)

BEFORE OVEN AFTER OVEN
AFSL-28 (Fused Coating)

BEFORE OVEN AFTER OVEN
CdO and Graphite (Fused Coating)

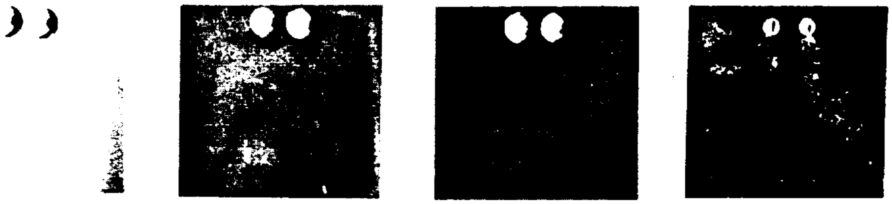
INCONEL X-750 COUPONS (650°C)

Fig. III.5 Photograph of Coatings Before and After Oven Test

BEFORE OVEN AFTER OVEN
Kamen Des (Chemical Adherent Coating)

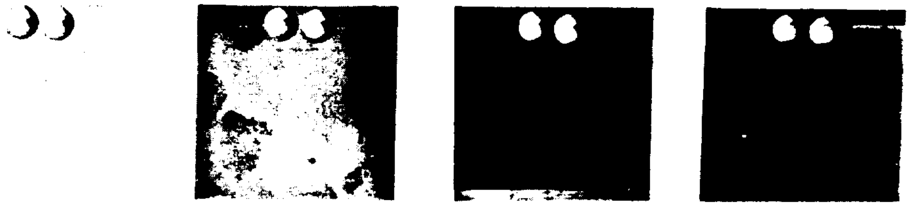
BEFORE OVEN AFTER OVEN
Uncoated Foil

INCONEL X-750 COUPONS (650°C)



BEFORE OVEN AFTER OVEN
WC (Sputtered)

BEFORE OVEN AFTER OVEN
MLF-5 (Fused Coating)



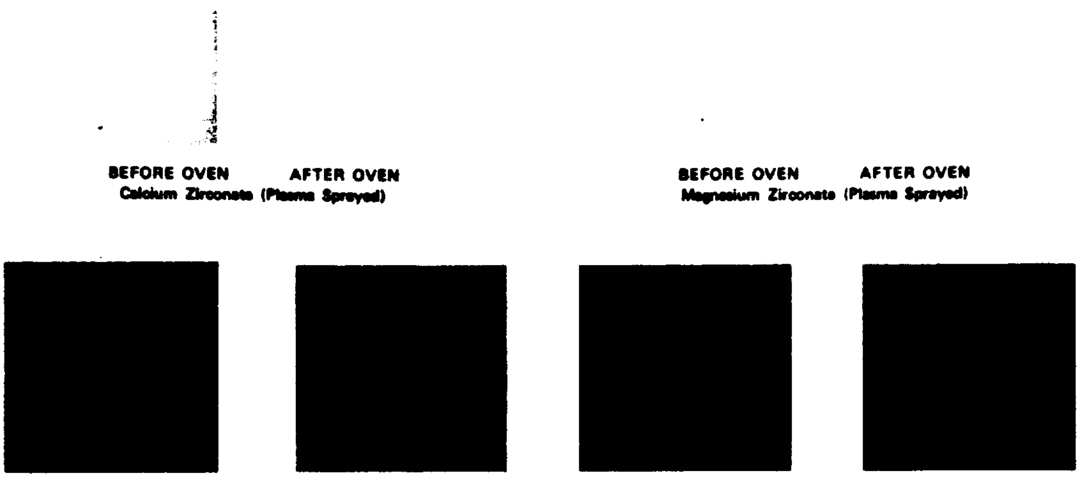
BEFORE OVEN AFTER OVEN
Cr_3C_2 (Sputtered)

BEFORE OVEN AFTER OVEN
CdO and Graphite (Fused Coating)

BEFORE OVEN AFTER OVEN
CdO and Graphite (Vapor Honed)

INCONEL X-750 COUPONS (540°C)

Fig. III.6 Photograph of Coatings Before and After Oven Test



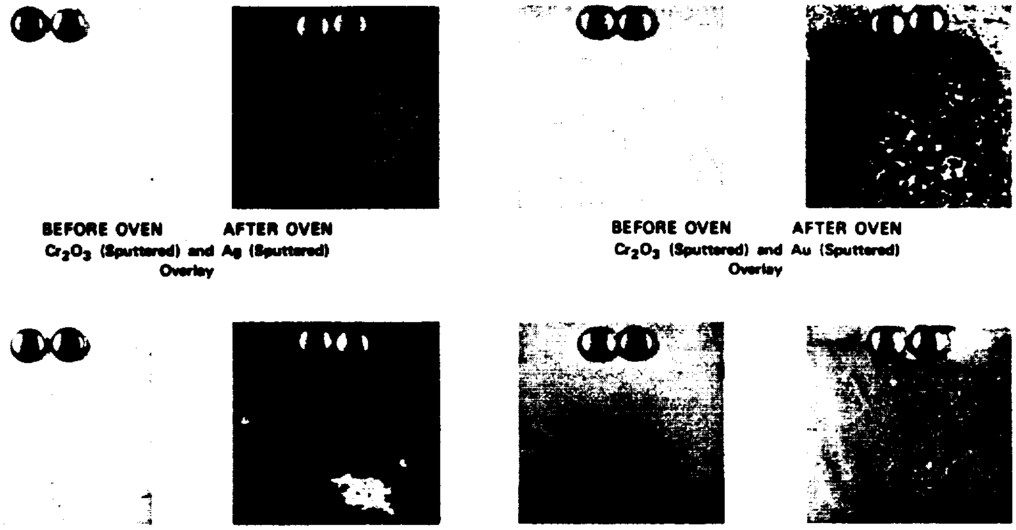
BEFORE OVEN AFTER OVEN
Calcium Zirconate (Plasma Sprayed)

BEFORE OVEN AFTER OVEN
Magnesium Zirconate (Plasma Sprayed)

BEFORE OVEN AFTER OVEN
Rokide C (Thermo Spray)

BEFORE OVEN AFTER OVEN
L103 (Detonation Gun)

A-286 COUPONS (650°C)



BEFORE OVEN AFTER OVEN
Cr_2O_3 (Sputtered) and Ag (Sputtered)
Overlay

BEFORE OVEN AFTER OVEN
Cr_2O_3 (Sputtered) and Au (Sputtered)
Overlay

BEFORE OVEN AFTER OVEN
TiC (Sputtered) and Ag (Sputtered)
Overlay

BEFORE OVEN AFTER OVEN
TiC (Sputtered) and Au (Sputtered)
Overlay

INCONEL X-750 COUPONS (540°C)

Fig. III.7 Photograph of Coatings Before and After Oven Test

Superficial Hardness Test

Plasma sprayed coatings were 150-250 μm (6-10 mils) thick. Superficial Rockwell hardness tests with a diamond indenter and the lightest load (15 N) were made on these coatings. As expected, no change in the hardness could be detected in the coupons after they had gone through oven test. The diffused coupons, nitrided, tufftrided, and borided were tested with a file. The hardness of a file is typically Rc 60 and the hardness of the diffused zone on the substrate (less than 500 μm deep) is about Rc 70. If the file slipped, as if gliding on glass, it implied that the substrate was harder than Rc 60, for example that a diffused zone was still there. If the file made a scratch on the substrate, it implied that the substrate was softer than the file, i.e. the diffused zone either had softened or had become brittle during thermal exposure and cycling, and been lost. This test was found to be quite meaningful.

Weight Gain

Records were made of the weight and the thickness of the coupons before and after the oven test. It was found that most of the coupons gained weight and that this was not representative of a good quality coating.

Metallurgical Examination of Selected Journal and Foil Coupons after Oven Test

Surface morphology of the coatings on journal and foil coupons was examined in the Scanning Electron Microscope (SEM). The X-Ray Energy Dispersive Analyzer (X-REDA) hooked to SEM was utilized to determine the elemental composition of specific particles or structures on the samples. In the case of sputtered coatings, X-REDA analysis gave the composition of the coating and the substrate since the thickness of the coating was only 5000°A (20 μ in.). By knowing the composition of the substrate, the composition of the coating can be obtained qualitatively. The analysis is ineffective in detecting elements with atomic number less than that of Na (11). Analyses by Reflection Electron Diffraction (ED) and X-ray diffraction have been made to determine the chemical compounds in the coatings. The analyses are limited to crystalline structures.

The base materials for foil and journal were Inconel X-750 and A-286. Some of the promising candidate coatings for start-stop tests were selected for metallurgical examinations. As the composition and morphology of sputtered coatings may depend very much on sputtering parameters, an examination of the sputtered coatings was deemed necessary; they were primarily selected for metallurgical examination. The results of the study are presented in Appendix D. The highlights and general observations are reviewed in Table III.1.

DISCUSSION OF THE RESULTS

The results of the 300-hour oven tests and the thermal cycle tests were very encouraging since several promising candidate materials survived these screening evaluations with little or no deterioration (Appendix D). Table III.2 shows the coating materials which survived the screening tests. Six coatings on the shaft showed practically no effect from the thermal exposure nor from the thermal cycling. They were given first priority for the start-stop bearing tests. Another five coatings were borderline and might have been selected in a large program. They were given second priority. Five coatings on the foil and an uncoated foil were found to be promising and given the first priority. Three coatings were found to be marginal. It is felt that further work should be done in developing the coatings, including the ones which looked marginal. A parametric study of the sputtering variables in the lower priority specimens could lead to a coating with desirable properties. A technique for coating CdO and graphite on foils has been developed at MTI. Although it seems that some of the graphite oxidizes at high temperature, it was selected to see if it could be used at temperatures slightly lower than 540°C (1000°F).

The number of candidate materials appears to be rather unwieldy, particularly if it is proposed that every possible foil and journal combination be evaluated. In addition, only one solid lubricant coating, the NASA PS-106 material survived the 650°C (1200°F) furnace and thermal cycle tests. Silver, which was one of the candidate coatings that was selected to be a high temperature, low shear strength solid film showed very poor adherence on the A-286 stainless steel substrate. In retrospect, this was a poor choice for the substrate since it had been found previously that silver platings are not good oxidation barriers. Any oxidation of the substrate will cause the silver to spall. Thus, this test result should not be used to rule out silver because it only shows that silver on A-286 steel is unsuitable.

TABLE III.1

RESULTS OF METALLURGICAL STUDIES OF COATED COUPONS BEFORE AND AFTER OVEN TEST

S. No.	Coatings	Metallurgical Technique	Results and Observations
1.	TiC (sputtered) on foil	Scanning Electron Microscope (SEM), X-Ray Energy Dispersive Analyser (X-REDA) and Reflection Electron Diffraction (ED)	TiC coating does not provide adequate protection and is transformed to TiO_2 during heat treatment.
2.	Si_3N_4 (sputtered) on foil	SEM, X-REDA and ED	The surface morphology and general appearance remain unchanged during oven test.
3.	Cr_2O_3 (sputtered) on foil	SEM, X-REDA and X-Ray Diffraction	The surface morphology and general appearance remain unchanged during oven test.
4.	Kaman DES on foil	SEM, X-REDA and X-Ray Diffraction	The coating remains unchanged. Kaman DES is virtually Cr_2O_3.
5.	HL-800 on foil	SEM, X-REDA and ED	Coating shows excellent dispersion of CdO and Graphite with their binder. CdO reacts with the binder and forms $CdSiO_3$ during oven test.
6.	$A\ell_2O_3$ (sputtered) on foil	ED	There was practically no build up of $A\ell_2O_3$ on the coupon.
7.	Tribaloy 800 (Plasma Sprayed) on journal	SEM and X-REDA	The coating has intergranular cracks on the surface and they are completely removed during grinding and hence present no problem.
8.	Heat treated INCO X-750 (For comparison)	SEM, X-REDA and ED	The surface contains spinel compound ($NiO.Fe_2O_3$)

TABLE III.2

1. Coating Materials For Foils From Oven Screening Tests

First Priority

1. TiC (sputtered)
2. Cr_2O_3 (sputtered)
3. Si_3N_4 (sputtered)
4. CdO + Graphite (Fused Coating), 540°C (1000°F)
5. Kaman DES (chemically adherent coating)
6. Uncoated Foil

Second Priority

1. B_4C (sputtered)
2. CrB_2 (sputtered)
3. Al_2O_3 (sputtered)

II. Coating Materials For Shaft From Oven Screening Tests

First Priority

1. Ni-Cr Bonded CrB_2 (Plasma Sprayed)
2. Tribaloy 800 (Plasma Sprayed)
   Ni-Aluminide Undercoat
3. Linde Cr_3C_2 with Ni-Cr binder
   (Plasma Sprayed)
4. Cr_2O_3 (sputtered)
5. NASA PS 106 (Plasma Sprayed)
   with Ni-Aluminide Undercoat
6. Kaman DES (chemically adherent coating)

Second Priority

1. TiC (sputtered)
2. B_4C (sputtered)
3. CrB_2 (sputtered)
4. CdO + Graphite (Fused coating), 540°C (1000°F)
5. Al_2O_3 (sputtered)

III. Recommendations of Coating Materials For Shaft From Late Entries In The Oven Tests (Future Program).

1. L103 (Detonation Gun)
2. Calcium Zirconate (Plasma Sprayed)
   Ni-Aluminide Undercoat
3. Magnesium Zirconate (Plasma Sprayed)
   Ni-Aluminide Undercoat

RECOMMENDED COATING COMBINATIONS FOR PARTIAL ARC BEARING START-STOP TESTS

Referring to Table III.2, six coatings were given first priority for use on the journal surfaces and five coatings (plus one uncoated foil) were selected as the most promising for the foils. The materials with first priorities were selected for the program. In Table III.3, all of these materials are listed together with crystal structure, hardness data and a list of the most stable oxides which would be formed at high temperature.

With the exception of TiC, all of these materials either have a hexagonal structure or contain compounds having this structure. This satisfies one of the criteria which was listed previously.

With the exception of the NASA PS 106 (35% Nichrome, 30% CaF_2 and 35% silver) and HL-800 (3 parts graphite and 1 part CdO with a binder) coatings, all of these materials are hard and are generally classified as wear resistant. This satisfies another criterion for material selection.

Finally, three of these materials, Tribaloy 800, TiC and Si_3N_4, form definite and beneficial oxide films in the temperature range of interest for this application. The Tribaloy 800 will develop complex oxides which are known to be beneficial in preventing surface damage during sliding. Both the TiC and the Si_3N_4 will form thin protective oxide films (TiO_2 and SiO_2) which protect the substrate from further oxidations - unless they are disrupted by the sliding process. While neither TiO_2 nor SiO_2 are particularly effective by themselves in preventing wear or surface damage, both can form complex oxides by reaction with oxide films on the opposing surface and thus can produce compounds with lower melting points. For example, if a CrB_2 coating was applied to the shaft and a TiC coating was applied to the foil, preoxidation of these coatings would produce a thin layer of $Cr_2O_3 + B_2O_3$ on the shaft and a surface layer of TiO_2 on the TiC coated foil. There is some evidence to indicate that this particular combination of oxides would form a softer glass layer which might protect the substrate coatings from damage.

Table III.4 shows the material combinations recommended for the partial arc bearing start-stop tests with criteria for selection. Table III.5 shows the selected combinations in a matrix form.

TABLE III.3

CRYSTAL STRUCTURE, VICKERS HARDNESS AND OXIDATION PRODUCTS OF COATING MATERIALS SELECTED FOR START-STOP TESTS

COMPOUND	STRUCTURE	VICKERS HARDNESS(a)	MOST STABLE OXIDATION PRODUCTS
CrB_2 + 25% Ni-Cr	CrB_2 is hexagonal	CrB_2~2150 (100 gram)	Cr_2O_3 B_2O_3 NiO
Tribaloy 800 52Co-28Mo-17Cr-3Si	Laves phase is hexagonal	Laves phase ~1100. Bulk 56-60Rc	CoO MoO_3 Cr_2O_3 SiO_2
Cr_3C_2	Cr_3C_2- coating also contains hexagonal	Cr_3C_2 2650 (50 grams)	Cr_2O_3
Cr_2O_3	Hexagonal	~9 Moh scale	Fully oxidized material
Kaman DES (Cr_2O_3 + proprietary)	SEE Cr_2O_3		
TiC	Cubic	~3200 (100 gram)	TiO_2
Si_3N_4	Hexagonal	~1700-2200 (100 gram)	SiO_2
CdO + graphite	CdO-cubic Graphite-hexagonal	Soft Coating	Not Applicalbe

(a) Actual values very dependent on method of preparation, porosity and purity.

TABLE III.4

COATING COMBINATIONS WITH CRITERIA FOR SELECTION FOR START-STOP TESTS

	FOIL	JOURNAL	CRITERIA FOR SELECTION
1.	TiC (sp., preox.)	CrB_2(P.S., preox.)	Possible eutectic oxide formation
2.	TiC (sp., preox.)	Kaman DES	Possible oxide interaction
3.	Cr_2O_3 (sp.)	Cr_2O_3 (sp.)	Crystal structure, past experience
4.	Cr_2O_3 (sp.)	Kaman DES	Same as above Also to determine if Kaman DES behaves like straight Cr_2O_3
5.	Cr_2O_3 (sp.)	Tribaloy 800 (P.S.)	Crystal structure. Also beneficial effect of oxide film on Tribaloy 800
6.	Kaman DES	Kaman DES	To see how this compares with 3.
7.	Kaman DES	Cr_2O_3 (sp.)	To compare with 4.
8.	CdO-Graphite (Fused)	Linde Cr_3C_2 (D.G.)	Effectiveness of solid film lubricant coating against plasma sprayed hard coating
9.	Si_3N_4 (sp., preox.)	CrB_2(P.S., preox.)	Possible oxide interaction. Crystal structure
10.	Uncoated	NASA PS-106 (P.S.)	Effectiveness of solid film lubricant
11.	CdO-Graphite (Fused)	NASA PS-106 (P.S.)	Effectiveness of soft against soft coating
12.	Kaman DES	NASA PS-106 (P.S.)	Effectiveness of Fluoride (soft) coating against Kaman DES (hard) coating
13.	CdO-Graphite (Fused)	Cr_2O_3 (sp.)	Effectiveness of solid film lubricant coating against sputtered hard coating (to compare with 8)
14.	TiC (sp., preox.)	Tribaloy 800 (P.S.)	Possible oxide interaction
15.	TiC (sp., preox.)	Cr_2O_3 (sp.)	To compare with 2
16.	Si_3N_4 (sp., preox.)	Tribaloy 800 (P.S.)	Possible oxide interaction
17.	Kaman DES	Tribaloy 800 (P.S.)	To compare with 5
18.	Cr_2O_3 (sp.)	CrB_2 (P.S., preox.)	Possible oxide interaction (to compare with 3, 4, 6 and 7)

sp. - sputtered P.S. - Plasma Sprayed D.G. - Detonation Gun

TABLE III.5

SELECTED COATING COMBINATIONS FOR START-STOP TESTS

Journal → / Foil ↓		1	2	3	4	5	6
		CrB_2 (P.S.)	Tribaloy 800(P.S.)	Linde Cr_3C_2 (D.G.)	Cr_2O_3 (sp.)	NASA PS-106 (P.S.)	Kaman DES
A	TiC(sp.)	①	⑭		⑮		②
B	Cr_2O_3(sp.)	⑱	⑤		③		④
C	Kaman DES		⑰		⑦	⑫	⑥
D	CdO-Graphite (F.C.)			⑧ 540°C	⑬ 540°C	⑪ 540°C	
E	Si_3N_4(sp.)	⑨	⑯				
F	Uncoated Foil					⑩	

sp. - sputtered
P.S. - Plasma Sprayed
D.G. - Detonation Gun
F.C. - Fused Coating

Since Cr_2O_3 has given good performance in rigid gas bearings, more combinations have been selected with Cr_2O_3 coating.

The thickness of the finished plasma sprayed coating was selected to be 65 to 90 μm (2.5 to 3.5 mils) on one side. The plasma sprayed coatings requiring undercoats were 140 to 165 μm (5.5 to 6.5 mils) thick (50 to 75 μm thick undercoat inclusive). An additional buildup of 100 to 125 μm (4-5 mils) on one side was required for proper finishing of the coating. The thickness of the sputtered coatings was selected to be about 5000 A (20 μ in). Kaman DES coatings were about 1.3 - 2.5 μm (0.05 to 0.1 mil) thick on the foil and 8-13 μm (0.3 to 0.5 mil) thick on the journal. The HL-800 coating was sprayed about 25 μm (1 mil) thick and burnished to about 8-10 μm (0.3 - 0.4 mil) thick.

IV. TEST FACILITY AND INSTRUMENTATION

TEST RIG DESCRIPTION

An existing MTI owned test rig, which was used in Part I of this program, was further modified to meet the start-stop requirements of the program. The test rig is shown in Figures IV.1 and IV.2. Figure IV.3 shows the complete test facility.

The support shaft was made of A-286 to match the material of the test journals and supported on two preloaded angular contact ball bearings. The shaft incorporated an integral heat dam consisting of a 28.4 mm (1.12 inch) long section 1.6 mm (.063 inch) thick which extended into the hot zone. The test journal was a light interference fit onto the shaft. The pilot diameter for the interface was coated with a 5-10 μm (.0002-.0004 inch) thick layer of Nickel-Chromium applied by the Electrolizing Company. The test journal was held in place with a tie bolt, also made of A-286, which was threaded into the shaft. Heat baffles mounted to the support housing interrupted the flow of heat out of the hot zone. In addition a 6.4 mm (.250 inch) thick Mycalex 500 disk acted as an insulator between the heat baffles and the support housing.

Oil for lubricating and cooling the support ball bearings was supplied through two oil jets 180° apart at each bearing. A water cooled heat exchanger in the oil supply loop removed heat from the oil. A water jacket in the support housing assisted in removing heat from the test end support ball bearing.

A double labyrinth seal with pressurized air supplied between the seals prevented the oil from traveling down the test shaft into the test bearing area.

The test spindle was driven by a 1 hp and 3450 RPM electric motor. The motor was attached to the main vertical support plate and connected to the spindle with a flat drive belt. A pulley ratio of 4:1 was used to obtain a 13,800 RPM spindle speed. The on-off cycle rate was controlled by adjustable timers. An impulse counter was used to count each cycle.

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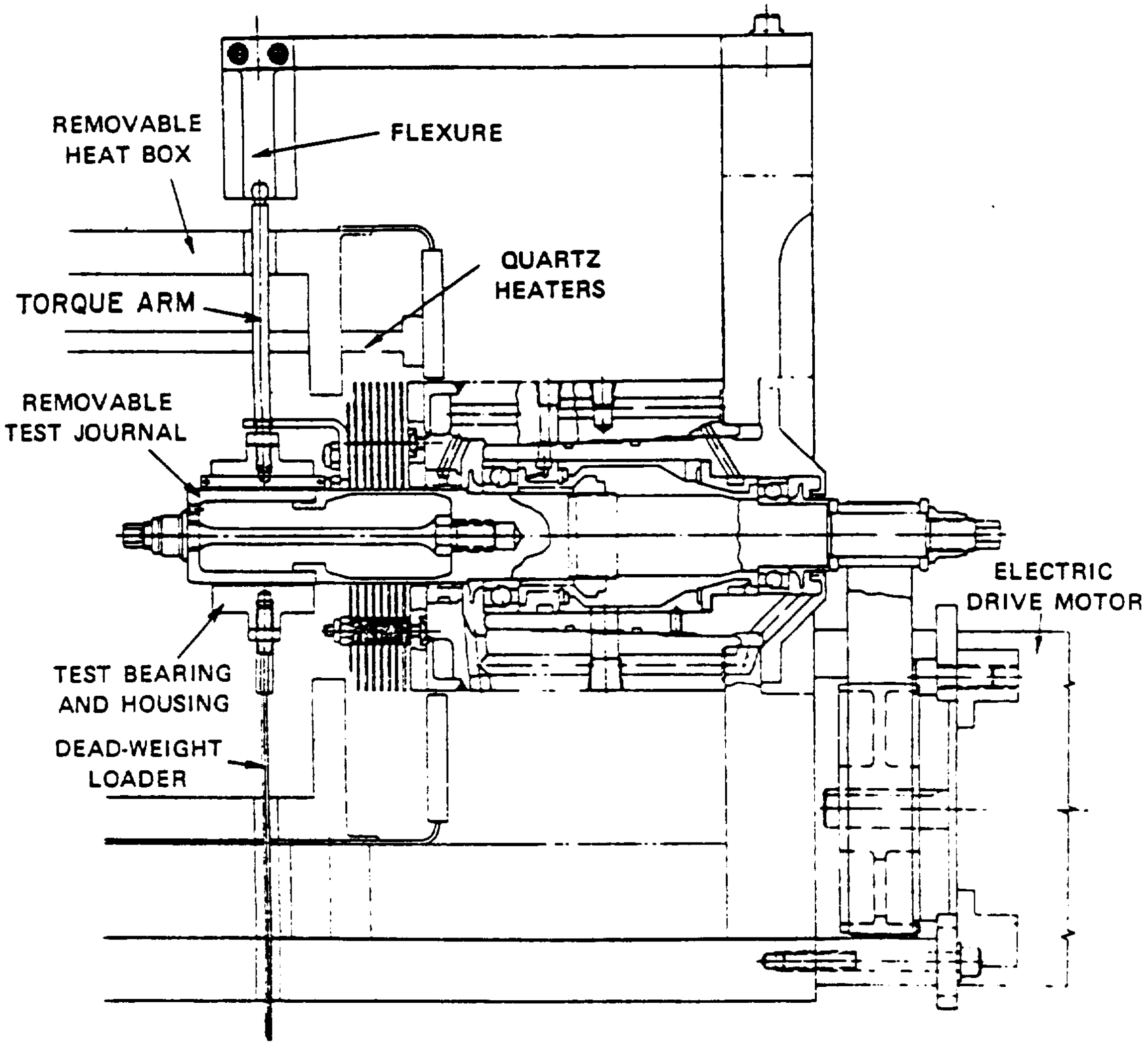

Fig. IV.1 Foil Journal Bearing Materials Test Rig

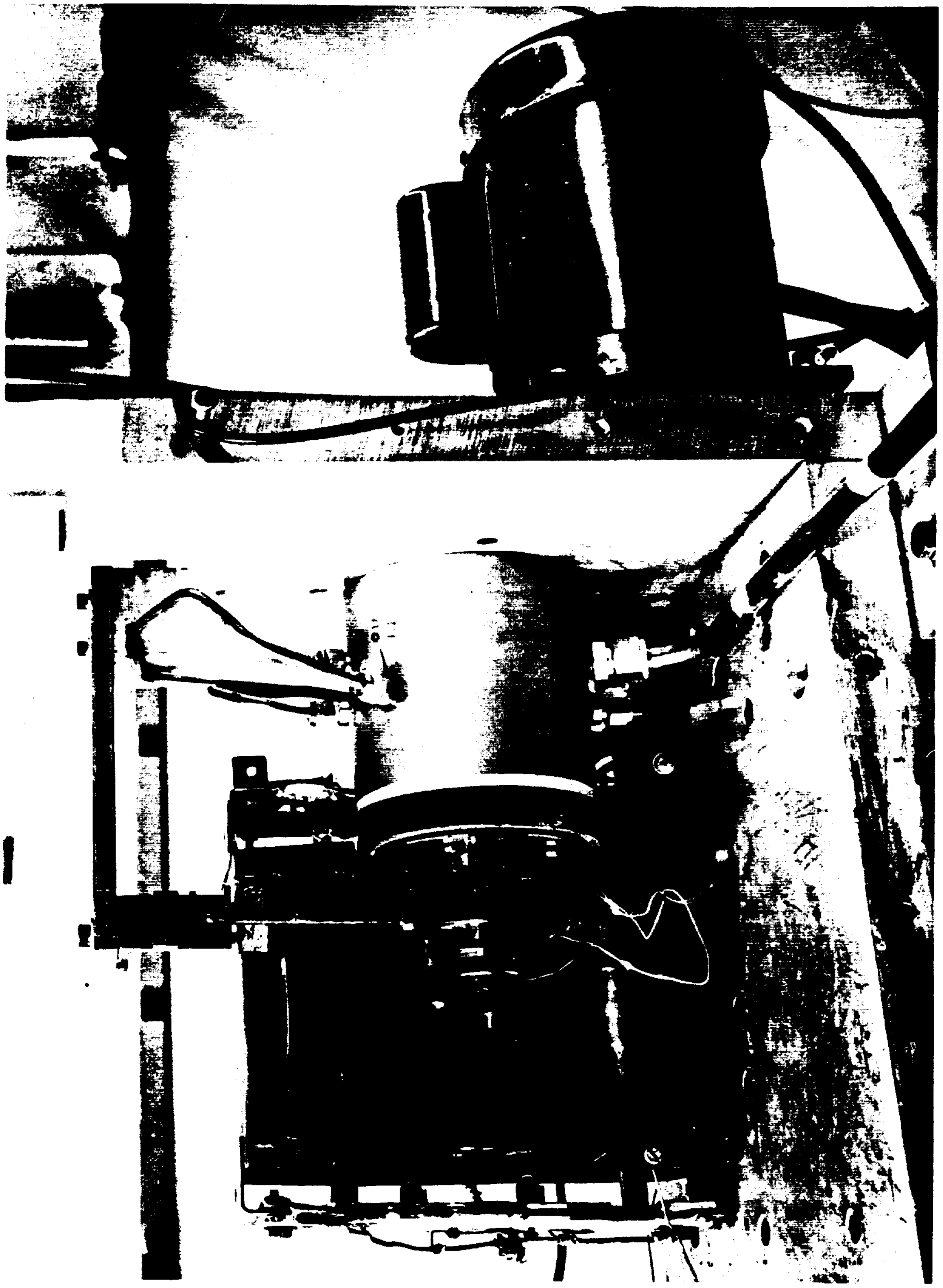

Fig. IV.2 Foil Journal Bearing Materials Test Rig

Fig. IV.3 Foil Journal Bearing Materials Test Facility

A heater box consisting of eight 500-watt quartz lamps was used to heat the test chamber to the required temperature. The test temperature was manually controlled by use of variacs to vary the voltage to the quartz lamps.

MEASUREMENTS AND INSTRUMENTATION

Rotor Speed

Rotational speed was measured by an MTI Fotonic Sensor™ fibre optic probe which responds to the once per revolution passing of a dark band painted on the test shaft. The output of the Fotonic Sensor was displayed on one channel of a two channel visicorder.

Test Temperature

The test bearing housing temperature was monitored using four (4) Type K, Chromel-Alumel thermocouples. The thermocouples were mounted on the outside of the housing 90° apart, then covered with a heat shield to prevent direct radiation from the quartz lamps. These thermocouples were used to monitor the test temperature. It had been determined during initial rig checkout that the test bearing temperature was essentially the same as that recorded on the bearing housing after the housing had been allowed to soak at the test temperature for 15 minutes. The output of the thermocouples were recorded on a Honeywell multipoint chart recorder.

Frictional Drag

The mechanical arrangement used to measure the frictional drag of the test bearing is shown in Figure IV.1. The floating foil bearing housing is restrained from rotation by a torque arm connected to the test bearing housing, and a flexure in the vertical plane acting through the bearing centerline. Bearing frictional drag causes deflection of the flexure which is measured by a capacitance proximity probe. The range of the capacitance probe used in the system was 0.254 mm (.010 inch). The output of the capacitance probe was recorded on one channel of a visicorder.

Test Bearing Load

Test bearing loading was accomplished by applying calibrated dead weights to the test bearing housing.

TEST BEARING AND TEST JOURNAL

The test bearing selected was a partial arc 38.1 mm (1.5 inch) diameter Hydresil^{TM} Journal Bearing. The test bearing and test journal are shown in Figure IV.4. The bearing diameter and mechanical design were based on the bearing used in Part I of this program. The bearing consists of a bump foil and a top or smooth foil. The smooth foil receives the coating to be evaluated. The two foils are individually attached to a "key" by spot welding and are separated by a spacer block. The key then fits into a slot in the floating bearing housing and is secured in place by tapered pins. This method of attaching the foils to the housing is not typical for hydresil applications, but did greatly facilitate changing test specimens while having the fewest number of test components.

To properly evaluate each coating combination, a newly coated foil was run against a clean journal surface which had not been tested against other foil coatings. To reduce the number of test journals required, a bearing of 19.05 mm (.75 inch) wide was used, which allowed the 44.5 mm (1.75 inch) wide journal surface to be used with two (2) foil coatings. The test bearing housing was indexed axially along the test sleeve to locate the test bearing over the appropriate section of the test journal.

A partial arc bearing was used rather than a complete bearing to simplify bearing fabrication and testing. The test bearing had a pad arc of approximately 186°. Through testing, it was determined that a pad of less than 180° resulted in rough bearing operation. The test bearing had one bump more than one half the total number of bumps in a complete circular bearing, which resulted in the 186° pad arc. Rotation of the journal was from the loose end into the weld. A complete L/D = 1, 360° pad bearing was used to evaluate the most promising coating combination identified from the partial arc testing.

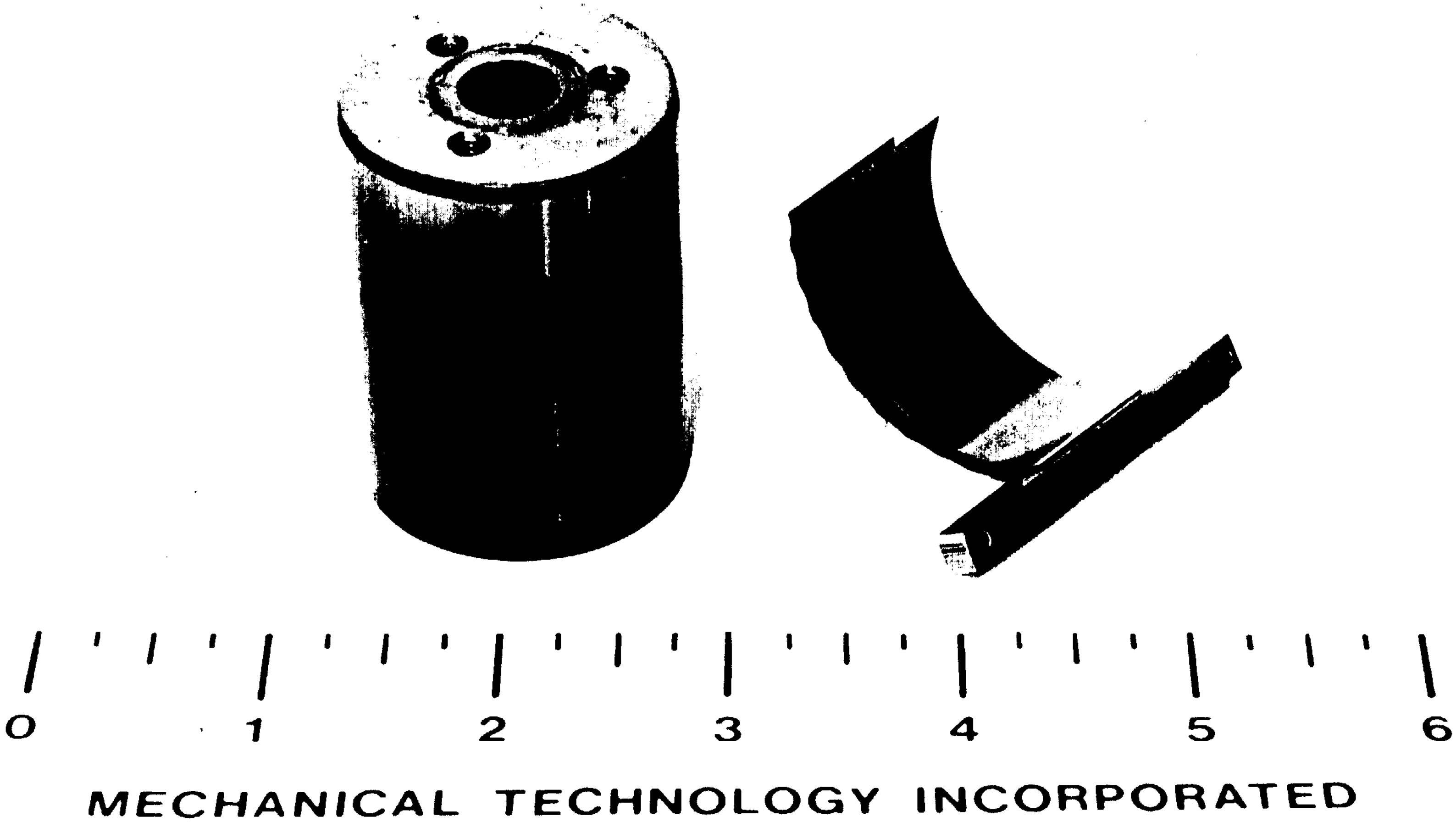

Fig. IV.4 Partial Arc Foil and Journal Test Specimens

Final machining of the journals was completed after the plasma sprayed coatings were applied and before the fused and sputtered coatings were applied. The journals were ground to be 5 μm (.0002 inch) round and 2.5 μm (.0001 inch) straight over the test area.

Typical static and dynamic runouts of the test journal surface after installation on the shaft were approximately 7.5 μm (.0003 inch).

V. MATERIAL START-STOP TEST RESULTS AND DISCUSSIONS

TESTING AND SCREENING TECHNIQUE

The start-stop tests were conducted with the facilities described in the previous chapter with one-half the number of test cycles at a test chamber temperature of either 540°C (1000°F) or 650°C (1200°F), and the other one-half at room temperature. The test format for the earlier batch of test specimens was as follows:

1. Five hundred cycles* at room temperature; 4 seconds on and 16 seconds off,
2. Five hundred cycles, at maximum temperature; 16 seconds on and 4 seconds off,
3. Repeat Step 1.
4. Repeat Step 2.

Later in the program, it was found beneficial to run the tests in reverse order, i.e., to run the tests at maximum temperature first. (This concept is discussed later in this report.)

During tests, the following test conditions were monitored and recorded:

- Serialization of specimens to include: materials, vendors, processing, and finishing data.
- Visual inspection of coatings before and after each 500 cycle period.
- Static breakaway torque of the bearing at the start and conclusion of each 500 cycles.
- Maximum starting torque.
- Ambient temperature.
- Maximum speed of spindle.
- Static and dynamic runout of test spindle.
- Unit loading.

---

* One cycle consists of one start and one stop.

Static breakaway friction torque was measured at the beginning of the test and after every 500 cycles of testing. After each 100 test cycles, a recorded trace of dynamic friction torque as a function of rotating speed during acceleration and deceleration was obtained for the purpose of estimating the general condition of the rubbing surfaces. Photographs of the journal and foil rubbing surfaces were taken periodically. The acceleration and deceleration of the drive motor was too fast to estimate the lift-off and touch-down speeds.

In the actual bearing application, the bearings are continuously flushed with high pressure air which should remove almost instantaneously any wear debris being formed at the sliding interface. To crudely simulate this in the start-stop tests, after every 500 cycles, the bearing and journal surfaces were flushed with air to remove any wear debris.

Initially, partial arc bearings of all the test combinations selected from static oven screening tests were made and tested to the maximum of 2000 start-stop cycles at approximately 14 kPa (2 psi) based on projected area. This was followed by one partial arc bearing start-stop test at increased loading with the most promising material combination, the unit projected specimen load in this test being 35 kPa (5 psi). Finally, a start-stop test of the most promising material combination resulting from partial arc tests was made using a complete foil bearing.

The following criteria were used to screen the start-stop test results:

- Static and dynamic breakaway friction torque at the start and conclusion of each 500 cycles.
- Visual inspection of the journal and the foil before and after each 500 cycles.
- Microscopic examination of the journal and the foil.

If the friction torque increased or the surfaces showed significant loss of coating or wear, the bearing was considered failed and the testing was terminated. Certain subjective judgment was required. Photographs of the bearing surfaces after the test and sometimes during the test were taken.

PARTIAL ARC BEARING TEST RESULTS AT 14 kPa (2 psi) LOADING

As explained in Section IV, foil bearing pads with a width of 19 mm (3/4") and a pad arc of 186° were made. This made it possible to conduct two separate tests on each journal (38 mm, bearing width) using parallel contact tracks. The results of the coating combinations tested are reported in Table V.1. The table shows the dynamic and static breakaway torques, surface roughnesses of the journal before and after tests, and comments on the surface appearance of the journal and bearing after test. Test No. 1 consisted of Linde Cr_3C_2 on the test journal and Hohman M-1284 (MoS_2 dry film) on the foil. This test was used as a baseline reference in evaluating rig performance and for the selected coating combinations. At the end of the 2000 start-stop cycle sequence which included: 500 cycles at room temperature; 500 cycles at 290°C (550°F); 500 cycles at room temperature; and 500 cycles at 290°C (550°F), both coatings were still in a serviceable condition with only light transfer of the dry film to the journal occurring. (See Figure V.1).

Test No. 2 consisted of CdO and Graphite (HL-800) coating on the foil and Det. Gun Cr_3C_2 on journal at 540°C (1000°F). After 500 start-stops at room temperature, the foil and journal were virtually unchanged. After 1000 start-stops, the journal was in serviceable condition, but the coating on the foil under the loaded region was worn through (See Appendix E for photographs of worn surfaces). It was apparent that 540°C (1000°F) was too high a temperature for the Graphite in the coating due to oxidation. The next test of this material combination (Test #9) was run at 370°C (700°F). At the end of the 2000 start-stop cycle sequence, the bearing and journal coatings were in serviceable condition with a light smooth polish of the foil coating mixture deposited on the journal (see Figure V.2). Even if the foil developed a bare spot, the light transfer coating deposited on the journal would provide adequate lubrication. It was judged that this combination would have performed through many more cycles. Talysurf traces of the journal before and after test are shown in Figure V.3. Visicorder traces of the test after 2000 start-stop are shown in Figure V.4.

In the next two tests (Nos. 3 and 4), plasma sprayed Tribaloy 800 was run against sputtered Titanium Carbide and Kaman DES coatings on the foil.

TABLE V.1

RESULTS OF TESTED COATING COMBINATIONS

Partial Arc Bearings

Load = 14 kPa (2 psi) based on bearing projected area

Test No.	Coating Combination	Foil and Journal Coatings	Max. Test Temp.	Breakaway Friction Coefficient						Surface Roughness of Journal µm (µ in.)		Results
					At Start	After 500 cycle	After 1000 cycle	After 1500 cycle	After 2000 cycle	Before	After	
1*	None Assigned (Baseline test)	Hohman M-1284 vs. Det. Gun. Cr_3C_2	290°C	S^1	.22	.22	.21	.28	.25	0.30 (12)	0.30 (12)	Completed test sequence at RT and 290°C. Both surfaces serviceable after test. Rated - successful.
				D^2								
2*	8	HL-800 vs. Det. Gun. Cr_3Cr_2	540°C	S	.19	.20				0.30 (12)	0.30 (12)	500 cyc. at RT - Foil and Journal OK. 500 cyc. at HT - coating worn through. Journal coating in serviceable condition. Rated - unsuccessful.
				D	.35	.43	.65					
3*	14	Sputtered TiC vs. Plasma Sp. Tribaloy 800	650°C	S	.27					0.71 (28)	0.66 (26)	Foil coating worn through after 30 cycles at room temperature. Journal surface grooved. Rated - unsuccessful.
				D	.48	.79 (30cyc)						
4*	17	Kaman DES vs. Plasma Sprayed Tribaloy 800	650°C	S	.29					0.71 (28)	0.81 (32)	Foil coating worn through after 10 cycles at room temperature. Journal surface grooved. Rated - unsuccessful.
				D	.5	.7 (10cyc)						
5*	None Assigned	HIT-Lube vs. Det. Gun. Cr_3C_2	540°C	S	.22	.23				0.30 (12)	-	500 cyc. at room temperature. 500 cyc. at HT - coating completely worn under load on foil. Journal OK. Coating cracked when foil was bent to form the bearing. Rated - unsuccessful.
				D	.52	.56	1.0					
6*	None Assigned	Kaman DES vs. Det. Gun. Cr_3C_2	650°C	S	.30					0.30 (12)	0.30 (12)	Foil coating worn through after 10 cycles at room temperature. Journal OK. Rated - unsuccessful.
				D	.5	.70 (10cyc)						
7*	10	Uncoated vs. Plasma Sprayed NASA PS-106	650°C	S	.29	.33	.23			0.30 (12)	Very Rough	500 cyc. at RT - foil had heavy wear and journal heavily scratched. 500 cyc. at HT - foil heavily scratched plus journal material deposits. Journal worn 90 µm (3-1/2 mils) Rated - unsuccessful.
				D	.37							
8*	12	Kaman DES vs. Plasma Sprayed NASA PS-106	650°C	S	.31	.20 (100cyc)				0.30 (12)	3.0 (120)	After 100 cyc. at room temperature coating on foil worn. Rated - unsuccessful.
				D	.5	3.0 (100cyc)						
9*	8	HL-800 vs. Det. Gun. Cr_3C_2	370°C	S	.20	.21	.20	.26	.25	0.30 (12)	0.23 (9)	Completed test sequence at room temperature and 700°F. Foil polished along all bumps. Journal has nice coating of graphite. Both surfaces serviceable. Rated - successful.
				D	.26	.30	.5	.33	.5			
10*	4	Sputtered Cr_2O_3 vs. Kaman DES	650°C	S	.56	.61				0.28 (11)	1.93 (76)	500 cyc. at RT - coating on foil and journal is completely worn off. Journal has a lot of scratches. Rated - unsuccessful.
				D	1.13	1.48						

TABLE V.1 (Cont'd)

RESULTS OF TESTED COATING COMBINATIONS

Partial Arc Bearings

Load = 14 kPa (2 psi) based on bearing projected area

Test No.	Coating Combination	Foil and Journal Coatings	Max. Test Temp.	Breakaway Friction Coefficient						Surface Roughness of Journal μm (μ in.)		Results
					At Start	After 500 cycle	After 1000 cycle	After 1500 cycle	After 2000 cycle	Before	After	
11**	6	Kaman DES vs. Kaman DES	540°C	S	.27	.34	.53	.41	.41	0.28 (11)	0.29 (11)	500 cyc. at HT and 300 cyc. at RT - journal and foil polished on bumps. In next 3000 cycles, foil and journal unchanged. Wear debris formed at interface during HT testing, amount decreased with cycles. Rated - successful.
				D	.43	.43	.73	.43	.70			
			650°C	S		.29	.41	.32	.43			
				D		.52 (2500 cyc)	.78 (3000 cyc)	.52 (3500 cyc)	.70 (4000 cyc)			
12**	3	Sputtered Cr_2O_3 vs. Sputtered Cr_2O_3	650°C	S	.21	.52	.51			0.13 (5)	1.78 (70)	500 cyc. at HT - coating worn off on journal and appears to have many scratches. Some bare metal over bumps on foil. 500 cyc. at RT - journal has shiny bare metal look and rough. Rated - unsuccessful.
				D	.65	.43	.70					
13**	7	Kaman DES vs. Sputtered Cr_2O_3	650°C	S	.30	.30	.43			0.13 (5)	1.07 (42)	500 cyc. at HT - journal had fine scratches, foil is polished over all bumps. 500 cyc. at RT - journal worn to bare metal; Kaman DES embedded to journal surface. Rated - unsuccessful.
				D	.39	.39	.78					
14**	15	Sputtered TiC vs. Sputtered Cr_2O_3	650°C	S	.24	.32				0.13 (5)	0.46 (18)	Journal has many scratches and coating is worn off. Foil coating changed color to light brown from steel grey. Fine scratches and coating worn off over "bumps". Rated - unsuccessful.
				D	.57	.48						
15*	13	HL-800 vs. Sputtered Cr_2O_3	370°C	S	.36	.30	.20	.21	.21	0.13 (5)	0.43 (17)	1000 cyc. at RT and 1000 cyc. at HT - foil polished along four bumps, journal has many fine scratches embedded with graphite. Rated - acceptable.
				D	.26	.30	.40	.30	.48			
16**	5	Sputtered Cr_2O_3 vs. Plasma Sprayed Tribaloy 800	650°C	S	.33	.30	.39	.47		0.91 (36)	0.23 (9)	500 cyc. at HT - journal and foil polished and shiny. 500 cyc. at RT - foil has bare metal on some bumps. 500 cyc. at HT - foil has loose powder and excessive metal showing on bumps. Journal polished and shiny. Rated - unsuccessful.
				D	.32	.52	.70	.48				
17**	16	Sputtered Si_3N_4 vs. Plasma Sprayed Tribaloy 800	650°C	S	.26	.37	.34			0.91 (36)	0.41 (16)	500 cyc. at HT - journal OK and foil coating worn through on some bumps. 500 cyc. at RT. Journal OK. Some bare metal on bumps. Some powder present on foil. Rated - unsuccessful.
				D	.35	.35	.61					

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TABLE V.1 (Cont'd)

RESULTS OF TESTED COATING COMBINATIONS

Partial Arc Bearings

Load = 14 kPa (2 psi) based on bearing projected area

Test No.	Coating Combination	Foil and Journal Coatings	Max. Test Temp.	Breakaway Friction Coefficient						Surface Roughness of Journal μm (μ in.)		Results
					At Start	After 500 cycle	After 1000 cycle	After 1500 cycle	After 2000 cycle	Before	After	
18**	2	Sputtered TiC vs Kaman DES	650°C	S	.25	.31	.47			0.33 (13)	0.51 (20)	500 cyc. at HT - journal had two scratches and some bare metal on foil. 500 cyc. at RT - journal had many fine scratches, foil down to bare metal in spots on bumps. Loose powder of Kaman DES on bumps. Rated - unsuccessful.
				D	.43	.43	.52					
19**	None Assigned	Uncoated vs. Plasma Sprayed NASA PS-120	650°C	S	.29	.41	.33			0.53 (21)	0.89 (35)	500 cyc. at HT - journal has many fine scratches and polished, loss in journal diameter 0.4 mil. Foil has a lot of scale, probably coating transferred from journal. 500 cyc. at RT - journal more polished, foil looks shinier with less scale. Rated - unsuccessful.
				D	.69	.61	.54					
20**	None Assigned	Uncoated vs. Plasma Sprayed NASA PS-120	540°C	S	.26	.39	.35	.42	.39	0.53 (21)	0.13 to 0.33 (5 to 13)	500 cyc. at HT - journal and foil polished slightly, some smearing of coating on foil. Rough stopping. 500 cyc. at RT and 500 at HT - slight build up of material on foil, journal has some scratches. 500 cyc. at RT - considerable polishing on foil and journal all over. Rough stopping. Rated - successful.
				D	.78	.87	1.13	1.04	1.04			

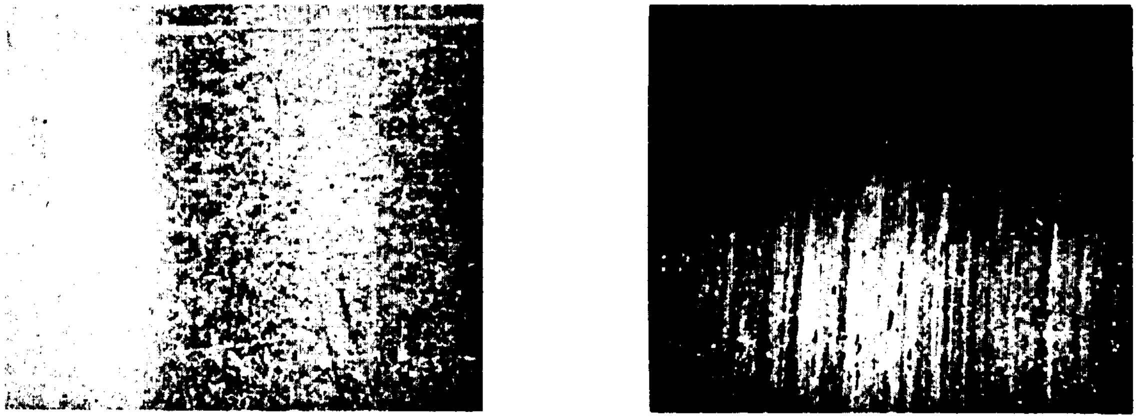
1 - Static at RT
2 - Dynamic at Test Temperature

* Test Cycle Sequence - 500 RT, 500 HT, repeated
**Test Cycle Sequence - 500 HT, 500 RT, repeated

M1284 ON FOIL 10X

Cr_3C_2 ON JOURNAL 10X

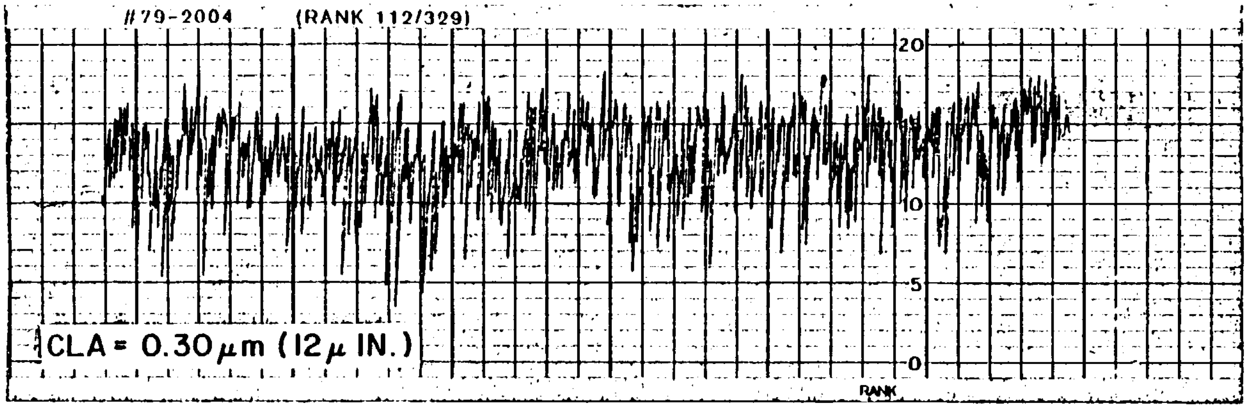
Fig. V.1 Photographs of Surfaces After Test at 1000 Cycles at RT and 1000 Cycles at 290° C (Test No. 1)



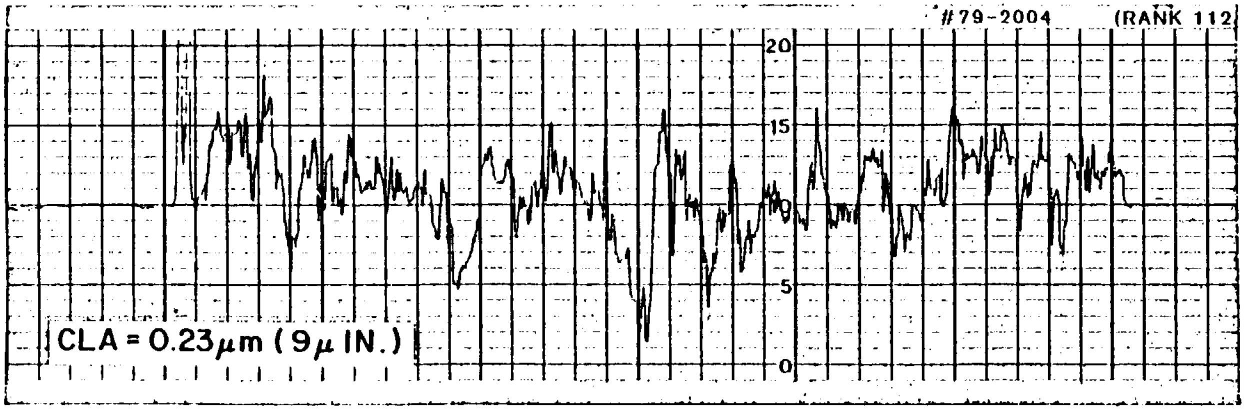
HL-800 ON FOIL 10X

Cr_3C_2 ON JOURNAL 10X

Fig. V.2 Photographs of Surfaces After Test at 1000 Cycles at RT and 1000 Cycles at 370° C (Test No. 9)



BEFORE TEST



AFTER TEST

VERTICAL MAG.: EACH SMALL DIV. = 0.13μm

HORIZONTAL MAG.: EACH SMALL DIV. = 250μm

Fig. V.3 Talysurf Traces of Chrome Carbide Coated Journal Tested Against HL-800 Coated Foil at Maximum Test Temperature 370°C (Test No. 9)

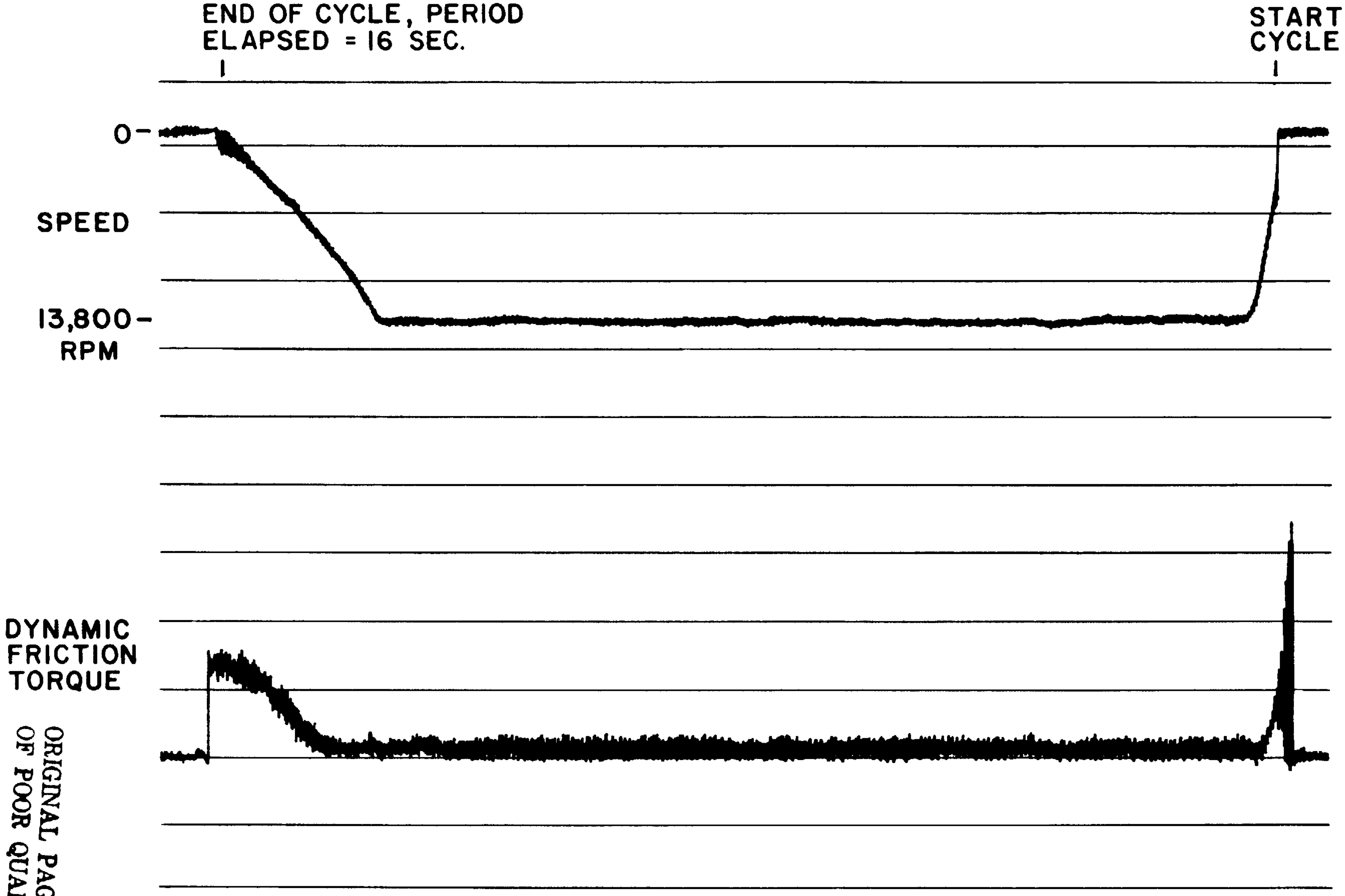


Fig. V.4 Visicorder Trace of Coating Combination Cr_3C_2 Versus HL-800 After 2000 Start-Stop Cycles

After a few cycles at room temperature, the tests were stopped in each case due to high dynamic breakaway torque readings, and the specimens were examined. Journals were grooved and foil coatings worn through in several locations under the loads (photographs of worn surfaces are included in Appendix E).

Since both foil coatings tested with the Tribaloy 800 journal coating caused the journal surface to groove, it was decided to repeat one of the foil coatings against a Cr_3C_2 journal surface. Kaman DES coating for the foil was selected (Test No. 6). After 10 cycles at room temperature, the dynamic torque had increased and the test was stopped. The foil coating was again worn through under the load region, but there appeared to be no damage to the journal surface (for photographs see Appendix E). It was concluded at that time that the Linde Cr_3C_2 was a better journal surface than Tribaloy 800 under these test conditions.

A new dry film lubricant, Hi-T-Lube (a proprietary coating of General Magnaplate Corporation, Linden, New Jersey, believed to be based on MoS_2 and recommended for 540°C operation) was put on the foil. The coating cracked slightly during bending of the foil. The bearing with this coating was operated against Linde Cr_3C_2 journal (Test No. 5). The coating was worn under the loaded region after 1000 cycles (for photographs see Appendix E).

None of the combinations in tests numbered 7, 8, and 10 survived the room temperature cycles.

In the test set-up, the shaft was rigidly mounted and the bearing was free to float and move axially along the shaft approximately 1.3 mm (0.050 in.). In machinery application, the bearing is usually ridigly mounted and the axial motion of the shaft is restricted by a thrust bearing. It was felt that this axial motion of the bearing in our tests might fatigue the interface particles. Since the axial motion does not exist in real application; after the eighth test it was restricted to about 130-250 μm (5-10 mils) of motion.

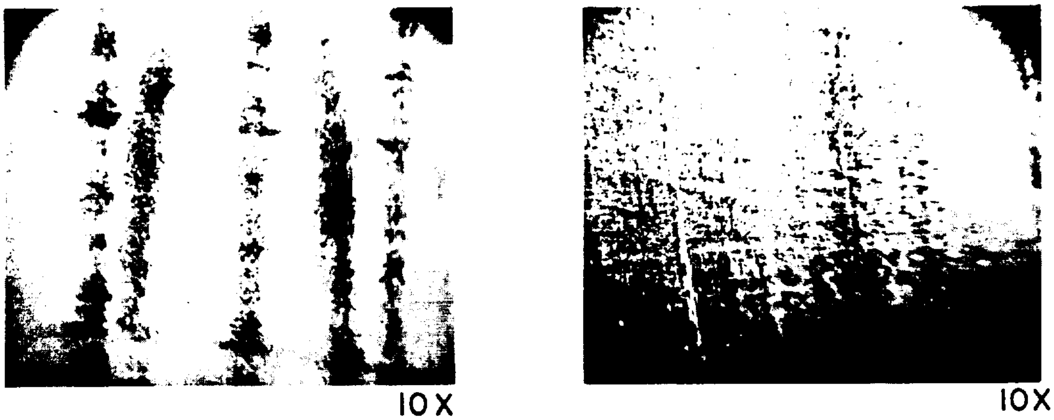
It was anticipated that the hard coatings would behave better at high temperature for the following reason: preoxidation of the coating combinations provides a thin protective oxide layer; many ceramic combinations can interact at higher temperatures to form eutectic compounds which behave as low shear strength solid films. In addition, continuous exposure of the bearing surfaces to oxygen at high temperatures during testing may replenish the oxide film (reactive replenishment concept).

The bearing loading under sliding contact is localized and, as a result, the thin coatings could be damaged. A reactive replenishment process could re-form the coating by surface oxidation and save it from destruction during the run-in period.

After Test No. 10, the hot cycles were run first in order to encourage rapid initial oxidation, and the test cycle was changed to the following:

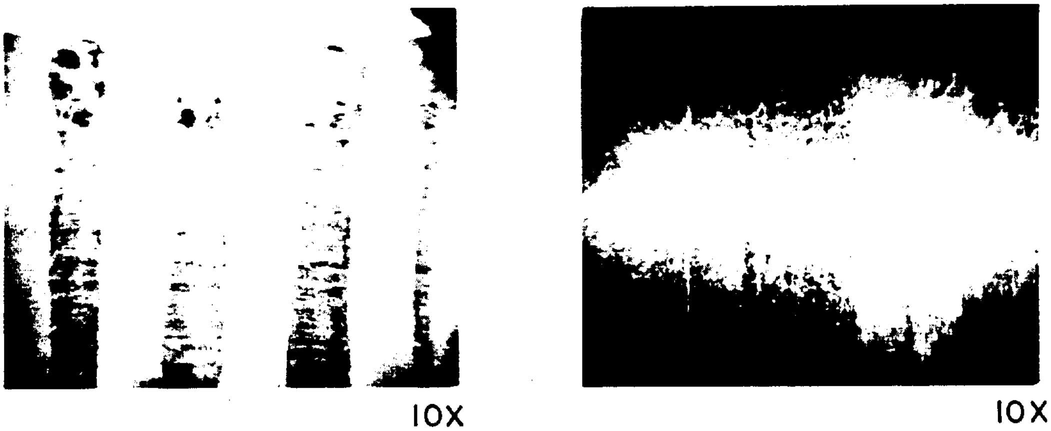
- 500 cycles at maximum test temperature; 16 seconds on and 4 seconds off.
- 500 cycles at room temperature; 4 seconds on and 16 seconds off.
- Repeat the sequence.

The Kaman DES coating on journal and foil (Test No. 11) successfully completed 1000 cycles at 540°C (1000°F) and 1000 cycles at room temperature. Since bearing surfaces after 2000 start-stop cycles were in serviceable condition, they were tested for an additional 1000 cycles at 650°C (1200°F) and 1000 cycles at room temperature. Foils and journals were polished in the first 1000 cycles. During the last 3000 cycles, there was practically no change in their surface appearances (for photographs of surfaces, see Figure V.5). In the first 1000 cycles, some wear debris was probably collected at the interface since dynamic friction traces showed a significant amount of oscillations even when lift-off had occurred. The oscillations almost disappeared as the testing continued. It is to be noted that, in an actual bearing application, the bearings are continually flushed with high pressure air which should remove almost instantaneously any wear debris being formed. The Talysurf traces of the journal before and after test are shown in Figure V.6.



KAMAN DES ON FOIL
AFTER 1000 CYCLES
AT 540°C AND 1000
CYCLES AT RT

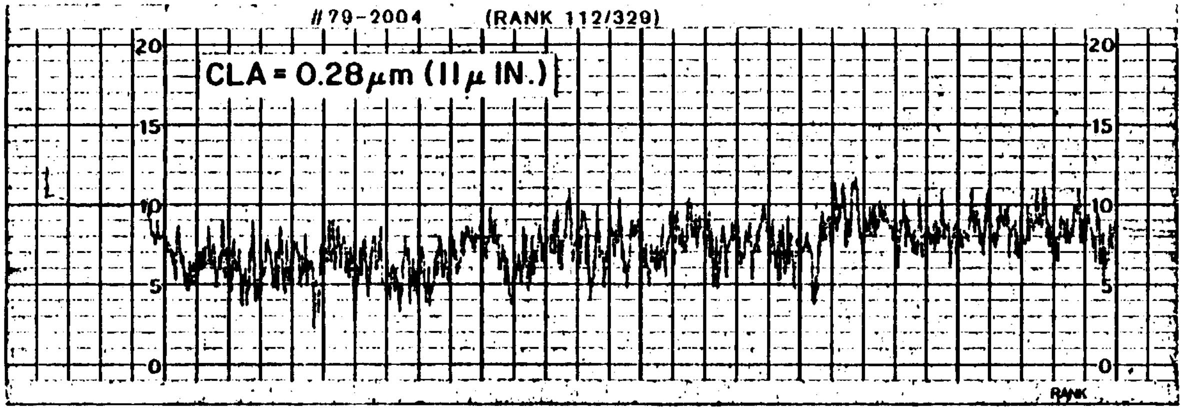
KAMAN DES ON JOURNAL
AFTER 1000 CYCLES
AT 540°C AND 1000
CYCLES AT RT



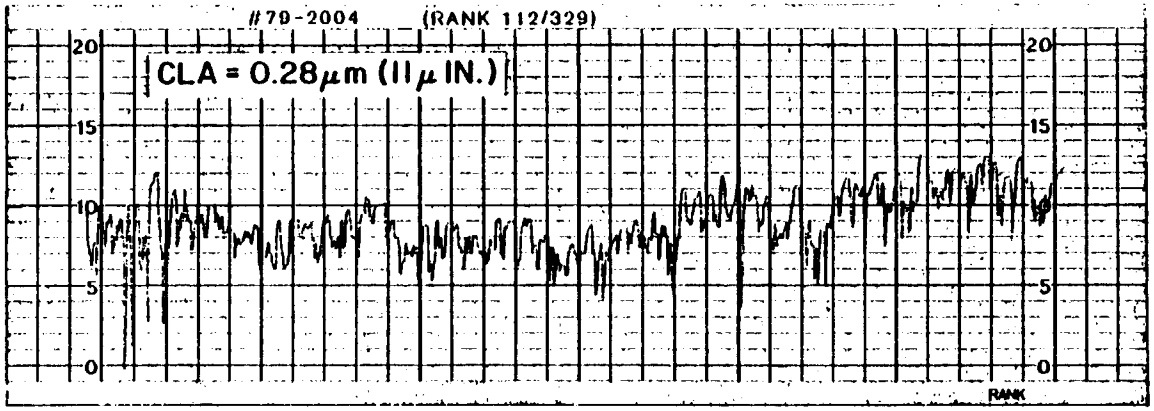
KAMAN DES ON FOIL
AFTER 1000 CYCLES AT
540°C, 1000 CYCLES
AT 650°C AND 2000
CYCLES AT RT

KAMAN DES ON JOURNAL
AFTER 1000 CYCLES AT
540°C, 1000 CYCLES AT
650°C AND 2000 CYCLES
AT RT

Fig. V.5 Photographs of Surfaces After Test (Test No. 11)



BEFORE TEST



AFTER TEST

VERTICAL MAG: EACH SMALL DIV. = .25μm

HORIZONTAL MAG: EACH SMALL DIV. = 250μm

Fig. V.6 Talysurf Traces of Kaman DES Coated Journal Tested Against Kaman DES Coated Foil After Test for 1000 Cycles at 540°C, 1000 Cycles at 650°C and 2000 Cycles at RT (Test 11)

After successful testing of Kaman DES versus Kaman DES, tests were conducted for maximum of 2000 start-stop cycles on sputtered Cr_2O_3 versus itself; Kaman DES versus sputtered Cr_2O_3; and sputtered TiC versus sputtered Cr_2O_3 (Tests 12 through 14). In all three tests, coatings came off and the surfaces became rough (for photograph of the surfaces see Appendix E).

The next test (numbered 15) consisted of HL-800 on the foil and sputtered Cr_2O_3 on the journal and was conducted at a maximum test temperature of 370°C (700°F). After completion of the test, the journal had some fine scratches and the foil surface was polished at a position corresponding to four support foil bumps (see Figure V.7). The coating was rated acceptable. However, the combination 8 in Test 9 is preferred over combination 13 in Test 15. The next three tests (16 through 18) consisting of coating combinations sputtered Cr_2O_3 versus Tribaloy 800; sputtered Si_3N_4 versus Tribaloy 800; and sputtered TiC versus Kaman DES, did not pass through the complete test.

H. Sliney at NASA-Lewis modified the composition of the coating designated NASA PS 106 and formulated a new coating, NASA PS 120, consisting of 60 percent Tribaloy 400, 20 percent silver, and 20 percent calcium fluoride. This formulation substitutes the laves phase cobalt alloy Tribaloy 400 for the nichrome that has been used in the NASA PS 106 coating. The coating was applied on the journal and was unsuccessfully tested against uncoated foil at 650°C (1200°F), Test No. 19. A lot of journal coating was smeared on the foil surface. It was apparent that the journal coating became soft at 650°C (1200°F). It was felt that the coating combination might perform well at 540°C (1000°F). The next test was repeated at 540°C (1000°F), Test No. 20. After the 2000-cycle test sequence, the journal had some fine scratches and some of the journal coating had transferred and lightly smeared onto the foil (see Figure V.8). Talysurf traces of the journal before and after test are shown in Figure V.9. A Visicorder trace during the test after 1000 cycles is shown in Figure V.10. The coating combination was rated successful, but it is felt, however, that more work will be needed on the coating to improve its hardness and reduce the porosity.

HL-800 ON FOIL 5X

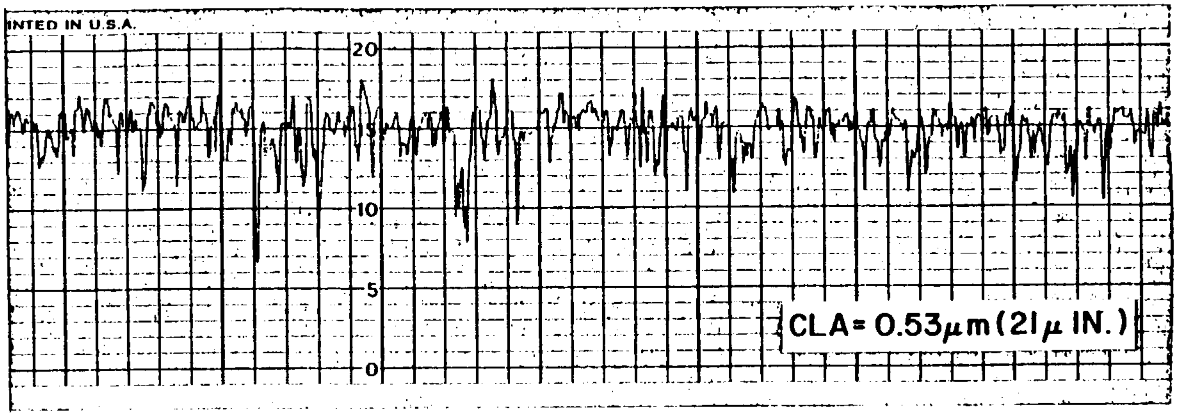
Cr_2O_3 ON JOURNAL 5X

Fig. V.7 Photographs of Surfaces After 2000 Cycles (Test No. 15)

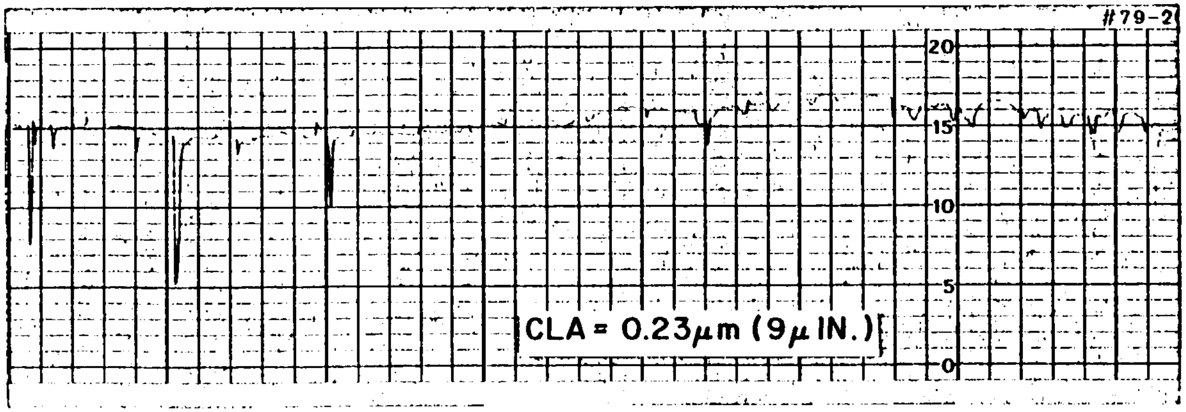
UNCOATED FOIL 2X

NASA PS120 ON JOURNAL 5X

Fig. V.8 Photographs of Surfaces After Test for 1000 Cycles at 540° C and 1000 Cycles at RT (Test No. 20)



BEFORE TEST



AFTER TEST

VERTICAL MAG: EACH SMALL DIV. = 0.5μm

HORIZONTAL MAG: EACH SMALL DIV. = 250μm

Fig. V.9 Talysurf Traces of NASA PS120 Coated Journal Tested Against Uncoated Foil for 1000 Cycles at 540°C and 1000 Cycles at RT (Test No. 20)

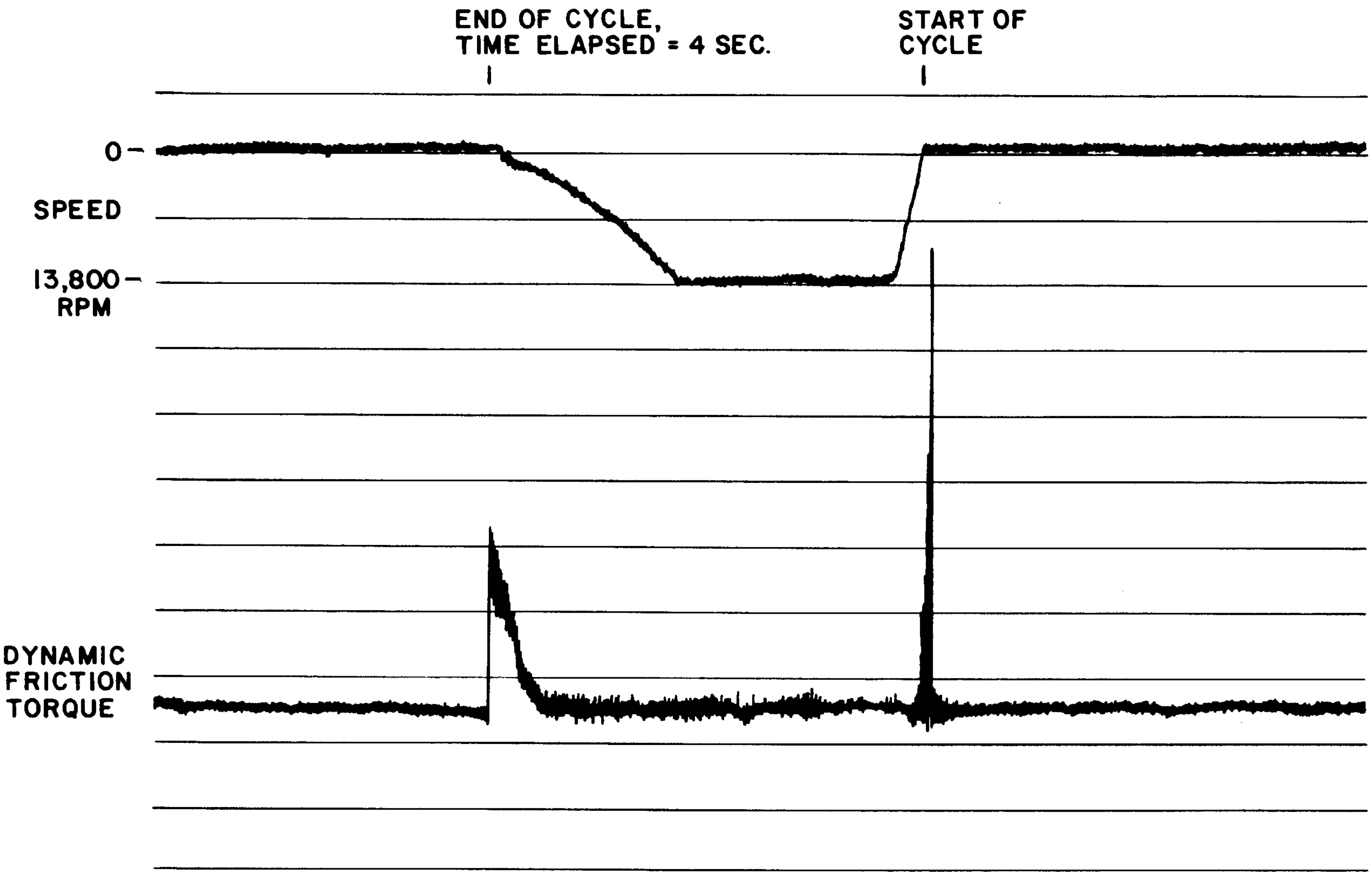


Fig. V.10 Visicorder Trace of Coating Combination NASA PS 120 Versus Uncoated Foil After 500 Cycles at 540° C and 500 Cycles at RT (Test No. 20)

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One of the coatings scheduled to be tested in start-stop tests, chrome di boride (Plasma sprayed), coating on journal could not be ground. The coating was very soft and porous. The coating was repeated on a second batch of journals and coating again was found to be soft. No other vendor especially experienced in CrB_2 could be located. Consequently, test combinations 1, 9, and 18 in Table III.5 were not tested.

In reviewing the partial arc testing, it was evident that none of the sputtered coatings performed well. It was believed that the cause of the difficulties might be two-fold: the coatings may be too thin and the bonds may not be good enough. A metallurgical examination of a sputtered coating at this stage was considered necessary to gain an understanding of the bonding mechanism. A Si_3N_4 coated foil was rolled and examined under scanning electron microscope and no cracks were found (Figure V.11). A Si_3N_4 coated foil was sectioned prior to any testing and examined under SEM, and X-REDA analysis was also carried out. Figure V.12 shows the line scan and X-ray image of Si on the sectioned foil. It was found that the silicon nitride coating was well bonded and of the intended proper thickness (5000°A).

Despite this, the sputtered coatings could be easily polished off by an emery paper. It appeared that although silicon nitride coating seemed to be well bonded in the as-sputtered condition, it demonstrated a poorer bond during sliding test. Some further work on the improvement of the bond of the sputtered coatings during sliding should be carried out.

PARTIAL ARC BEARING TEST RESULTS AT 35 kPa (5 psi) LOADING

The most promising candidate Kaman DES versus Kaman DES, which was shown to have the capability to operate up to 650°C (1200°F), was tested at 35 kPa (5 psi) loading. The results of the combination tested are reviewed in Table V.2. After completion of this test the journal looked virtually unchanged and the foil had some microscopic patches of bare metal showing which were very shiny and smooth (Figure V.13).

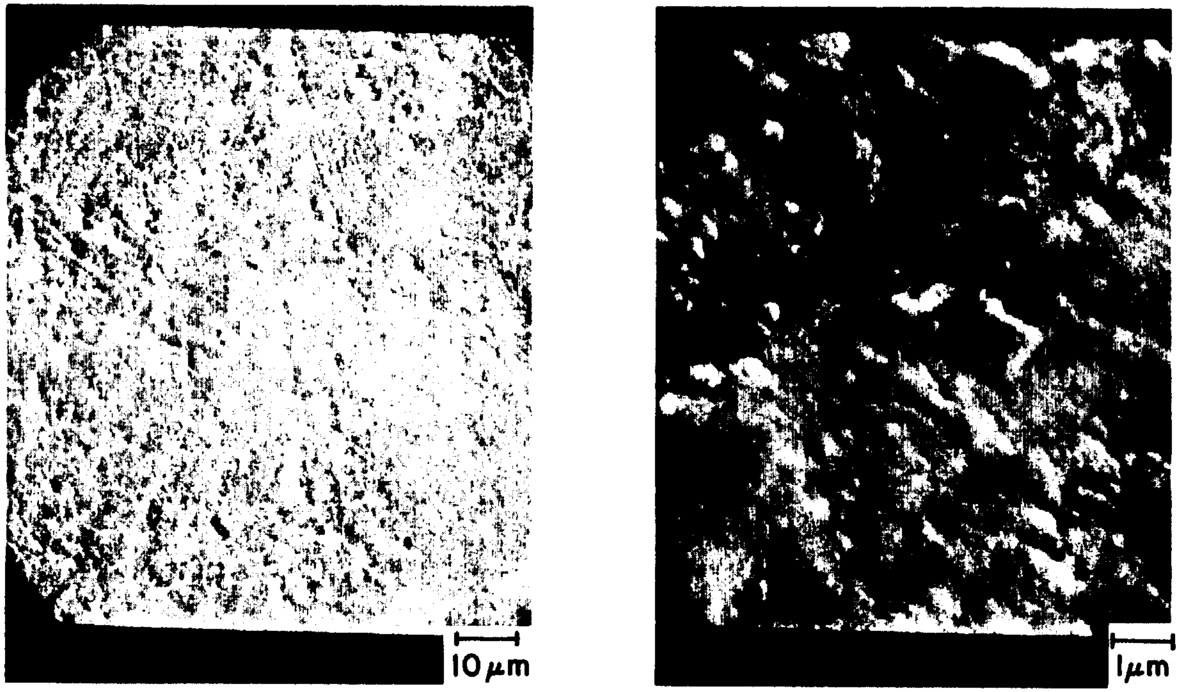
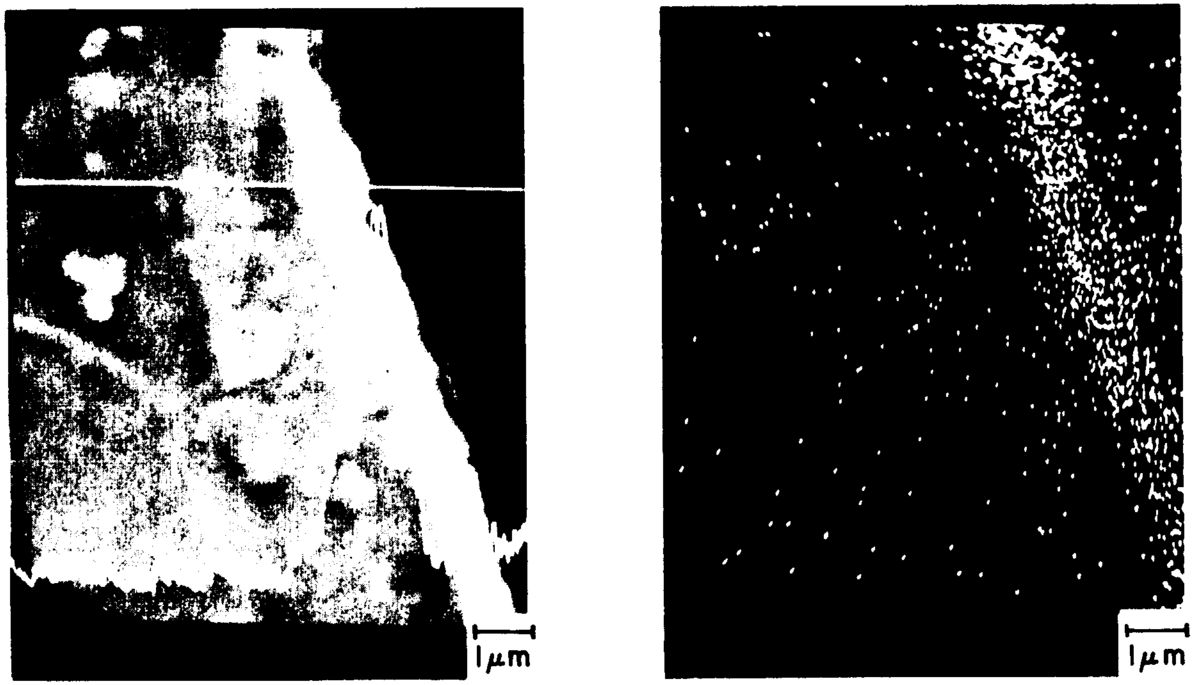


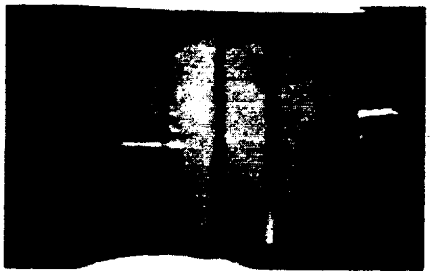
Fig. V.11 SEM Photograph of Sputtered Silicon Nitride on Inconel X-750 Foil, After Rolled



LINE SCAN OF Si

X-RAY IMAGE OF Si

Fig. V.12 SEM Micrographs of the Cross Section of Sputtered Silicon Nitride Foil



5X

KAMAN DES ON FOIL AFTER
500 CYCLES AT 650°C
AND 500 CYCLES AT RT

5X

KAMAN DES ON FOIL AFTER
1000 CYCLES AT 650°C
AND 1000 CYCLES AT RT

5X

KAMAN DES ON JOURNAL
AFTER 1000 CYCLES AT
650°C AND 1000 CYCLES
AT RT

Fig. V.13 Photographs of Surfaces After Test (Test No. 21)

TABLE V.2

RESULTS OF TESTED COATING COMBINATIONS

Partial Arc Bearings

Load = 35 kPa (5 psi) based on bearing projected area

Test No.	Coating Combination	Foil and Journal Coatings	Max. Test Temp.	Breakaway Friction Coefficient						Surface Roughness of Journal µm (µ in.)		Results
					At Start	After 500 cycle	After 1000 cycle	After 1500 cycle	After 2000 cycle	Before	After	
21**	None Assigned	Kaman DES vs. Kaman DES	650°C	S[1]	.31	.32	.51	.33	.48	0.33 (13)	0.36 (14)	500 cyc. at HT-Journal and foil lightly polished, some brown powder on foil. 500 cyc. at RT-Journal has three fine scratches and some bare metal showing on foil. 500 cyc. at HT and 500 cyc. at RT-very fine scratches on journal filled with loose powder. Foil has some bare metal with microscopic patches and shiny appearance. Rated-successful.
				D[2]	.28	.42	.52	.52	.52			

**Test cycle sequence - 500 HT, 500 RT and repeated.

1 - Static at RT
2 - Dynamic at test temperature

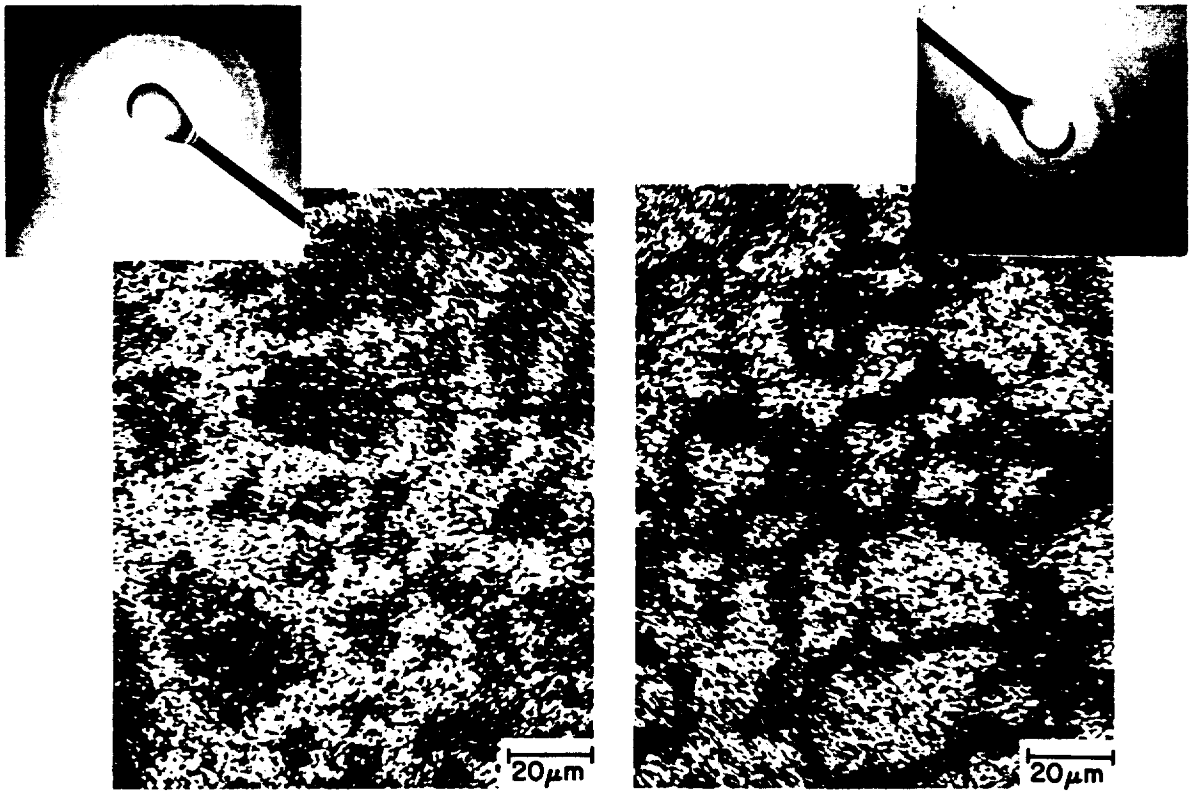
A metallurgical examination was conducted to determine the composition and condition of the shiny, heavily loaded zone. Reflection electron diffraction was performed. It yielded two different patterns. One of the patterns was identified as NiO. The second pattern was harder to interpret and was tentatively identified as elemental chromium (for diffractograms, see Figure V.14 (b) and (c)).

The sample was then examined in the Scanning Electron Microscope (SEM) equipped with an X-ray energy dispersive analyzer. The SEM photographs in Figures V.14 and V.15 were taken in the back-scattered electron image mode. The yield of back-scattered electrons from a sample increases with increasing atomic number. Therefore, the lighter colored zones seen in Figures V.14 (a) must be of higher average atomic number than the darker zones. X-ray analysis bears this out as it shows the lighter colored areas to be rich in nickel while the darker colored areas are richer in chromium (Figures V.14c and V.14b, respectively). The rough, granular appearance of the chromium rich area resembles that of the less worn, coated area seen in Figure V.15. The nickel rich areas are likely to be the Inconel substrate showing through where the coating has worn away.

Figure V.15 provides a comparison of the shiny, heavily worn and dull, lightly worn areas.

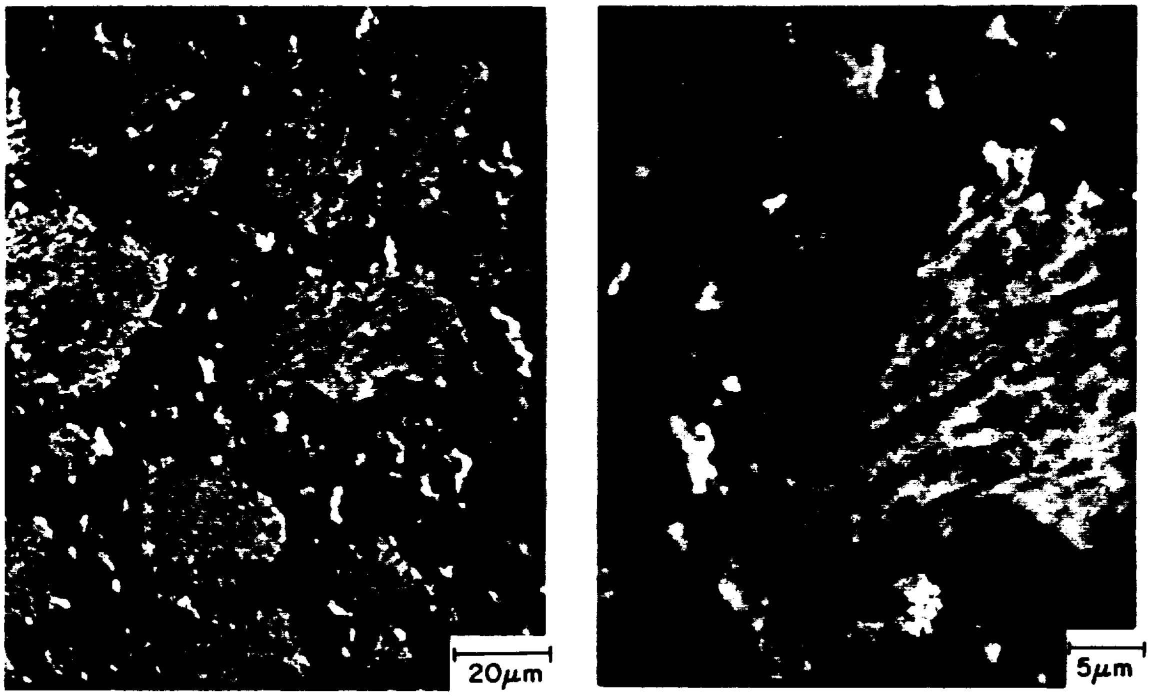
It was concluded from the analysis that the patches of the remaining coating found on the heavily loaded area are rich in chromium and the worn surface is NiO which resulted from Inconel surface oxidation during testing. The structure of the patches almost gives the impression that they coincide with the grain boundaries. It is hypothesized that, during the coating process of Kaman DES, first the chromium from the slurry is bonded to grain boundaries, and the chromium deposited later is oxidized with liberated oxygen available in the slurry and, as a result, Cr_2O_3 is deposited on the surface. As indicated earlier, the bulk of Kaman DES coating is essentially Cr_2O_3. After testing, the later deposited Cr_2O_3 is all worn away and the initially deposited chromium is still present on the surface. This may mean that the initially

(a) BACK SCATTERED ELECTRON IMAGE

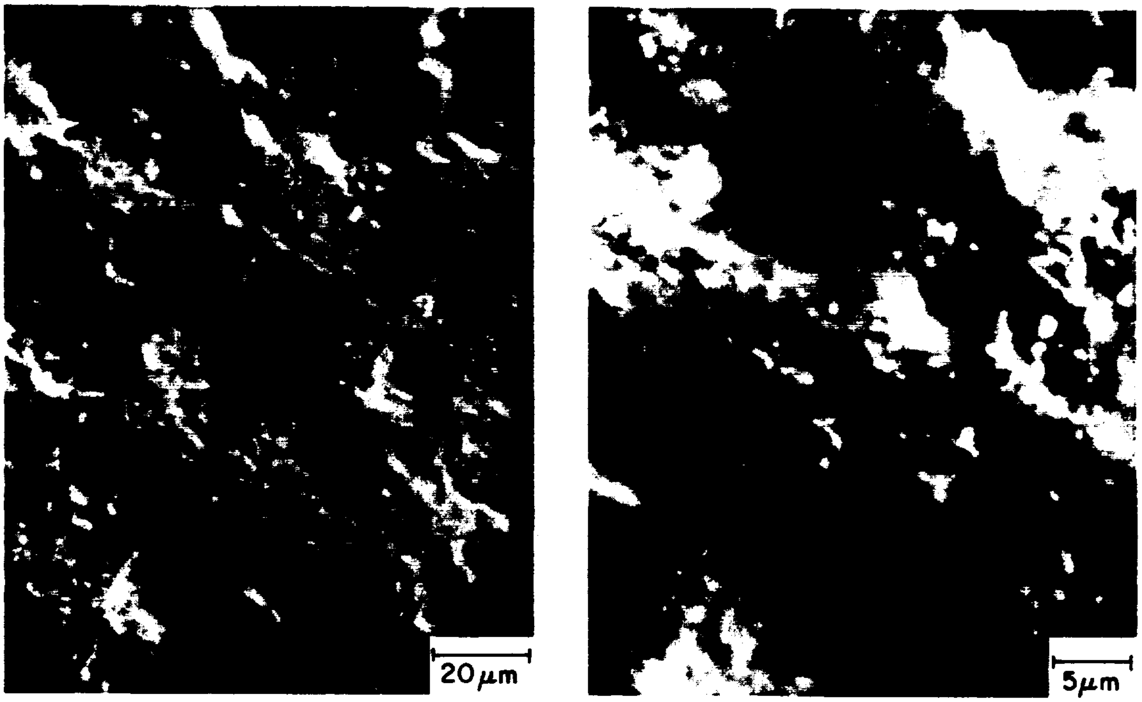


(b) Cr Kα X-RAY IMAGE (c) Ni Kα X-RAY IMAGE

Fig. V.14 SEM Micrographs and Diffractographs of Kaman DES Coated Inconel X-750, Heavily Worn Area (Test No. 21)



(a) HEAVILY WORN AREA (b) HEAVILY WORN AREA



(c) LIGHTLY WORN, COATED AREA (d) LIGHTLY WORN, COATED AREA

Fig. V.15 SEM Micrographs of Kaman DES Coated Inconel X-750 (Test No. 21) deposited chromium (inter-metallic layer) has a really good bond and this may be the key to the overall good bond and, as a result, better wear life of Kaman DES coating as compared to sputtered Cr_2O_3.

START-STOP TESTS OF COMPLETE BEARINGS

A complete foil bearing 38 mm dia x 38 mm wide (1½" dia x 1½" wide), tested with a Kaman DES coating on journal and foil, was conducted at 14 kPa (2 psi) loading based on bearing projected area. The results are reported in Table V.3. There was a considerable amount of loose wear debris formation at the interface which apparently could not easily escape, and, as a result, it damaged the surface. The test was discontinued after 1000 cycles consisting of 500 cycles at 650°C (1200°F) and 500 cycles at room temperature because there were several bands of bare metal on the journal. The foil was polished over all bumps and several bands of polished marks were running across the width of the foil (see Figure V.16).

A repeat test should be conducted with a complete bearing before any conclusions can be drawn. The wear may be the result of poor coating adhesion of this batch or even bearing foil irregularities.

TABLE V.3

RESULTS OF TESTED COATING COMBINATION

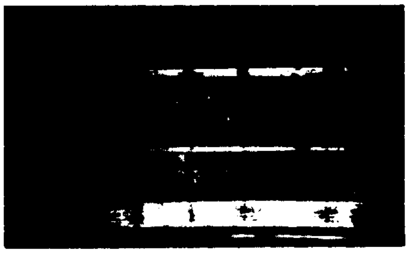
Full Width Complete Bearings

Load = 14 kPa (2 psi) based on bearing projected area

Test No.	Coating Combination	Foil and Journal Coatings	Max. Test Temp.	Breakaway Friction Coefficient						Surface Roughness of Journal µm (µ in.)		Results
					At Start	After 500 cycle	After 1000 cycle	After 1500 cycle	After 2000 cycle	Before	After	
22*	None Assigned	Kaman DES vs. Kaman DES	650°C	S^1	1.0	0.8	0.90	--	--	0.28 (11)	0.46 (18)	500 cyc. at HT - Fine scratches on journal, foil polished on all bumps. Several bands of polish marks running circumference of foil. 500 cyc. at RT - Several bands of bare metal on journal and foil. Rated - unsuccessful.
				D^2	1.3	.53	1.48					

*Test cycle sequence - 500 HT, 500 RT and repeated.

1 - Static at RT
2 - Dynamic at test temperature



KAMAN DES ON FOIL

KAMAN DES ON JOURNAL

Fig. V.16 Photographs of Surfaces After 500 Cycles at 650° C 500 Cycles at RT (Test No. 22)

VI. CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The most promising coating combinations from the program aimed at developing material coatings to withstand start-stop cycles in a 540° - 650°C (1000° - 1200°F) environment for an air lubricated compliant journal bearing, are listed below:

Foil Coating	Journal Coating	Maximum Temp °C (°F) Test
HL-800™ (CdO and Graphite) 8-10 μm thick	Det. Gun and Ground Ni-Cr bonded Cr_3C_2 60-90 μm thick	370* (700)
Uncoated and Heat-treated	Plasma Sprayed NASA PS 120 with Ni-Aluminide undercoat 140-165 μm thick	540 (1000)
Kaman DES (Proprietary Cr_2O_3) 1.3 - 2.5 μm thick	Kaman DES 8-13 μm thick	650 (1200)

The combinations listed above have completed a total 2000 start-stop cycles each, consisting of 1000 cycles at maximum test temperature and 1000 cycles at room temperature at a load of 14 kPa (2 psi) in partial arc bearing tests. Kaman DES - Kaman DES has also completed the 2000 start-stop cycles at a load of 35 kPa (5 psi) in partial arc bearing tests.

It is believed that the intermetallic layer of chromium in the Kaman DES coating may be responsible for the good coating bond and, as a result, provide better wear life.

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*It can probably be used up to 400°-430°C

It is believed that the hard coatings behave better if they are run at high temperature first for the following reasons: Preoxidation of the coating combinations provides a thin protective oxide layer and many ceramic combinations can interact at higher temperatures to form eutectic compounds which behave as low shear strength solid films; and also oxidation of the bearing surfaces at high temperatures during testing replenishes the oxide film continuously (reactive replenishment). This may be especially important during the run-in period.

The Kaman DES/Kaman DES coating system in a full bearing test did not do very well. It is tentatively concluded that the wear debris collected at the interface could not escape and did most of the damage. It is also possible that the coating bond might not have been adequate.

Even well bonded coatings will wear and as a result there is loose debris collected at the interface. In the case of hard coatings (e.g. Cr_2O_3 and Cr_3C_2 etc.), it is important that the formation of the wear particles be minimized and that whatever debris is formed should escape from the bearing interface; otherwise, it could be abrasive. Journal coatings are put on by plasma spraying and are relatively thick and well bonded, and usually show little wear. The foil coating, due to the foil flexibility, takes a lot of abuse, especially during starting. In order to get adequate coating retention on a thin foil which goes through flexing during operation, the coating should be very thin. The successful hard coating Kaman DES is only 1.3 - 2.5 μm (50-100 μ in.) thick and has worked very well. In some prior work, thicker coatings of this composition have presented problems. It is concluded that the thin coatings of hard materials (less than 2.5 μm (100 μ in.)) are ideal for the foils.

RECOMMENDATIONS FOR FUTURE RESEARCH

In these studies a baseline has been established at different operating temperature levels with three coating combinations. It is recommended that future work have the following major objectives:

- Apply the major effort to continuing the material and process development on the three most promising combinations.

- Maintain a parallel effort on both hard and soft coatings because of potential problems with loose abrasive particles associated with hard coatings.
- Introduce new candidate combinations for advanced automotive gas turbines.

The most promising coating systems identified should be subjected to a careful review to determine what variables might be significant in affecting and improving long life performance. Quality control of coating processes and methods should be established.

Formation of the wear debris in hard coatings seems to be a problem as the wear particles may be abrasive. Techniques should be developed to remove the particles from the bearing interface. Forced air may be introduced to remove the particles which is consistent with the real application.

Because of the above possible difficulties with hard surface coatings, it is believed that a parallel emphasis should be maintained on the softer coating. $PbO.SiO_2$ coating acts as a good lubricant in 480° - 650°C (900°-1200°F) range. Since interface temperatures may be quite high even in room temperature ambient due to rubbing before lift off, the coating may function reasonably well over the entire temperature range. Additives such as silver should be explored to broaden the temperature range.

A technique should be developed to improve the bond and ductility of the coatings put on by sputtering.

Both the new cobalt base alloy coating L103, and Zirconates, which successfully completed oven screening test should be further evaluated.

The oxide treatment of cemented carbide tools has improved tool life. Oxide treatment changes TiC base to Ti-C-O (oxycarbide of Titanium). Oxycarbides have lower free energy than carbide and, therefore, are more stable. It is thought that the oxycarbide coatings on the bearing and journal surfaces may provide good friction and wear properties. Oxycarbide powders for plasma spraying technique may be made by coating TiC particles with TiO by a Metco technique. A sputtering target can also be made from the coated particles. During sputtering, a chemical reaction probably will take place and sputtered coating may be made of oxycarbides.

A list of recommended candidates for future work is given in Table VI.1.

TABLE VI.1

FUTURE DEVELOPMENT OF PROMISING CANDIDATES

Item No.	Coating Combinations	Present Efforts in Foil Bearings	Process Variables
1.	Kaman DES	Extensive	Vary thickness, composition or Processing parameters
2.	Tribaloy 400, CaF_2 and silver coating	Extensive	Vary composition and additives
3.	Sputtered Cr_2O_3 and Cr_3C_2	Moderate	Interlayers, overlayers and metallic binders in the coating
4.	Soft Lubricants (i) CdO and Graphite	Extensive	Variation in composition and continued testing at higher temperatures
	(ii) $PbO.SiO_2$	None	Evaluate standard composition for test application and add Ag to improve room temperature characteristics
	(iii) CaF_2-BaF_2 Eutectic	None	a. Evaluate CaF_2-BaF_2 and additions of Ag. b. Evaluate AFSL-28 coating
5.	Preoxidation	None	Evaluate preoxidized Inconel X-750 vs. itself.
6.	Titanium Oxycarbides (Ti-C-O)	None	Development and Use

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APPENDIX A

STRESSES IN THE TOP FOIL OF THE FOIL BEARING DUE TO DYNAMIC LOADING

In a hydrodynamic resilient bearing construction, the bearing is comprised of a smooth top foil and a "bump" or convoluted foil. The bump foil gives distributed elastic support to the top foil on which the bearing load is applied. For calculation purposes, as a first approximation, the top foil is considered to be a flat rectangular beam pinned at one end, free at other and simply supported in between (Figure A.1). Although the bumps provide elastic support yet there are assumed to be rigid for simplicity. The assumption will give a conservative estimate of the bending stress level in the top foil.

Clapeyron's three moment equation was used for solving this continuous beam problem, (see Reference 15). Figure A.2 shows the free body diagrams of two beam segments. From the continuous beam theory it follows:

$$M_A + 4\ M_B + M_C = -\ \omega \ell^2/_2 \tag{A.1}$$

$$M_i = M_n = 0 \tag{A.2}$$

Where,

M = moment
ω = weight per unit length
ℓ = distance between the supports

If n is an even number, due to symmetry

$$M_{(m+1)} = M_{(n-m)} \tag{A.3}$$

If n is an odd number, due to symmetry

$$M_{(m)} = M_{(n-m+1)} \tag{A.4}$$

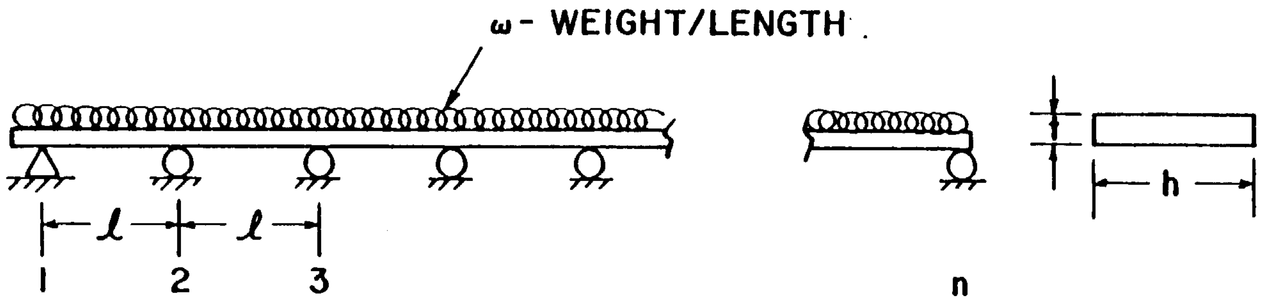

Fig. A.1 Idealized Schematic of Top Foil

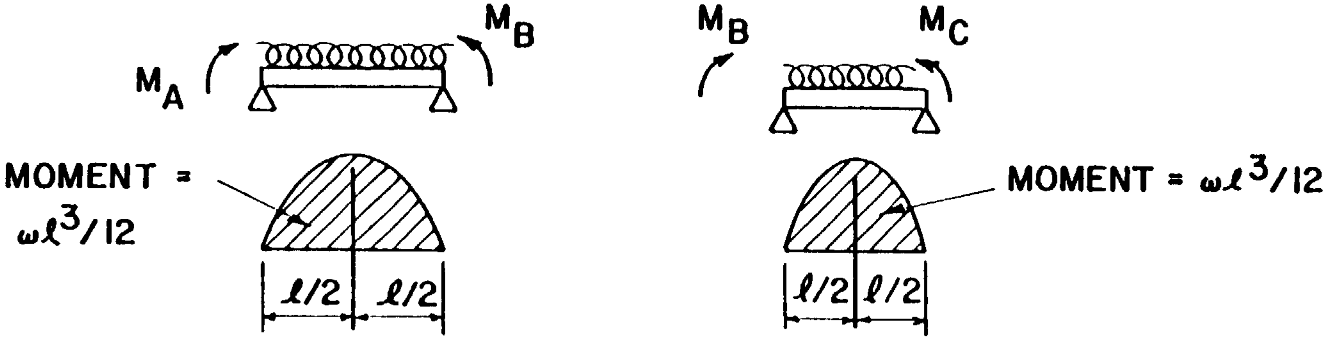

Fig. A.2 Free Body Diagrams of Beam Segments

The moment in any segment can be calculated using Equations (A.1) to (A.4). Then the stress, σ can be calculated by the following relation (Reference 15):

$$\sigma = \frac{M}{(ht^2/6)} \qquad \text{(A.5)}$$

Where,

σ = stress
h = width of the foil
t = thickness of the foil

Assuming that:

n = 9, ℓ = 4.5 mm (0.18 in.), t = 102 μm (0.004), and load = 210 k Pa (30 psi)

From Equations (A.1) and (A.4) it is shown that:

$$M_{max} = \frac{-15}{134} \omega \ell^2$$

$$\text{and } \sigma_{max} = \frac{-45}{67} \frac{\omega \ell^2}{ht^2}$$

$$= 281 \text{ M Pa } (40{,}800 \text{ psi})$$

APPENDIX B

FLASH INTERFACE TEMPERATURE CALCULATIONS

The flash (instantaneous) temperature rise at the interface depends on the mechanical, thermal, topographical, and the tribological properties of the mating surfaces and sliding conditions. From Bhushan and Cook [16, 17], the temperature rise for a square slider ($2\ell \times 2\ell$) rubbing on a semi-infinite solid in low speed sliding pertinent to this problem is given as:

$$\bar{\theta} = \frac{fV}{k_1 + k_2} \left[0.44\, H\, \bar{d}_{max} + \ell \bar{\sigma} \right] \qquad (B.1)$$

If L (Peclet Numer) $= \dfrac{V\bar{d}_{max}}{K} < 1$

where,

$\bar{\theta}$ = average flash temperature, °C(°F)

f = coefficient of friction

V = sliding speed, mm/s (in/s)

k_1, k_2 = thermal conductivity, W/(m.K) (lb/(sec°F)) of sliding members

K = thermal diffusivity, mm^2/s ($in.^2/sec$)

H = bulk hardness of the softer material, Pa (psi)

$\bar{\sigma}$ = mean contact stress, Pa (psi)

$\bar{d}_{max}$ = maximum value of junction diameter during the life of an asperity contact, mm(in)

ℓ = half length of square slider, mm(in)

From experience it is found that $\bar{d}_{max}$ for many metal to metal combination varies from 13 μm (5×10^{-4} in.) to 25 μm (10^{-3} in.).

To obtain an idea of the order of magnitude of the interface temperature, the case of A286 rubbing on Inconel X-750 is considered. Relevant thermal, mechanical and topographical properties needed are:

k_{A286} = 14.73 W/(m.K) (1.84 lb/(Sec. °F))

$k_{Inconel}$ = 12.09 W/(m.K) (1.51 lb/(sec. °F))

H_{A286} = 6.38 GPa (9.257×10^5 psi)

$H_{Inconel}$ = 3.48 GPa (4.98×10^5 psi)

$\ell \simeq$ 6.3 mm (.25 in.)

$\bar{d}_{max} \simeq$ 13 μm (5×10^{-4} in)

Substituting these values in Equation B.1 gives:

$$\bar{\theta} = fV\,[0.7 + 0.0002\bar{\sigma}]$$

where V is in mm/s and $\bar{\sigma}$ is in k Pa

$\simeq 0.7\ fV$ since in this case $\sigma \leq 210$ kPa.

Lift off speed in many application is 3000 to 6000 rpm depending on loading. Typically for 14 kPa (2 psi), the lift off speed is roughly 4000 rpm. For 38 mm (1.5 in) diameter bearing, the surface speed becomes

$V \simeq$ 7980 mm/s (314 in./sec)

At higher speeds (close to lift off speed), it is seen that the interface temperatures are pretty high and as a result there may be formation of protective oxide layer at the interface which may reduce the friction considerably. For demonstration purposes, it is assumed that

$f \simeq 0.05$

then

$$\bar{\theta} = 0.7 \times 0.05 \times 7980$$
$$= 279°\text{C } (534°\text{F})$$

APPENDIX C

PREPARATION OF HL-800 COATING

Introduction

Graphite was one of the first widely used inorganic solid lubricant. The slippery texture of the graphite is believed to be due to layer lattice type of crystal structure (a hexagonal crystal structure which shears readily in a direction parallel to the basal planes of the crystals). Adsorbed water vapor and oxygen are necessary for graphite in order to achieve desired low friction and wear.

The effectiveness of the graphite as a lubricant (reduction of its shear strength) is associated with the formation of adherent films on the lubricating surfaces. The presence of some oxides or salt may improve the adherence of the graphite films (References 5 and 8). Several reasons have been given for the improved adherence. Some researchers believe that it may be due to the formation of interstitial or intercalation compound by reaction of these materials with graphite. It is also suggested that such compounds would serve as bonding media for the graphite. Friction tests in the temperature range of room temperature to 540°C (1000°F) of powder of graphite and graphite mixed several soft metallic salts and oxides were conducted by Peterson and Johnson [8]. They found that the coefficient of friction of graphite in air is quite low at room temperature, but increases at temperatures above about 95°C (200°F). The friction coefficient again drops at 425°C (800°F) and graphite acts as an effective lubricant up to 540°C (1000°F). See Figure C.1. Several metallic compounds, PbO, CdO, sodium sulphate, and cadmium sulphate were mixed with graphite. It was concluded that cadmium oxide and graphite mixture lubricated most effectively in the entire temperature range up to 540°C (1000°F). Figure C.2 shows the friction data of 2/3 CdO and 1/3 graphite mixture. Graphite used in their test was a high purity electric-furnace synthetic graphite. Synthetic graphite was selected due to its higher temperature capability than natural graphite.

In the experiments of Peterson and Johnson, the graphite and cadmium oxide powder was added at the rubbing interface. The authors' knowledge, to date no coating of this mixture has been developed. It was believed that the

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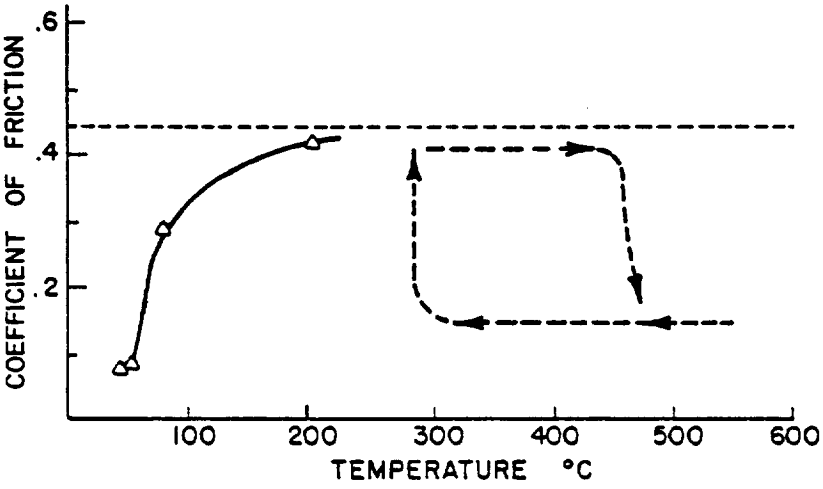

Fig. C.1 Lubrication with Graphite Alone, Sliding Velocity 29 mm/s Load 180 N.

Peterson & Johnson [8]

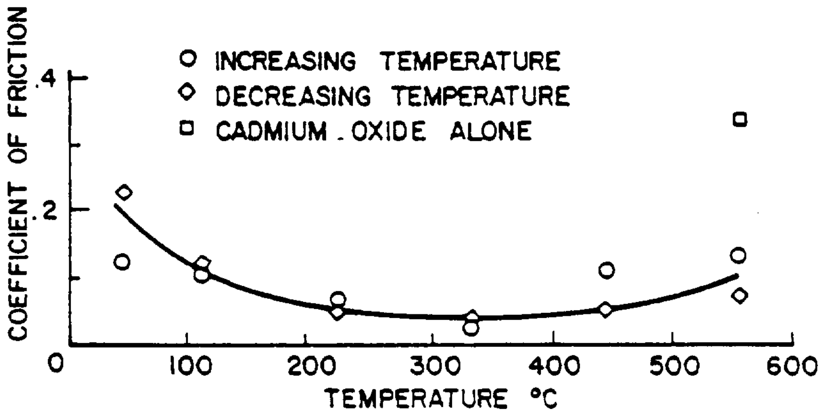

Fig. C.2 Lubrication with Cadmium Oxide - Graphite Mixture. Sliding Velocity 29 mm/s, Load 180 N.

Peterson and Johnson [8]

coating would reduce the oxidation of graphite compared to loose graphite powder at high temperatures (it has been later confirmed).

Preparation of the Coating

Past experience has shown that 3 parts graphite and 1 part cadmium oxide provide good lubricity in the bearings. This proportion was selected for our selection. Sodium sillicate has commonly been used as a binder in putting solid lubricant coatings. Many wetting agents (e.g. NPX made by Union Carbide) have been used to disperse the solution properly.

Graphite used was a 99.9 percent pure electric-furnace synthetic graphite made by Joseph Dixon, Crucible Co. It was very fine powder with 95 percent of the particle sizes finer than 325 mesh (95-325). Cadmium oxide was 99.9 percent pure (commercially pure) material with 95 percent of the particle sizes finer than 200 mesh. This powder was made by Materials Research Corp. Sodium sillicate (water glass), made by Philadelphia Quartz, was 99 percent pure with a composition of 8.9 percent Na_2O, 28.7 percent SiO_2 and balance water. Wetting agent used was Absol 895 with a cloud point of 65°C (for its effective use, the temperature of the solution should be 65°C).

Higher contents of sodium sillicate are not desirable as it is abrasive. By trial and error it was found that about 30 percent by weight (water content not included) of sodium sillicate gave adequate bond of the coating.

The mixture of CdO and graphite (1:3) was dissolved in distilled water. It was ball milled for roughly 4 hours. Just before spraying, sodium sillicate and one drop of Absol was added and it was stirred vigorously. The solution was heated to about 65°C (150°F) before spraying. It was sprayed by an air brush about 25 μm (1 mil) thick onto a carefully prepared substrate.

The sprayed coating was left at room temperature conditions for 30 minutes. It was then baked in an oven at 65°C (150°F) for two hours and then at 150°C (300°F) for 8 hours. The coating was burnished 8 to 10 μm (0.3 to 0.4 mil) thick.

APPENDIX D

THE EXAMINATIONS OF TEST SAMPLES BEFORE AND AFTER STATIC OVEN SCREENING

D-1 VISUAL INSPECTION

Table D.1 Examination of Coated A286 Coupons

Table D.2 Examination of Coated Inconel X-750 Coupons

TABLE D.1

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gms	Thickness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
1	TiC (S.)	Before	Steel Gray	Nothing came off	—	Could see substrate topography	—			One edge of coating was of the same color as A286 after oven (24). On other areas, coating was very thin and substrate could be seen.	II
		After	Substrate Color	Nothing came off	—	Looked the same except at one edge	—	.0276	—		
2	B_4C (S.)	Before	Dark Gray	Nothing came off	—	Could see surface topography of substrate	—			Coating darker. Bare metal not visible in microscope, after oven test. Quite spotty.	II
		After	Slightly darker	Nothing came off	—	Spotty look. Could not see bare metal.	—	.078	—		
3	CrB_2 (S.)	Before	Steel Gray	Nothing came off	—	Could see surface topography of substrate	—			Coating became darker. Oxidized. Can see substrate on half of the area.	II
		After	Black	Nothing came off	—	Darker substrate less visible	—	.0177	—		
4	CrB_2 (P.S.)	Before	Dark Gray	Very little came off	Pressure reqd to make scratch		73-74 (15-N)≡ 27 Rc			Looked the same after Oven Test.	I
		After	Nearly Black	Very little came off	Same	could not see because of poor reflection	75-80 (15-N)≡ 30-40 Rc	.0056	--		
5	Cr_2O_3 (P.S.)	Before	Grayish Black	Nothing came off			91-93 (15-N)≡ 62-65 Rc			One edge chipped off about 10 mm x 2 mm.	NO
		After	Black	Nothing came off		looked the same	90 (15-N)≡ 60 Rc	.0316	—		
6A	Borided A-286 (650°C)	Before	Silver Gray	Nothing came off	Scratch with a file - file slips as if on glass					Borided case layer spalled. It probably was very brittle.	NO
		After	Dark Gray	Nothing came off	made a scratch with file			-1.0133	—		

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TABLE D.1 (Cont'd)

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gms	Thick-ness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
6B	Borided A286 (540°C)	Before	Silvery Gray	Nothing comes out	Scratch with a file - file slips as if on glass	—	—			Surface layer completely loose. Diffused layer extremely brittle and could not tolerate high temperature soak and cycling.	NO
		After	Black	—	Made a scratch with file			—	—		
7	Tribaloy 800 (P.S.)	Before	Gray	Nothing came off			79-86 (15 N) ≡ 38-52 RC			Top of coating oxidized; Polishing the top, shows base coating. Scratching had no effect.	I
		After	Greenish Black	Very little came off	Same	Could not see coupons because of color	90-92 (15 N) ≡ 61 Rc	.114	—		
8	Metco Cr_3C_2 (P.S.)	Before	Greenish Gray	Nothing came off			88-89 (15 N) ≡ 57 RC			Coating unchanged in appearance and hardness.	II
		After	Almost Black	Nothing came off	Same	Could not see due to poor reflection	87-90 (15 N) ≡ 54-60 RC	.0508	—		
9	Linde Cr_3C_2 (D.G)	Before	Gray	Nothing came off	—		90-91 (15 N) ≡ 61 Rc			Metco and Linde look good. We selected Linde as this is done by detonation gun. Coatings by det. gun have better bond and are denser.	I
		After	Grayish Black	Nothing came off	—	Could not see due to poor reflection	91-92 (15 N) ≡ 63 Rc	.0806			
10	Cr_2O_3 (S.)	Before	Almost Black	Nothing came off	—	Could see bare surface	—			No change in appearance	I
		After	Darker	Nothing came off	—	Looked about the same	—	.0152	—		
11	WC (S.)	Before	Gray	Nothing came off	Could see bare metal texture		—			Changed color to yellow - oxidized to tungsten oxide. Came off by tape test.	NO
		After	Yellow	Most of the coating came off	Looked smooth		—	.0088	—		

TABLE D.1 (Cont'd)

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gms	Thickness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
12A	NASA PS 101 (PS)	Before	White with Greenish spots	Nothing came off						The color of spalled coating had red tint. Coating spalled at one edge during thermal cycling. Could be due to mismatch in thermal expansion due to glass in the coating.	NO
		After	White with Greenish Spots Reddish Tint where Coating Spalled	Nothing came off	Needed same force to make scratch	Looked the same		.3504	0.05		
12B	NASA PS 106 (PS)	Before	White with Greenish spots	Nothing came off			75-78 (15-N) ≡31-36 Rc			Looked the same. No Effect.	I
		After	White with Greenish spots	Nothing came off	Needed same force to scratch	Looked the same	76-77 (15-N) ≡33-34 Rc	.3345	0.1		
13	Cr_3C_2 (S.)	Before	Gray	Nothing came off	Smooth scratch					Coating lost at places.	NO
		After	Bluish Gray	Nothing came off	Smooth scratch needed same force	Could see topography of substrate	—	.0058	—		
14	Electrolyzed A286 (Cr plating)	Before	Shiny white	Nothing came off			76 (15-N) ≡ 32 Rc			Lost all the coating. Could see undercoating at places.	NO
		After	Greenish, Copper color	Nothing came off		Could not see coating due to poor reflection	75-76 (15-N)≡ 31 Rc	.042	—		
15	Silicon Nitride (sputtered)	Before	Dark Gray	Nothing came off	—	Could see surface topography	—			Most of the coating disappeared. Some parts were burnt away. Coating left on one edge.	NO
		After	Bare substrate + black	Nothing came off	—	Bare metal could be seen at places	—	.0488	—		
16	Al_2O_3 (S.)	Before	Green and Red	Nothing came off	—	Could see surface topography	—			Coating looked intact. It was felt that there was not enough coating.	II
		After	Similar but darker	Nothing came off	—	Looked the same	—	.0115			

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TABLE D.1 (Cont'd)

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gms	Thickness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
17	Al_2O_3 (P.S.)	Before	White	Nothing came off			88-91 (15-N)≡ 56-62 Rc			Cracking and flaking occurred around edges during thermal cycling. Was intact before cycling.	NO
		After	White	Nothing came off	Needed same force to scratch where the coating is left.	Looked the same	85-90 (15-N) ≡ 50-60 Rc	-.0891			
18A	CdO + Graphite F.C. (540°C)	Before	Black	Nothing came off	—					Some portions were greyish black-where some graphite is left. Other places graphite was gone. CdO could be seen in streaks. Lost about 30% of coating by weight.	II
		After	Gray with streaks of reddish brown	Nothing came off	—			-.0077			
18B	CdO + Graphite (F.C.) (650°C)	Before	Black	Nothing came off	—		—	—		Looked ok. Lost some graphite.	NO
		After	Nearly Black	Nothing came off	Needed same force to make scratch		—	.0153			
19	Ni-Co Electro Plated	Before	Silvery Gray	Nothing came off						Chipped off at edges and at other places. Changed color.	NO
		After	Black	Nothing came off	Very easy to make a scratch			.0126	—		
20A	Nitrided A286 (650°C)	Before	Gray	Nothing came off	Could not make scratch with the file	—	82 (15-N)≡ 45 Rc			Compound zone spalled off. Hardness probably retained due to diffused layer. Compound zone (top surface) pretty brittle.	NO
		After	Gray	Coating was loose	Pretty hard to make a scratch. Lost little strength	—	82-85 (15 N)≡ 45-50 Rc	-.5	—		
20B	Nitrided (540°C)	Before	Gray	Nothing came off						Coating chipped off during thermal cycling. Compound layer is brittle and flakes during cycling.	NO
		After	Black	Coating was loose				-.121			

TABLE D.1 (Cont'd)

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gm	Thickness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
20C	Tufftrided A286 (650°C)	Before	Black	Nothing came off	File test - can't make a scratch	—	81-83 (15 N)≅ 42-46 Rc			Compound zone spalled.	NO
		After	Black	Coating was loose	Pretty hard to make a scratch	—	94 (15N)≅ 65 Rc	-.0692	—		
20D	Tufftrided (540°C)	Before	Black							Coating chipped off during thermal cycling. Top layer probably was too brittle.	NO
		After	Black					-.0595			
21	NASA PS 100 P.S.	Before	Silvery Gray	Nothing came off	—		—			Coating completely loose and peeled off. Top of the coating looked about the same except greenish on the edges. Heavy oxide (black)is seen at the interface.	NO
		After	Greenish on edges	—	—		—	-.4369			
22	SiC in electrodeless nickel	Before	Gray	Nothing came off						Lost coating at all places. Coating was loose.	NO
		After	Nearly Black	Some came off	Loose - easily made scratch	Spotty		.0077	—		
23	Kaman DES	Before	Grayish Black	Nothing came off		Could see substrate topography	—	—		Looked the same.	I
		After	Grayish Black	Nothing came off	Needed some force to make scratch	Looked the same	—	—	—		
24	Uncoated Coupons (650°C)	Before	A286				29-30 Rc				N/A
		After	Darker	Nothing came off	Easier to put the scratch		30 Rc	0	—		

TABLE D.1 (Cont'd)

EXAMINATION OF COATED A286 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Surface Adhesion Test by Scotch Tape	Scriber Scratch Test Under Microscope	Microscope Examination (X400 max)	Coating Hardness	Wt Gain Gms	Thickness Gain mm	Comments (After Oven Testing)	Selected for Start-Stop Test Priority
25	Kokide C Flame Spray (658°C)	Before	Black	Nothing came off		Coating was rough				Most of the coating came off during soak temperature It came much more rapidly during thermal cycling.	NO
		After	Black	Nothing came off where coating is left		Same wherever coating is left		-1.43			
26	Mag. Zirconate (P.S.)	Before	White	Nothing came off		Fairly smooth				Coating looked the same. Coating color turned yellowish. No cracking and peeling.	YES for future tests
		After	Yellowish	Nothing came off	Took the same force to make a scratch	No cracking and peeling		.046			
27	Calcium Zirconate (P.S.)	Before	White	Nothing came off		Fairly smooth				Edges of the coupon turned more yellowish than rest of it. Coating looked the same. No cracking or peeling.	YES for future tests.
		After	Yellowish	Nothing came off	Took the same force to make a scratch	No cracking and peeling		.0412			
28	Linde L 103 (D.G.)	Before	Black	Nothing came off		Rough Well bonded				Coating looked the same. Green spots disappeared during oven test. No cracking or peeling.	YES for future tests
		After	Black	Nothing came off	Took same force to make a scratch.	Looked the same		.0405	—		

S. - Sputtered
P.S. - Plasma Sprayed
D.G. - Detonation Gun
F.C. - Fused Coating

TABLE D.2

EXAMINATION OF INCONEL-X750 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Flex Test 2×10^5 Cycles Total Flex 0.14 mm	Surface Adhesion Test by Scotch Tape	Scratch Test Under Microscope	Microscope Examination (X400 max)	Wt Gain Gms	Thickness Gain mm	Comments	Selected for Start-Stop Test Priority
A	B_4C (S.)	Before	Dark Gray	OK	Nothing came off	—	Could see substrate topography			The surface had irridescondence of Green and Red with Blue spot. Coating oxidized	II
		After	Irrideu-caudence of Green, Red, Blue		Nothing came off	—	Dull, spotty	-.0029	—		
B	TiC (S.)	Before	Steel Gray	OK	Nothing came off	—	Could see surface topography of substrate			Looked about the same except slight change in color.	I
		After	Brown-Greenish		Nothing came off	—	Looked the same	-.0043	—		
C	CrB_2 (S.)	Before	Steel Gray	OK	Nothing came off	—	Could see topography of base metal			Coating oxidized. Darker in color and had some spots at places. Coating probably missing at places.	II
		After	Dark Gray		Nothing came off	—	Looked spotty darker. Some places could see substrate	.0011	—		
D	TiB_2 (S.)	Before	Silvery Gray	OK	Nothing came off					Coating changed color to yellow. It flaked off. Probably TiB_2 oxidized to TiO_2 as TiO_2's characteristic color is golden yellow.	NO
		After	Golden Yellow	—	Most of coating came off	Easier to scratch	Could see some structure		—		
E	Ag (S.)	Before	White	OK	Nothing came off	—	Nice and shiny			The surface formed a loose dusty film (brown in color). One end was dark gray. Substrate oxidized and pushed the coating. (substrate was not oxidation resistant)	NO
		After	Brown Gray		Brown film came off	—	Dark film	.0078	—		
F	Cr_2O_3 (S.)	Before	Nearly Black	OK	Nothing came off	—	Could see substrate topography			Smooth. Coating had same appearance and color. Coating was intact.	I
		After	Nearly Black		Nothing came off	—	Looked same	.0004	—		

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TABLE D.2 (Cont'd)

EXAMINATION OF INCONEL-X750 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Flex Test 2 x 10^5 Cycles Total Flex 0.14 mm	Surface Adhesion Test by Scotch Tape	Scratch Test Under Microscope	Microscope Examination (X400 max)	Wt Gain Gms	Thickness Gain mm	Comments	Selected for Start-Stop Test Priority
G	Al_2O_3 (S.)	Before	Irridescendence of Green and Red	OK	Nothing came off	—	Could see the topography of foil			Coating looked extremely thin to start with. Vender probably did not provide desired thickness. There was little difference in appearance.	II
		After	Slightly darker		Nothing came off		Could not see topography of surface.	.0008	—		
H	WC (S.)	Before	Gray	OK	Nothing came off	Made a smooth scratch	Could see texture of substrate			WC oxidized, coating came off by tape. At edges yellowish coating was thicker.	NO
		After	Yellowish		Nothing came off	Scratched easier and coating seemed brittle	Could see texture of substrate	.0053	—		
I	MLF-5 (F.C.)	Before	Grayish Black	OK	Nothing came off					Coating became loose and chipped off. Whatever coating was left - had whitish color. Wherever coating came off - yellowish color.	NO
		After	Whitish of coating left Yellowish where coating came off.		Nothing came off			-.084	—		
J	Silicon Nitride (S.)	Before	Dark Gray	OK	Nothing came off	—	Could see substrate topography			It looked good in the center. At one edge there were some dark patches.	I
		After	Black on edges		Nothing came off	—	Center the same. Edges burnt and have dark film	.0001	—		
K	AFSL -28 (F.C.)	Before	Dark Gray	OK	Nothing came off					Coating changed color from dark gray to light greenish Coating chipped off and the base metal curled up.	NO
		After	Light Green		Coating was loose			.0876	—		
L	Cr_3C_2 (S.)	Before	Silvery Gray	OK	Nothing came off	Made a smooth scratch	Could see bare metal			At one edge it is dark in color like substrate, the area next to it is purple then yellow. We lost coating on one and the remaining coating reacted.	NO
		After	Yellowish at one edge dark like substrate		Nothing came off	Made a smooth scratch		-.0003	—		

TABLE D.2 (Cont'd)

EXAMINATION OF INCONEL-X750 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Flex Test 2×10^5 Cycles Total Flex 0.14 mm	Surface Adhesion Test by Scotch Tape	Scratch Test Under Microscope	Microscope Examination (X400 max)	Wt Gain Gms	Thickness Gain mm	Comments	Selected for Start-Stop Test Priority
M 1	CdO + Graphite (F.C.) (540°C)	Before	Black	OK	Very little came off looked OK	Smooth scratch				Lost some graphite. Graphite oxidized.	I
		After	Yellowish Gray & Blackish Gray		Nothing came off	Smooth scratch		—	—		
M 2	CdO + Graphite (F.C.) (650°C)	Before	Black	OK	Nothing came off	—	—			Lost lot of the graphite	NO
		After	Yellowish Black	—	Nothing came off	—		—	—		
M 3	CdO + Graphite (F.C.) Vapor honed (540°C)	Before	Black	OK	Nothing came off	—	—			Vapor honed foil retained most of the graphite. The coating looked good.	I
		After	Nearly Black	—	Nothing came off	—	—				
N	Kaman DES (C.A.)	Before	Grayish Black	OK	Nothing came off		Could see substrate topography			Looked the same.	I
		After	Grayish Black	—	Nothing came off	Needed same force to make scratch	Could see substrate topography	—	—		
O	Uncoated Foil	Before	Shiny	—	—	—				Looked the same except duller.	I
		After	Duller	—	—	—	One portion darker in color	.0017	—		
P	TiC (S.) + Ag (S.)	Before	Silver	OK	Nothing came off	—	Could see surface topography			Silver disappeared from the edges, some remaining in the center.	NO
		After	Dull Color		Nothing came off	Same		.0014	0.015		

TABLE D.2 (Cont'd)

EXAMINATION OF INCONEL-X750 COUPONS - OVEN SCREENING TESTS

No.	Coating	Before/ After Oven Test	Color	Flex Test 2×10^5 Cycles Total Flex 0.14 mm	Surface Adhesion Test by Scotch Tape	Scratch Test Under Microscope	Microscope Examination (X400 max)	Wt Gain Gms	Thickness Gain mm	Comments	Selected for Start-Stop Test Priority
Q	TiC (S.) + Au (S.) overlay	Before	Gold	OK	Nothing came off					Gold disappeared slightly at places. Loose at places	NO
		After	Dull Gold	OK	Gold came off			.0046	0.008		
R	Cr_2O_3 (S.) + Ag (S.) overlay	Before	Silver	OK	Nothing came off					Some areas were of same color as Cr_2O_3, remaining had some silver left.	NO
		After	Black, dull Silver	OK	Nothing came off			.0002	0.008		
S	Cr_2O_3 (S.) + Au (S.) overlay	Before	Gold	OK	Nothing came off					Gold came off at most of the places.	NO
		After	Black & Gold	OK	Nothing came off			.0021	0.006		

S. - Sputtered
F.C. - Fused Coating
C.A. - Chemical Adherent Coating

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D-2 DISCUSSION OF METALLURGICAL EXAMINATIONS OF COATED A286 AND INCONEL X-750 COUPONS

Scanning Electron Microscope and EDAX Studies

Samples selected for scanning electron microscope (SEM) and X-Rays Energy Dispersive Analyzer (X-REDA) studies were:

TiC sputtered coating on foil
Si_3N_4 sputtered coating on foil
HL-800 fused coating on foil
Cr_2O_3 sputtered coating on foil
Kaman DES coating on foil
Tribaloy 800 plasma sprayed coating on journal
Heat treated and uncoated Inconel X-750 coupon.

Coupons of the above coatings were examined as received and after oven test.

Figure D.1 shows the surface morphology of a heat heated Inconel X-750 coupon (uncoated) for later comparisons. Figure D.2 shows the surface appearance of sputtered TiC coating (as received). In comparing Figure D.2 with Figure D.1, it was believed that the coating followed the topography of the substrate. The coating was believed to be dense and did not have any cracks. There were many particles on the surface. Examination of the surface showed that there were no foreign particles present on the coating. The elemental analysis (X-REDA) of the particles and rest of the coating showed that all of the coating is made of Titanium carbide.

Figure D.3 shows the surface appearance of the sputtered TiC coating after exposed to high temperature. The scratch in Figure D.3(a) was probably done in handling the coupon. The particle density on this coupon was higher and their size was statistically larger than before oven coupon. It was again believed that the particles were an integral part of the coating. X-REDA analysis of the particles and rest of the coating showed that the coating was composed of Titanium carbide and the substrate underneath has elements expected in Inconel (primary elements Ni, Cr, Fe and Ti). The analysis of the particles and the substrate underneath showed that they had elements such as Al, Si, S, Fe, P, and very little Ti and Cr. Some particles had considerable Ni and others had very little. From this observation, it was postulated that microspots

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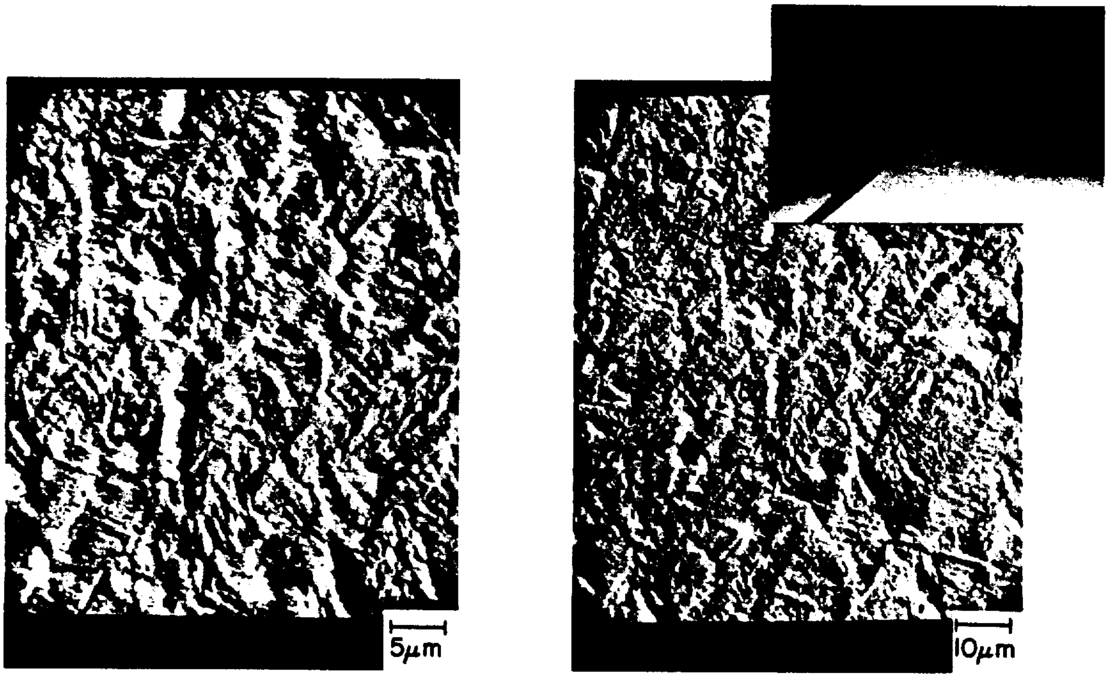
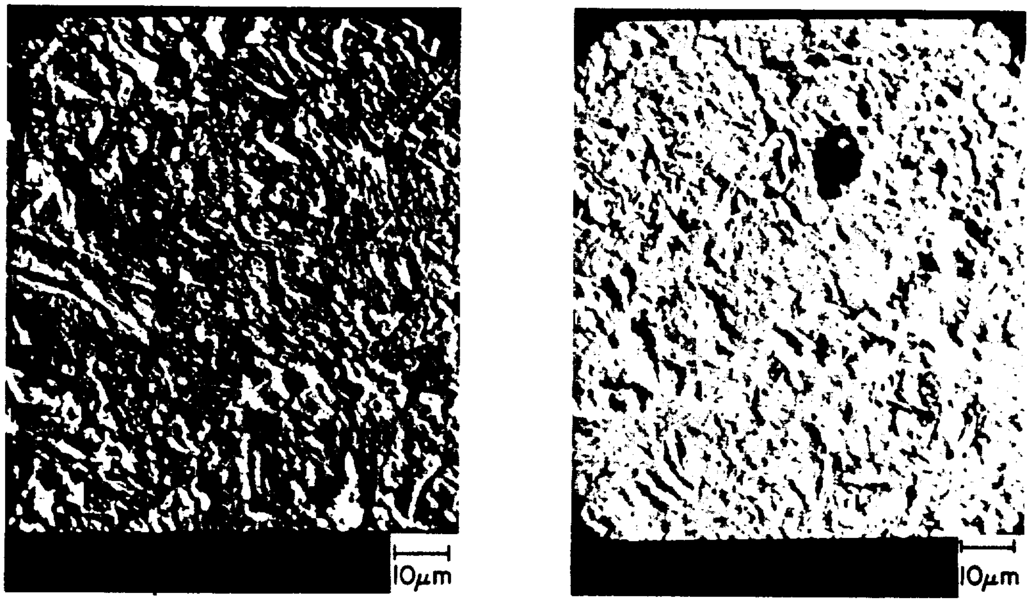


(a) (b)

Fig. D.1 Typical SEM Micrographs and Diffractogram of Heat Treated Inconel X-750



(a) (b)

Fig. D.2 Typical SEM Micrographs of Sputtered TiC on Inconel Base (as received)

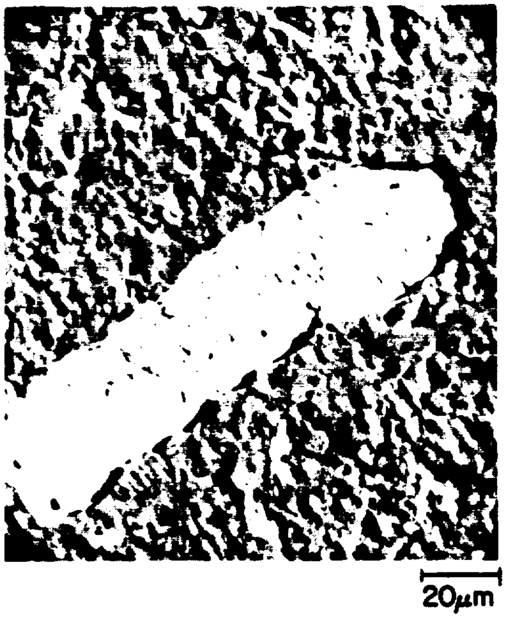
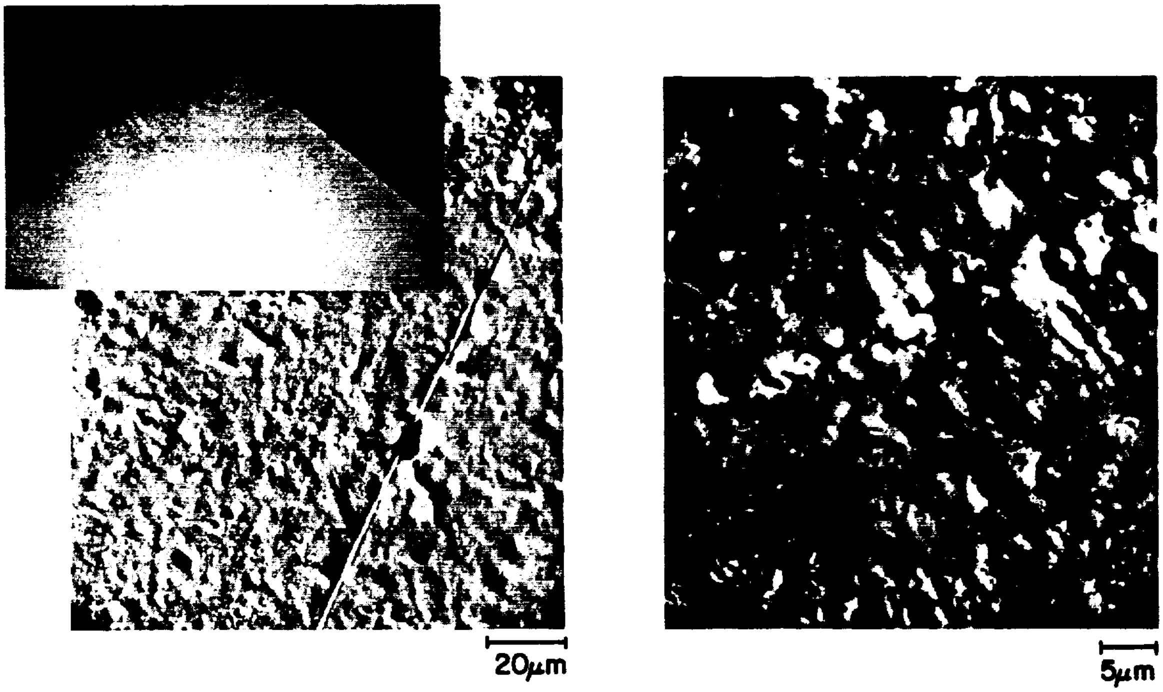


Fig. D.3 Typical SEM Micrograph of Sputtered TiC (as received) with a Man Made Scratch



(a) (b)

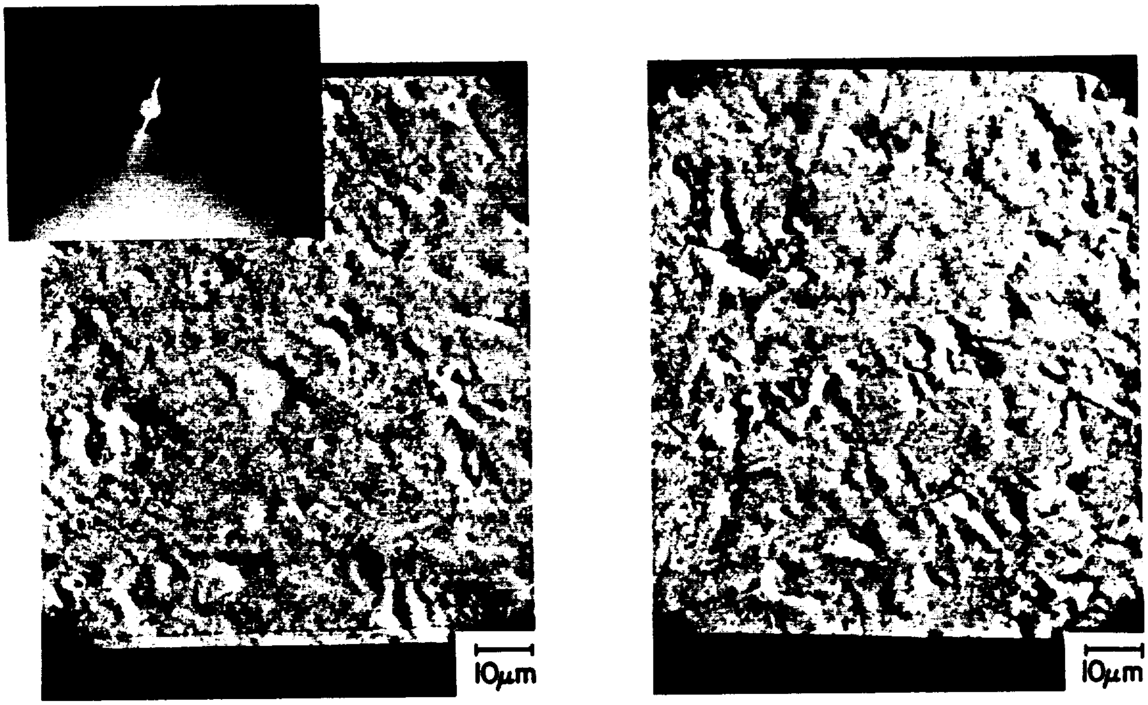
Fig. D.4 Typical SEM Micrographs and Diffractogram of Sputtered TiC on Inconel Base (After Oven) of the substrate with inclusions and little Cr content oxidized and pushed the coating away. It is believed tentatively that TiC coating doesn't provide enough protection to avoid the local oxidation.

Scratch test was performed on the TiC coating after oven exposure to evaluate its ductility. In this test a scratch was made with a scriber and it was examined for any branch cracking. From examination of the scratch (in Figure D.4) it was apparent that no branch cracks were formed which suggests that the coating was ductile. At this time, it was felt that scratching may push the debris onto the surface and may lead to misinterpretation of the surface texture. Therefore, no more scratches were made on other coatings.

Figure D.5 shows the surface appearance of the Si_3N_4 sputtered coating. The coating appeared to be dense and had no cracks. The valleys on the surface came from the substrate topography (Figure D.1). Portion of Si_3N_4 coating was oxidized during oven test. Figure D.6 shows non-oxidized and oxidized area of the coating after oven test. The coating after oven test looked about the same as before exposure. Oxide layer on the coating after oven test formed no shadow in tilting the specimen with respect to light in SEM study which implies that it had practically no thickness. The layer could be easily scrapped off and Si_3N_4 color appeared underneath. X-REDA analysis confirmed that the coating was composed of Si compounds.

Figure D.7 shows the surface appearance of Cr_2O_3, sputtered coating on Inconel base. Comparisons, with uncoated Inconel X750 surface (Figure D.1), show that the coating follows the surface texture. The photograph taken at a magnification of 10,000 X, to study the surface morphology (Figure D.7b), shows that the coating has nodular growth. Nodules grow within columnar structure and project to the surface with domes. The structure looks like peelings of an orange or a cauliflower. Similar results have been reported by Spalvins [18,19]. Spalvins postulated that the nodules grow from the nucleation sites present on the substrate. He concluded that the nodules have undesirable effects on mechanical properties; cracks are initiated at the nodules when the coating is stressed by mechanical forces. It is felt that evidence of such growth is to be expected at higher magnifications (X10,000) although further tests should be carried out to establish if the

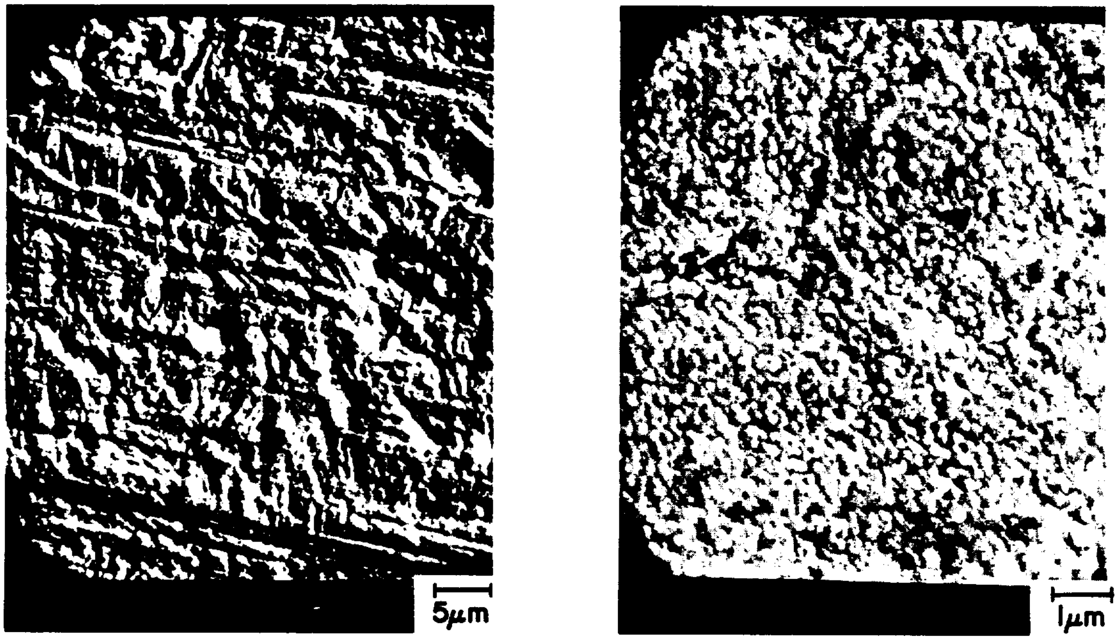
Fig. D.5 SEM Micrograph of Sputtered Si_3N_4 on Inconel Base (as received)



(a)

(b)

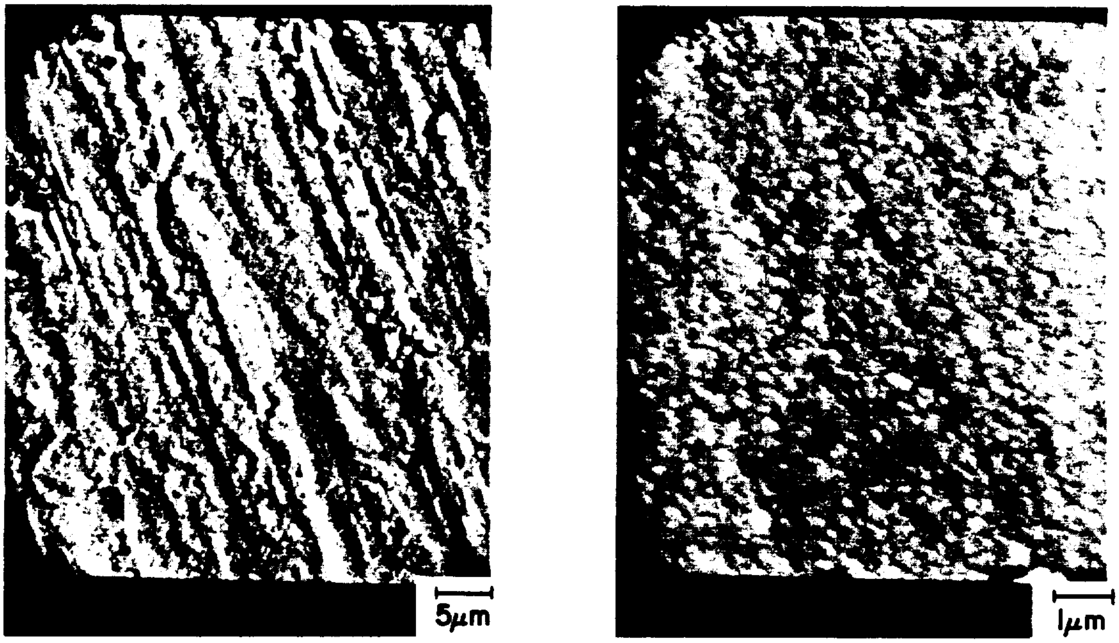
Fig. D.6 SEM Micrographs and Diffractogram of Sputtered Si_3N_4 on Inconel Base (after oven)



(a)

(b)

Fig. D.7 Typical SEM Micrographs of Sputtered Cr_2O_3 on Inconel Base (as received)



(a)

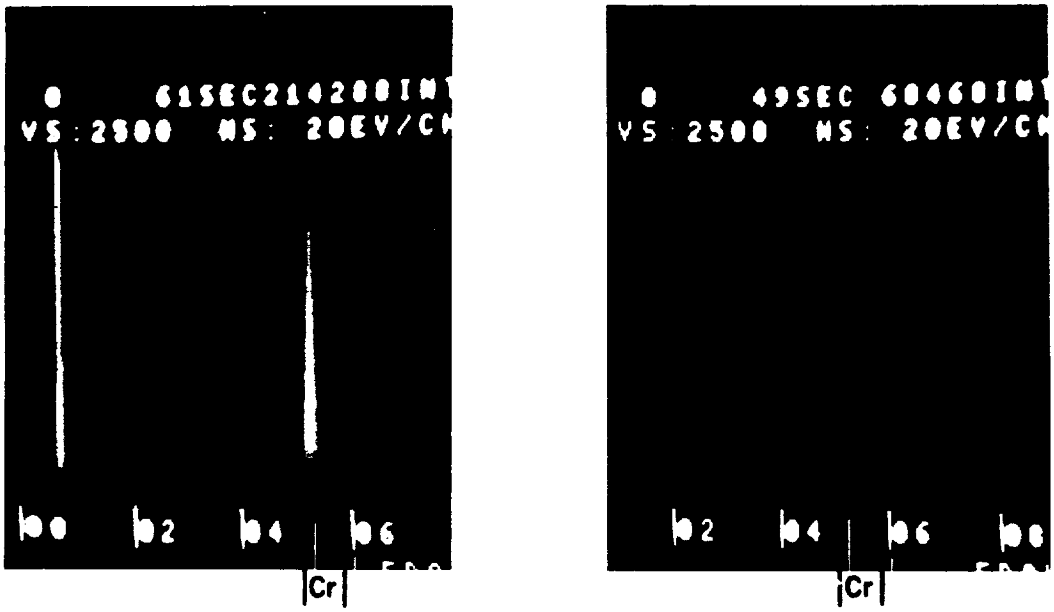
(b)

Fig. D.8 Typical SEM Micrographs of Sputtered Cr_2O_3 on Inconel Base (after oven) nodular growth is indeed undesirable. X-REDA analysis of the coating reveals that the surface has primarily Ni, Cr, Fe, Ti and trace amounts of W, Cℓ, S and Aℓ. Cr content is about two-third that of Ni. This shows that the coating is made of Cr compound (Cr_2O_3), which is what it should be.

Figure D.8 shows the surface appearance of Cr_2O_3 coating after it has been exposed to high temperature. The coating looked similar to "as sputtered" (before oven) coating except it had many small particles. The X-REDA of the coating showed that chromium was of somewhat higher intensity after oven treatment as might be expected. (Chrome oxide concentrates on Inconel X750 surfaces during air oxidation). The other constituents and their quantities are similar. The composition of the fine particles was the same as that of the rest of the coating (Figure D.9).

Figure D.11 shows the surface appearance of Kaman DES chemically adherant coating on the Inconel X750 base prior to oven testing. The coating looked rougher and had lots of particles. X-REDA analysis showed that coating had strong chromium peak and weak peaks of Ni and trace amounts of Fe and Ti. The Cr peak is about 3 times that of Ni, which shows that the coating is a compound of Cr (Cr_2O_3) and is quite thick since elemental analysis did not pick-up the elements of substrate as much as in the case of sputtered coatings. Figure D.12 shows the surface appearance of the Kaman DES coating after oven exposure. The coating did not seem to have as many particles but had more cavities. Possibly some of the particles came off and formed the cavities. X-REDA analysis of the coating showed that chromium and nickel peaks increased in intensity after heat treatment and additional weak titanium and iron peaks appeared (Figure D.10). It is suspected that Ti, Fe and enhanced Cr and Ni were all present as oxides and were caused by solid state diffusion of these elements from the Inconel foil into the coating during the oven tests.

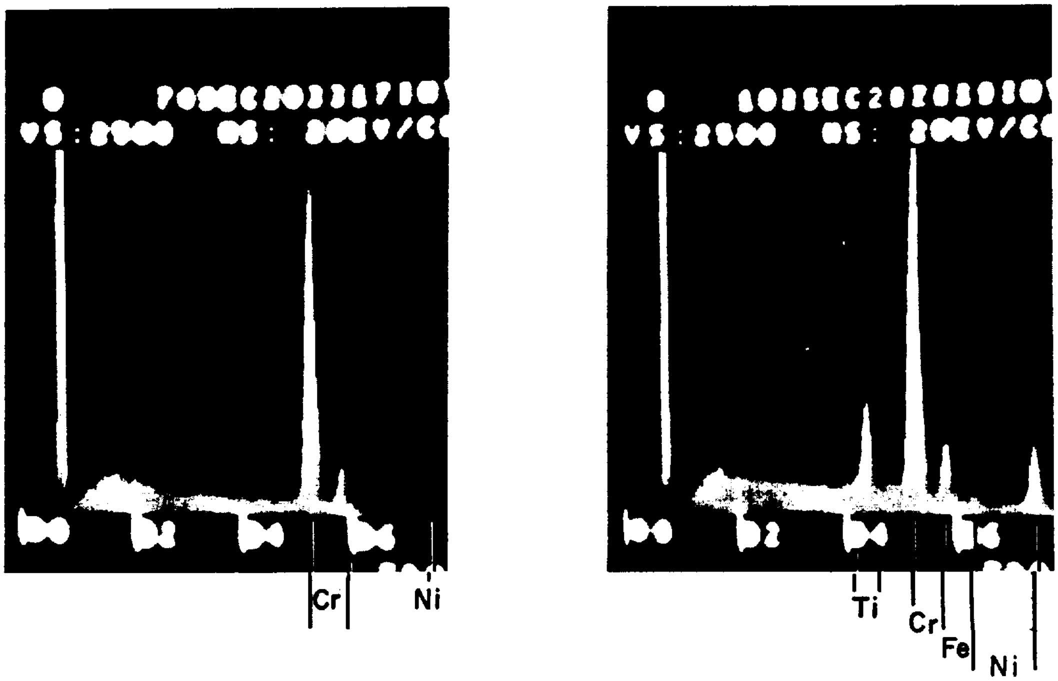
Figure D.13 shows the surface appearance of the HL-800 coating. X-REDA analysis showed that small particles were CdO coated with sillicate. CdO particles were quite randomly mixed with graphite and were bonded with sillicate.



AS RECEIVED

AFTER OVEN

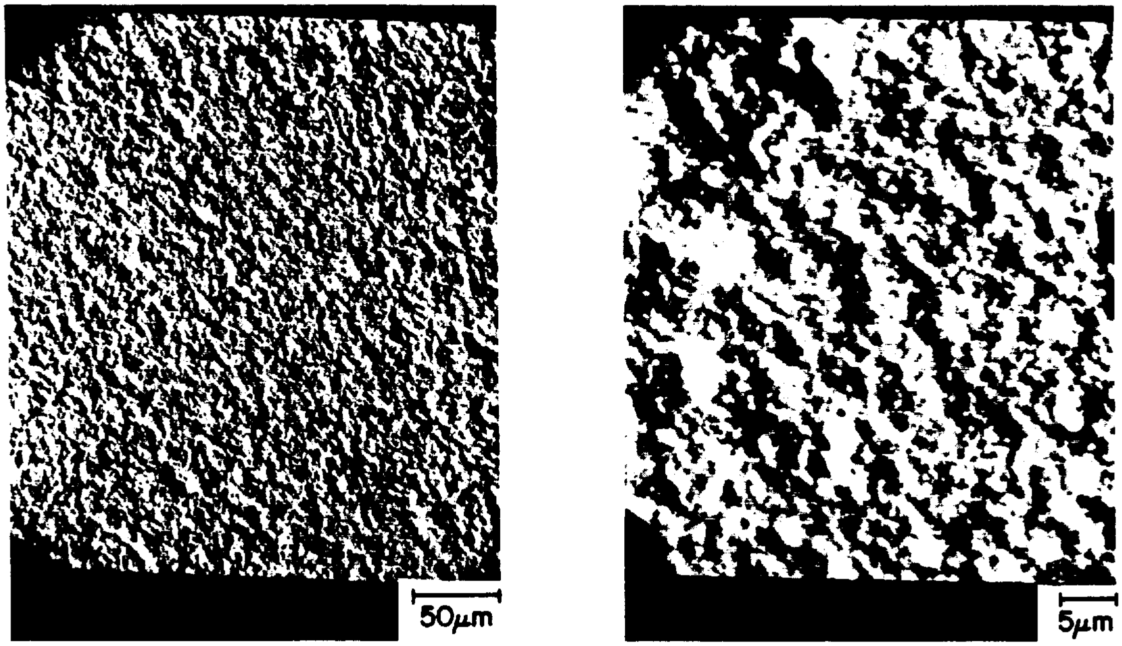
Fig. D.9 EDAX Image of Cr_2O_3 (sputtered)



AS RECEIVED

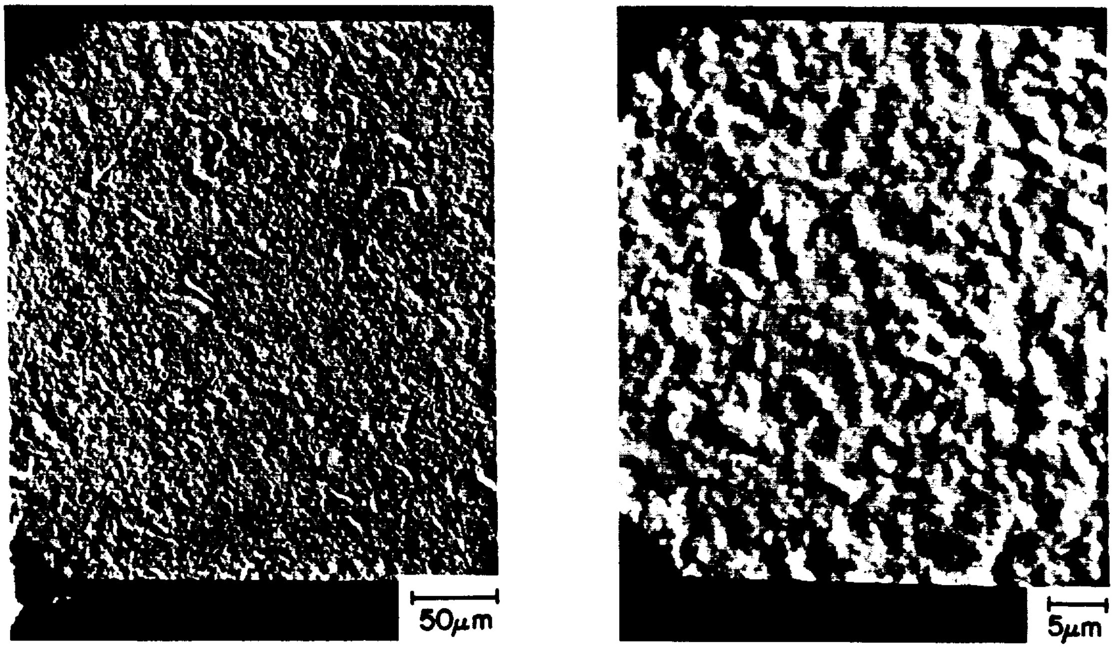
AFTER OVEN

Fig. D.10 EDAX Image of Kaman DES



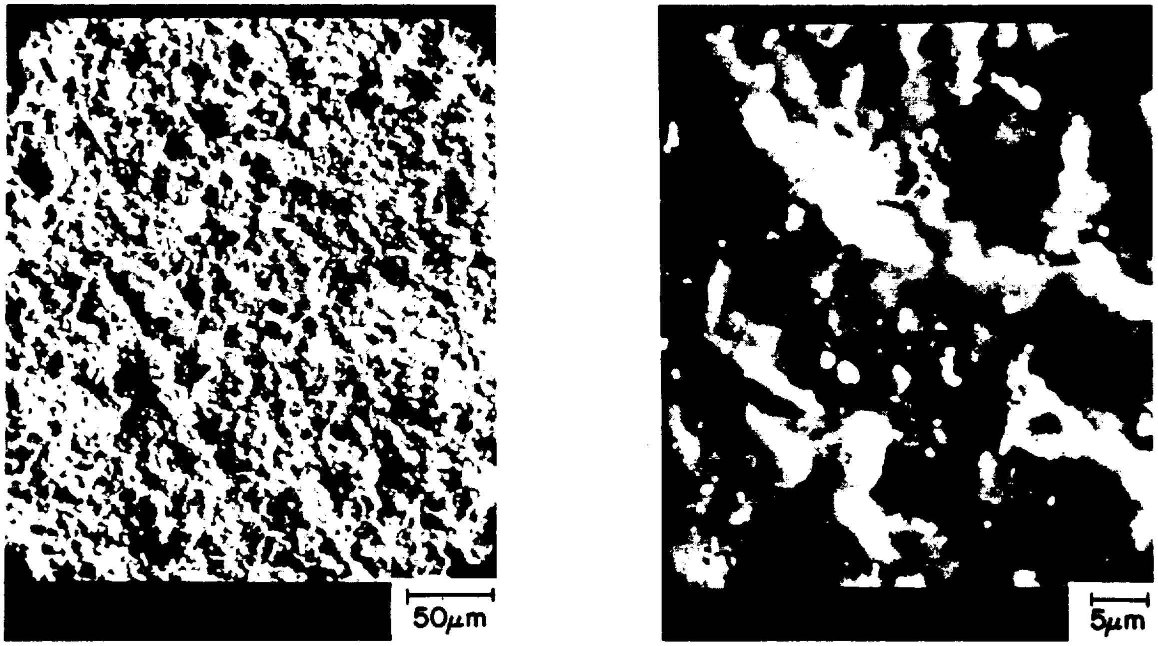
(a) (b)

Fig. D.11 Typical SEM Micrographs of Kaman DES on Inconel Base (as received)



(a) (b)

Fig. D.12 Typical SEM Micrographs of Kaman DES on Inconel Base (after oven)



(a) (b)

Fig. D.13 Typical SEM Micrographs of HL-800 on Inconel Base (as received)

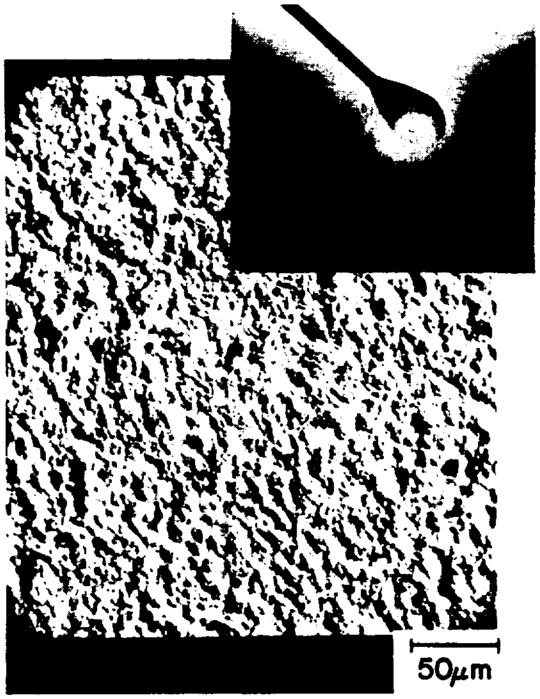


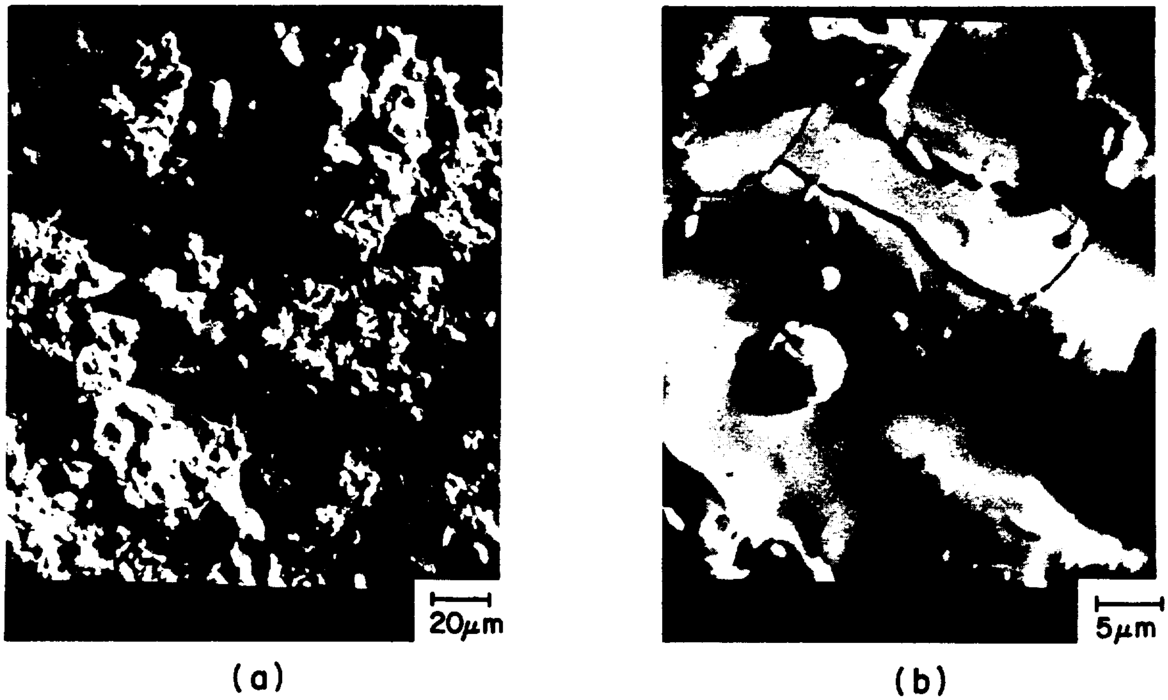
Fig. D.14 Typical SEM Micrograph and Diffractogram of HL-800 on Inconel Base (after oven)

Figure D.14 shows the coating after oven test. Elemental analysis and visual examination of the coating showed the composition of the coating was the same as that of the coating before oven tests except that the graphite content is less. The coating looked intact. CdO particles were not as prominent. They may have reacted with other components in the coating.

Figure D.15 shows the general appearance of Tribaloy 800 plasma sprayed coating on journal (A286 base material). The coating is as sprayed but not ground. There are many intergranular cracks on the surface (Figure D.15b). The cracks may have been formed by cooling of the hot particles coming in contact of the cooler substrate (typically 150°C) during plasma spraying. To establish if these cracks were only on the surface or throughout the coating thickness, the coating was ground and then examined for cracks (See Figure D.16). It was found that the cracks were only on the surface and not through the coating. Since the coating is ground before usage, they should present no problem. The pits and voids on the surface indicated that the coating was quite porous. Figure D.17 shows the surface appearance of the "as sprayed" Tribaloy 800 coating after the oven test. No apparent cracks were observed in this coating. It is believed that the cracks may be obscured by oxidation of the surface during oven test.

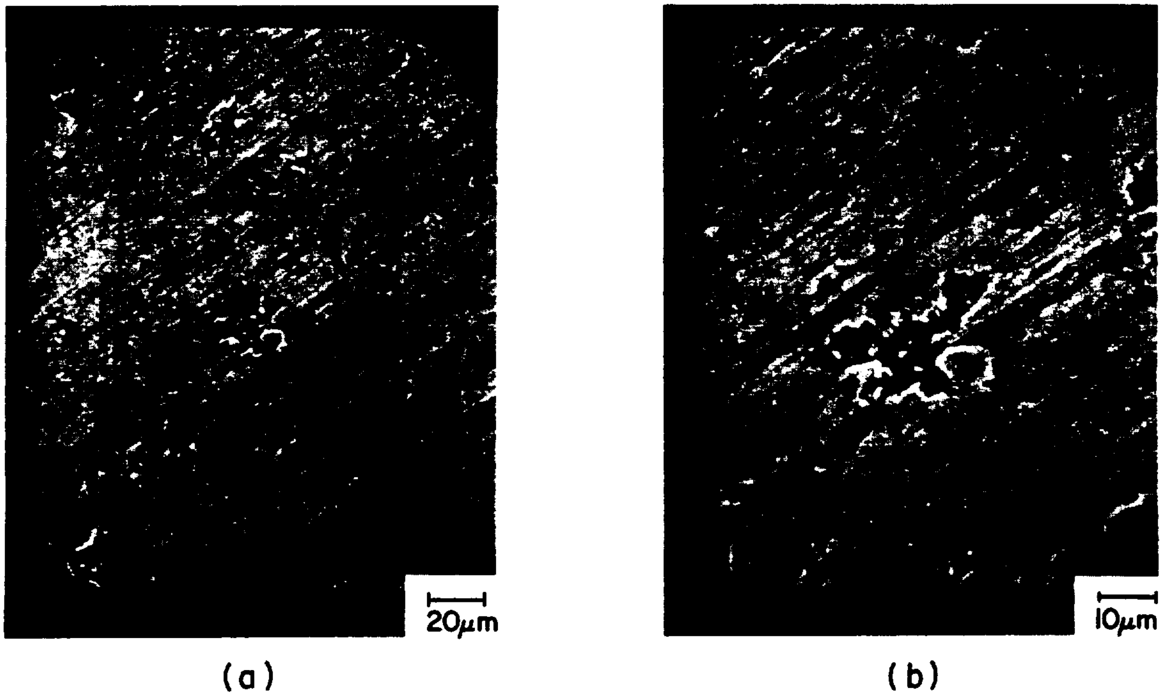
Reflection Electron Diffraction and X-ray Diffraction Studies

The composition and properties of sputtered coatings depend on sputtering parameters. Reflection electron diffraction analysis of some of the promising coatings was carried out to evaluate their chemical composition. In case of sputtered $A\ell_2 O_3$, it was suspected that the vendor had not applied an adequate thickness. The analysis was carried out to see if there was any $A\ell_2O_3$ at all on the surface. The samples examined had gone through oven test and they were: TiC sputtered on foil, Si_3N_4 sputtered on foil, HL-800 on foil after oven, $A\ell_2O_3$ sputtered on foil before oven, Inconel base metal as a control. The following specimens were analyzed with X-Ray Diffraction technique: Cr_2O_3 sputtered on foil and Kaman DES on foil. The ED examination of TiC revealed that coating oxidized to TiO_2 during oven test (for diffractogram, see Figure D.4a). The diffraction patterns of Si_3N_4 and SiO_2 are very close and it could not be resolved if the coating was Si_3N_4, SiO_2 or both (for pattern, see Figure D.6a).



(a) (b)

Fig. D.15 Typical SEM Micrographs of Plasma Sprayed Tribaloy 800 on A286 Base (as received)



(a) (b)

Fig. D.16 Typical SEM Micrographs of Plasma Sprayed Tribaloy 800 on A286 Base (surface ground)

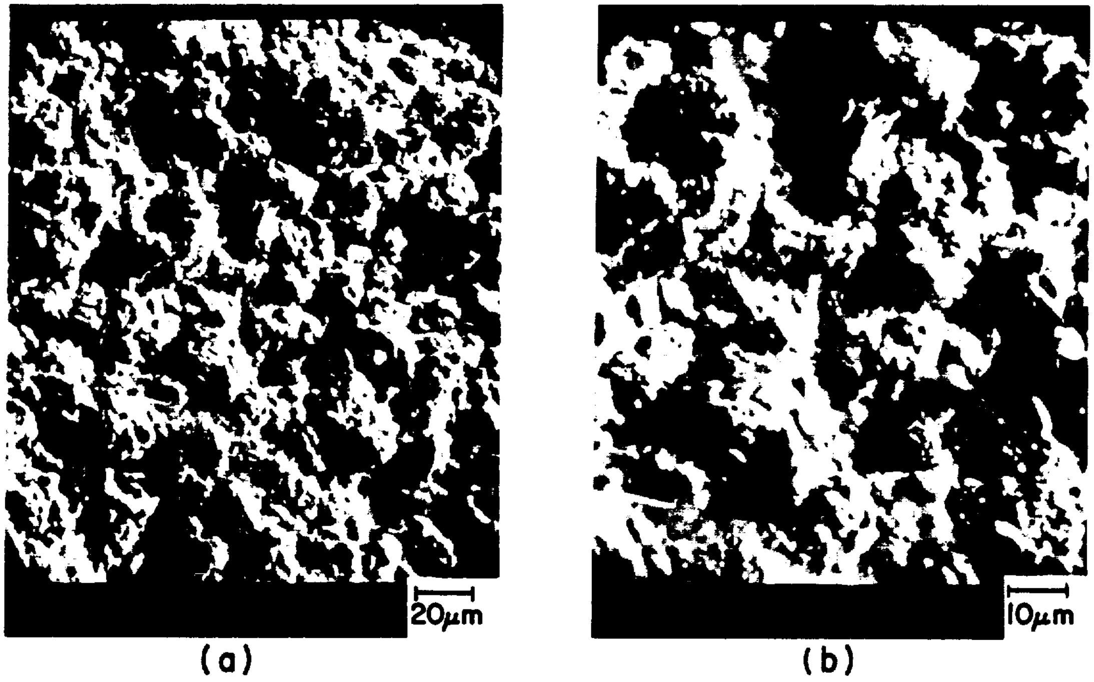


Fig. D.17 Typical SEM Micrographs of Tribaloy 800 (After Oven)

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The diffraction pattern of HL-800 coating (Figure D.14) showed that the coating after the oven test changed to $CdSiO_3$ and Graphite.

No patterns at all could be obtained from $A\ell_2O_3$ coating before oven tests, even though numerous runs were made on different pieces at various angles and orientation. Only after roughening the surface by scratching it, could a pattern be obtained of $^{\alpha}Fe$ and Cr.. X-REDA analysis suggests that there is slight amount of $A\ell$ compound on the surface. It is believed that there was very little $A\ell_2O_3$ coating on the surface and it could not be picked up by ED. There is very slight possibility, if any, that $A\ell_2O_3$ is present, and its structure is amorphous so that patterns could not be obtained. Patterns of an Inconel X-750 heat treated coupon (Figure D.1b) suggest that the surface is composed of spinel compound, $NiO.Fe_2O_3$ which is formed during heat treatment.

X-ray diffraction studies, of Inconel base coated with Cr_2O_3 (sputtered) and Kaman DES, detected chromium oxide (Cr_2O_3) and nickel solid solution. Both coatings before and after oven treatment were predominantly Cr_2O_3. The nickel solid solution peaks were derived from the Inconel X-750 substrate.

APPENDIX E

PHOTOGRAPHS OF BEARING SURFACES AFTER START/STOP TESTS

The Appendix includes the photographs (Figures E.1 to E.15) of the journal and foil surfaces after start/stop tests, not included in Chapter V.

10X
HL-800 ON FOIL

10X
Cr_3C_2 ON JOURNAL

Fig. E.1 Photographs of Surfaces After 500 Cycles at 540° C and same at RT

10X
TiC ON FOIL

10X
TRIBALOY 800 ON JOURNAL

Fig. E.2 Photographs of Surfaces After 30 Cycles at RT (Test No. 3)

10X
KAMAN DES ON FOIL

10X
TRIBALOY 800 ON JOURNAL

Fig. E.3 Photographs of Surfaces After 10 Cycles at RT (Test No. 4)

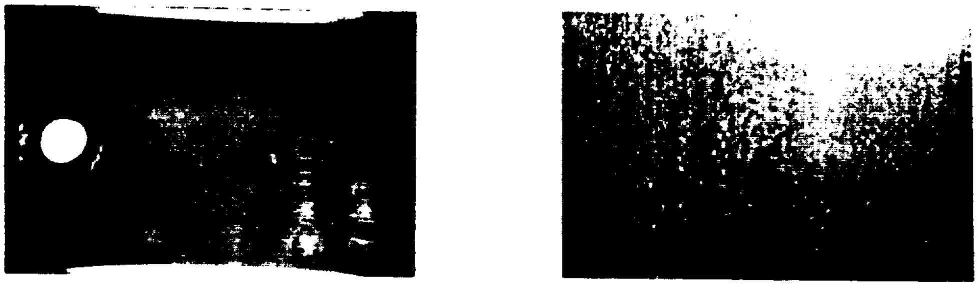
10X

Hi-T LUBE ON FOIL

10X

Cr_3C_2 ON JOURNAL

Fig. E.4 Photographs of Surfaces After 500 Cycles at 540° C and same at RT



10X

KAMAN DES ON FOIL

10X

Cr_3C_2 ON JOURNAL

Fig. E.5 Photographs of Surfaces After 10 Cycles at RT (Test No. 6)

10X

UNCOATED FOIL

10X

NASA PS 106 ON JOURNAL

Fig. E.6 Photographs of Surfaces After 500 Cycles at 650° C and same at RT

5X KAMAN DES ON FOIL

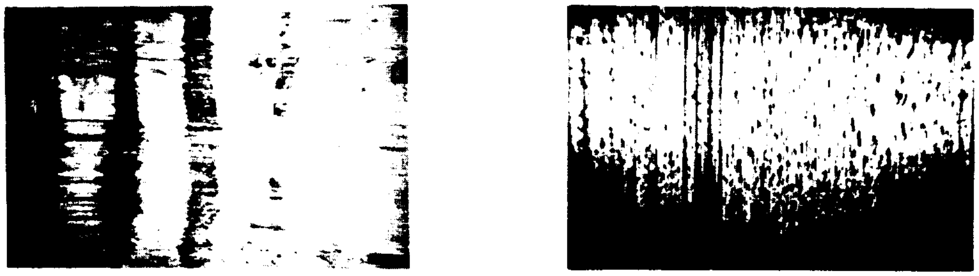
5X NASA PS 106 ON JOURNAL

Fig. E.7 Photographs of Surfaces After 100 Cycles at RT (Test No. 8)

5X Cr_2O_3 ON FOIL

5X KAMAN DES ON JOURNAL

Fig. E.8 Photographs of Surfaces After 500 Cycles at RT (Test No. 10)



5X Cr_2O_3 ON FOIL

5X Cr_2O_3 (SP.) ON JOURNAL

Fig. E.9 Photographs of Surfaces After 500 Cycles at 650° C and same at RT (Test No. 12)

5X

KAMAN DES ON FOIL

5X

Cr_2O_3 (SP.) ON JOURNAL

Fig. E.10 Photographs of Surfaces After 500 Cycles at 650° C and same at RT (Test No. 13)

5X

TiC ON FOIL

5X

Cr_2O_3 (SP.) ON JOURNAL

Fig. E.11 Photographs of Surfaces After 500 Cycles at 650° C (Test No. 14)

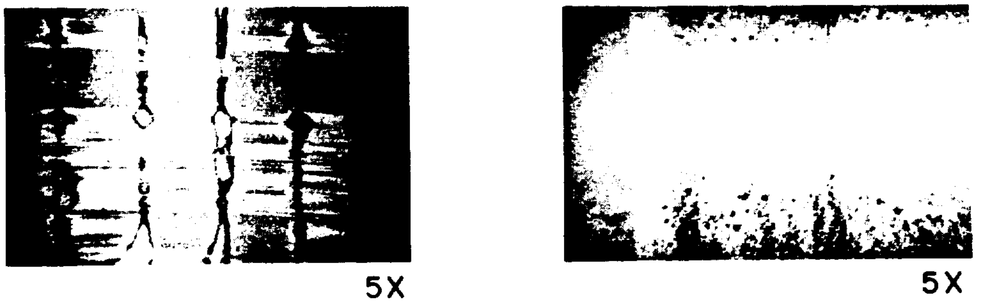
5X

Cr_2O_3 ON FOIL

10X

TRIBALOY 800 ON JOURNAL

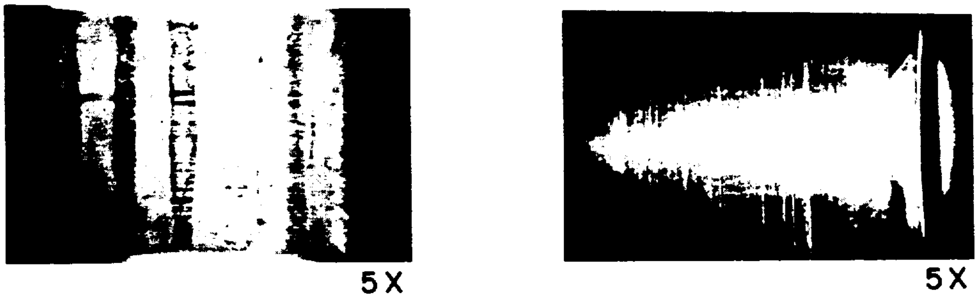
Fig. E.12 Photographs of Surfaces After 1000 Cycles at 650° C and 500 at RT (Test No. 16)



Si_3N_4 ON FOIL

TRIBALOY 800 ON JOURNAL

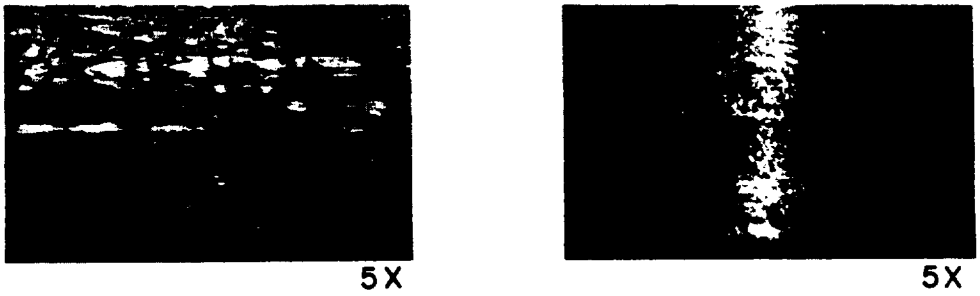
Fig. E.13 Photographs of Surfaces After 500 Cycles at 650° C and same at RT (Test No. 17)



TiC ON FOIL

KAMAN DES ON JOURNAL

Fig. E.14 Photographs of Surfaces After 500 Cycles at 650° C and same at RT (Test No. 18)



UNCOATED FOIL

NASA PS 120 ON JOURNAL

Fig. E.15 Photographs of Surfaces After 500 Cycles at 650° C and same at RT (Test No. 19)