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WEAR-RESISTING OXIDE FILMS FOR 900°C

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ABSTRACT

A study was conducted to develop low-friction, wear-resistant surfaces on high-temperature alloys for the temperature range 26°C to 900°C. The approach investigated consisted of modifying the naturally occurring oxide film in order to improve its tribological properties. Improvement was needed at low temperatures where the oxide film, previously formed at high-temperature, spalls due to stresses induced by sliding. Experiments with titanium, tungsten, and tantalum additions showed a beneficial effect when added to nickel and nickel alloys. Low friction was maintained down to 100°C from 900°C. For unalloyed nickel friction and surface damage increased at 400°C to 500°C. Other approaches proved less successful and require further study.

1.0 INTRODUCTION

For the past 50 years, temperatures in advanced heat engines have been increasing. New-generation engines will require lubricants for 1000°C and higher. One of the most critical applications is the regenerator seals on the automotive gas turbine. In this seal, a metal plate slides against a porous ceramic surface for several thousand hours at speeds on the order of 10 cm/sec. Other critical applications include cages for ceramic rolling-contact bearings, control surfaces for advanced jet engines and variable-area nozzles for turboramjet propulsion systems which operate to temperatures of 1650°C. Some lubrication scheme will have to be devised for such applications.

Current high-temperature solid-lubricant work is being carried out by Sutor (1), King (2), Murray (3), Erdemir (4)(5), Sliney (6)(7), and Peterson (8)(9)(10). Sutor and King have identified effective solid lubricants in the 800°C range but have not divulged the compositions. Erdemir has studied lubrication with silver and boric oxide. Murray has been studying the use of tungstates as solid lubricants to 800°C. Much of the understanding of effective high-temperature solid lubrication is due to Sliney, who explored the use of fluorides and mixed fluorides as lubricants, and developed bearing materials and plasma-spray coatings based on these concepts. Using calcium fluoride as a lubricant, Ford Motor Co. developed a series of plasma-spray coatings (11)(12)(13) for use in

regenerator seals operating to 900°C. For temperatures above 900°C, the only recent work is due to Sliney (7). He studied rare earth oxides and fluorides as solid lubricants to 1000°C. He concludes that powdered CeF_2 and LaF_3 can lubricate nickel-base super alloys to temperatures of 1000°C. Rare earth oxide powders La_2O_3 and CeO_2 gave friction coefficients of 0.20 or less above 800°C but higher friction (0.50 to 0.60) at room temperature. Peterson has been studying oxide lubrication for the past 10 years. The approach being investigated is to study the lubricating properties of oxides which occur naturally on metal surfaces and to develop alloys which form these oxides. Current work involves rhenates and molybdates.

For long-term usage above 900°C it will probably be necessary to use oxide lubricants. If effective ones can be found, then a simple solution would be available for an application like the regenerator seal: fabricate it with an alloy which forms a lubricating oxide. The objective of this study was to explore this concept for the regenerator seal. The work, performed at Wear Sciences under Argonne National Laboratory contract 20082401, consisted of a literature search to identify suitable alloy compositions, fabrication of appropriate materials and determination of their friction-temperature behavior.

2.0 BACKGROUND

For temperatures above 900°C, nickel-base alloys are the most suitable structural materials. The question then becomes what alloying elements need to be added to provide reasonable friction and wear values over the broad temperature range 26 to 900°C. The friction and wear values for a typical nickel-base alloy are shown in Figure 1 and Figure 2 (14). Friction coefficients are 30-minute values from separate tests for Inconel 750X sliding against itself using a hemispherically tipped (0.95 cm diameter) pin sliding on a flat plate. Friction is constant and high up to 700°C, where it decreases to 0.30 due to the formation of NiO. If a cyclic test is run where friction is continuously measured as the temperature is changed, friction decreases at approximately the same temperature in each cycle. Upon cooling, friction remains low to approximately 300°C.

Wear/Time curves for sliding at 26°C and 982°C are shown in Figure 2. It can be seen that the wear rate is very low at the high temperature. The indicated wear rate at 982°C yields a dimensionless wear coefficient¹ of 3.2×10^{-6} .

For the regenerator seals, a friction coefficient of 0.30 is probably adequate; however, that coefficient must be maintained down to room temperature. Thus, this nickel alloy would be adequate if the protective nickel oxide did not spall

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$$\text{Wear Coefficient} = \frac{\text{Wear Volume} \times \text{Hardness}}{\text{Load} \times \text{Sliding Distance}}$$

off the surface at 300°C. It is known from the metal-oxidation literature that certain alloying ingredients contribute to oxide-spalling resistance. It is possible that these or other alloying additions could promote tribological spalling resistance in high-temperature sliding. This is the thrust of the work reported herein.

The high-temperature wear rate found for this alloy is very good; however, an order-of-magnitude improvement may be needed for the regenerator application. If an alloy is found which gives adequate friction over the 26°C to 1000°C temperature range, further work may be necessary to improve wear rates.

3.0 MATERIALS SELECTION

A review of the oxidation literature suggests that a variety of approaches may be available to stabilize an oxide film during temperature cycling. These may be categorized as:

1. Internal oxidation
2. Ductile oxide films
3. High-adhesion films
4. Increased low-temperature oxidation
5. Stress-free films

These are discussed separately in the following paragraphs.

3.1 INTERNAL OXIDATION

An alloy consisting of a metallic base with small additions of solute atoms can oxidize internally under certain conditions. Oxidation proceeds by the appearance of oxide particles within the alloy and concentrated to a greater extent at the grain boundaries. This type of oxide formation produces a metal-bonded-oxide structure which could have the same effective tribological properties as the metal-bonded carbides. In any case, internal oxidation produces an oxide which is firmly pegged into the metallic surface since oxidation advances inwardly by oxygen diffusion.

Detailed analytic (15)(16)(17)(18) and experimental studies (19)(20)(21)(22) of internal oxidation show that there are several stages to the process, depending upon time and composition. Internal oxidation will take place when atomic

oxygen dissolves into the alloy surface, diffuses inwardly and forms internally precipitated oxide particles. At the advancing oxide front, the inward diffusion of oxygen and the outward diffusion of the solute atoms provide concentrations at the surface in excess of alloy concentration. A reaction will take place if the free energy of formation of the solute metal oxide is lower than that for any other constituent, the solvent metal has significant oxygen solubility and diffusivity and the solvent metal content is less than a critical value. The critical value is that value where a complete film is formed. For example, detailed studies of Ni-Si alloys (23) show that with silicon concentrations less than 3%, internal oxidation will take place with SiO_2 clusters in the nickel matrix. From 3 to 33% there is continuous film formation of irregular scales of NiO which contain various amounts of silicon. Above 33%, only a protective layer of SiO_2 is formed. This result is general for other solute and solvent constituents, except that the concentrations may vary; for internal oxidation, the concentrations should be about 3%.

Nickel as a solvent metal provides sufficient oxygen diffusion and solubility, and has a reasonably high free energy of formation ($-132.3 \times 10^3 \text{ kJ/kMole}$) at 927°C (24). Potential solute atoms which could be added to a high-temperature alloy are La(-1548) Y(-1541) Ta(-1523) Ce(-1426) Nb(-1385) Al(-1295)

Ti(-1191) and Si(-824).² Those elements which have been shown to promote internal oxidation are Be, Cr, Al, V, Tc (19); Si (21); Al, Ti (25); Y (26); Al (27); Tc, Ta (28) and Va (29). Based upon the available literature, the following elements were chosen to evaluate the internal oxidation approach: Ta, Ti, Y, Si and Ce. It is desired to know if the presence of these atoms at the alloy surface stabilizes the oxide film in sliding as well as in oxidation.

3.2 DUCTILE OXIDE FILMS

It is well known that the presence of ductile oxide films prevents excessive oxidation (30)(31). They are effective because they can flow rather than fracture during oxide growth and thermal cycling. It is also known that ductile oxide films provide good wear protection. For example, Evans et al (32) measured the ductility of NiO and found that it increased significantly in the 700 to 800°C range. From Figure 1, it can be seen that this is the temperature where friction decreased significantly. It can be suggested that a highly ductile NiO film will smear over the surface and prevent metal to metal contact.

Data are not available on temperatures where oxides become ductile especially when they are under pressure. However, Peterson (33) suggests that certain oxides like the molybdates,

²Numbers in parentheses are the free energy of formation at 927°C in the same units as given for nickel. (51)

tungstates and rhenates become effective when they are within 300°C of their melting point. Low room-temperature hardness is also a reasonable criterion. Some oxides with low hardness at room temperature are: Re_2O_7 , MoO_3 , BaO , V_2O_5 , CaO , ZnO and CuO .

Friction coefficients of these and other similar oxides (with various counterface materials as described in the listed references) have been assembled in Table 1. It can be seen that they give low friction coefficients at high temperatures in addition to having low hardness at room temperature.

In Reference 35, a study was conducted of the lubricating characteristics of oxides. It was concluded that those oxides which performed best at 700°C were those which flowed into a continuous film on the surface. The oxides which performed best in this regard were MoO_3 , WO_3 , CoO , ZnO , CuO , and SrO . It is this ability to form a film which controls tribological properties. More recently, Glascott, Stott and Wood (40) reported on the effectiveness of oxides in reducing sliding wear of alloys. They concluded that the rubbing produces debris which consists of oxides of the main alloy constituents. The rubbing process reduces the particle size and compacts the particles into a solid film on the surface. They noted that certain oxides (MoO_3 , ZnO , NiO , ZrO_2 , Cu_2O , MnO , and TiO_2) were "perfectly plastic" and should perform much better on alloy surfaces.

TABLE 1
FRICTION COEFFICIENTS OF OXIDES

OXIDE	TEMP°C	FRICTION COEFFICIENT	REF.
Re ₂ O ₇	316	.23	33
MoO ₃	700	.20	33
	595	.27	34
WO ₃	800	.39	34
	700	.55	35
	600	.56	33
V ₂ O ₅	600	.32	33
ZnO	700	.33	35
CuO	600	.22	33
	600	.25	36
CoO	625	.18	36
B ₂ O ₃	650	.14	35
			37
CeO ₂	>800	.20	38
CaO	700	.20	39
CuMoO ₄	600	.28	36
CoMoO ₄	600	.37	36
	800	.27	34
NiMoO ₄	700	.29	35
CuWO ₄	700	.41	35
FeWO ₄	700	.43	35
NiWO ₄	700	.51	35
CoWO ₄	800	.20	34

The friction properties of tantalum oxides (T_2O_5) have not been studied; however, this oxide has been shown to improve cyclic oxidation behavior. Its effectiveness was attributed to increased scale plasticity (41).

From the above results, the following elements were chosen to evaluate the "more ductile oxide" concept: W, Ce, B, Zn, Ca, and Ta. These oxides should be stable at 1000°C and would be oxidized preferentially to nickel in the alloy. They all, except Zn, could be added in small quantities to high-temperature alloys.

3.3 HIGH-ADHESION FILMS

It is well-known that the addition of small amounts of atoms that have a high affinity for oxygen significantly reduces the oxidation rate of alloys (42)(43)(44)(45). In the original work (45) this effect was found for atoms in the periodic groups II, III, IV, and V, with the effect decreasing from group II to group V and increasing with increasing atomic mass. This suggested the use of elements such as barium, lanthanum, cerium, zirconium, yttrium, and strontium. Although all of these have been used, attention has been focused primarily on yttrium and cerium. The reason for this effect is that with many alloys, the oxidation process consists of the outward diffusion of metal atoms through the oxide film. This process depletes metal from the oxide/metal interface, causing voids and poor film adhesion. Wittle and Stringer (46), in a comprehensive review, conclude

that "improvement in oxide scale adherence seems to be the major beneficial effect of these additions; and, of the mechanisms suggested, elimination of interfacial voids and peg formation seems to be of the most significance in most cases."

Of the above elements, Ba, Ce, Y and Zr were chosen to evaluate this effect. It is interesting to note that this adhesion enhancement seems to take place when either the elements or their oxides are added to the alloy (48).

3.4 INCREASED LOW-TEMPERATURE OXIDATION

It has been shown that the addition of molybdenum to nickel lowers the temperature at which the oxide becomes effective by 300°C (49). This is due to the formation of MoO_3 on the surface which is an effective lubricant. There are other ways of achieving this effect. First, nickel oxide is a cation-deficient semiconductor. If small amounts of metals are added which form higher-valency ions, the oxidation rate should be increased. That this is true has been experimentally verified (50) where 16 such solute metals were shown to increase the oxidation rate of nickel. Of these, the largest increases were found with zirconium, chromium, thorium and cerium, with amounts of less than one percent. Of these, zirconium and cerium seem the most suitable for alloy experiments or surface modification. Because of the small quantities needed, this approach would be particularly suitable for ion implantation. Vanadium is another possible addition which works by another method. At higher

interfacial temperatures, a molten oxide is produced which greatly increases the diffusion rates involved in the oxidation process. If the sliding interface flash temperature reaches 675°C, the melting temperature of V_2O_5 , then increased oxidation may occur. Thus NiZr, NiCe and NiV alloys could be used to evaluate this concept.

3.5 STRESS-FREE FILMS

Stress-free films would be formed with oxide films that are similar to the base metal in structure, thermal expansion, and molar volume. This is difficult to achieve, since oxides inherently have lower thermal expansion and larger molar volumes than metals. However, the values for nickel are compared with several oxides in Table 2.

TABLE 2				
Property	Ni	NiO	MgO	FeO
Thermal Expansion ¹	13.3	10	14	12.4
Molar Volume ²	6.6	10.9	11	13
Crystal Structure	Cubic	Cubic	Cubic	Cubic
¹ $\alpha \cdot 10^6 \text{ } ^\circ\text{K}^{-1}$ ² cm^3				

It can be seen that MgO and FeO have thermal expansion values closer to Ni than NiO and they also have lower free energies of formation. Therefore, they would be preferentially formed on the surface. The larger molar volume may be desirable, since it

puts the oxide into compression. Thus, experiments with NiMg or NiFe alloys could evaluate this concept. NiFe alloys are somewhat questionable, since an oxide other than FeO may be formed. A second problem is that NiO would also be formed at higher temperatures. Accordingly, experiments were only conducted with magnesium additions which form solid solutions with NiO.

4.0 MATERIALS PREPARATION

A summary of the materials approaches is shown in Table 3. It can be seen that some of the additive elements apply to more than one approach. Also shown in Table 3 are the quantities which apply to each approach based upon the cited literature.

Although these elements (with the exception of zinc) have been added to high-temperature alloys, there was no attempt at this stage of the program to evaluate their effect on the other properties such as strength and corrosion resistance. The purpose in this initial stage was only to evaluate the concept of oxide retention during sliding over a broad temperature range. If one approach is successful, its practicality for specific applications could be developed in later investigations.

It was decided to work primarily with nickel and nickel-base alloys. Such alloys are being used for regenerator seals and are almost exclusively used for various temperatures above 1000°C. As shown in Table 4, four different materials techniques were used to evaluate the concepts summarized in Table 3. Late in the program, a fifth technique, pitted surfaces, was added. This technique did not evaluate any of the Table 3 materials approaches for oxide retention, but rather merely added a desirable high-temperature solid lubricant into nickel alloy surfaces.

TABLE 3 MATERIALS APPROACHES					
	INTERNAL OXIDATION	DUCTILE FILMS	STRESS-FREE FILMS	HIGH ADHESION	LOW-TEMP OXIDATION
Cerium	✓	✓		✓	✓
Yttrium	✓			✓	
Zirconium				✓	✓
Tantalum	✓	✓			
Titanium	✓				
Boron		✓			
Magnesium			✓		
Barium				✓	
Calcium		✓			
Vanadium					✓
Zinc		✓			
Tungsten		✓			
Silicon	✓				
% Addition (Wt)	1-3	5-20	>1	>1	5

TABLE 4 MATERIALS TECHNIQUES				
	BINARY ALLOYS	OXIDE DIFFUSION	IMPLANTATION	COMMERCIAL ALLOYS
Cerium		✓		✓
Yttrium				✓
Zirconium		✓	✓	✓
Tantalum	✓			
Titanium	✓	✓	✓	✓
Boron	✓		✓	✓
Magnesium		✓	✓	
Barium		✓		
Calcium		✓		
Vanadium			✓	
Zinc		✓		
Tungsten	✓	✓		✓
Silicon			✓	

These materials techniques are discussed in the following paragraphs.

4.1 BINARY ALLOYS

Binary nickel alloys were prepared containing 2 and 20 weight percent of tantalum, titanium, and tungsten. A Ni-1.5B alloy was also prepared. Small ingots of these alloys were fabricated by mixing the powders and melting them in an inconsumable-electrode arc furnace. In order to obtain high-purity samples, all of the powders were first heated to high temperatures for one hour in a vacuum furnace. This expelled

the dissolved gases. The mixed powders were placed in a water-cooled copper crucible which served as the anode in the argon-arc furnace. The cathode was a tungsten rod supplied with sufficient amperage to melt all the alloy constituents. When necessary, the alloys were remelted several times to assure a uniform distribution. The structure was microscopically examined and small specimens were prepared for friction testing. All of these specimens were prepared, at no cost to the project, by Prof. S. Z. Li at the Institute for Metal Research in Shenyang, China.

4.2 OXIDE DIFFUSION

In reference 52, the authors found that superficially applied oxide powders applied as powders on nickel-chromium alloys at high temperatures (1000°C) could interact with the naturally occurring oxide and affect oxidation resistance and the nature of the surface oxide. They coated nickel-alloy surfaces with MgO , Cr_2O_3 , Co_3O_4 and CeO_2 and heated them in air or oxygen for 1 hour at 1000 - 1200°C . They found that the oxides were incorporated into the scale and that CeO_2 reduced the oxidation rate.

This simple approach seemed ideal for this study since it changed the nature of the oxide films, but should not influence the properties of the alloy. For example, it is much easier to form a tungstate this way than by adding tungsten to the alloy.

Unfortunately, there was no way to control the process; the thermodynamics determined what oxides were formed.

Because of the simplicity of preparing such specimens, almost all of the Table 4 materials were tried.

The procedure was as follows: An alcohol slurry of each oxide powder was made. It was painted onto the surface of a nickel-200 flat which had been ground to a finish of 0.1 μ m CLA. The coating thickness was approximately 1mm. The coating was air dried. The specimen was then placed into a furnace and heated for approximately 10 hours at 1000°C. The specimens were furnace-cooled, removed at room temperature and examined.

In Table 5, various weights are given for the specimens involved, both before and after heating. The delta column marked "1" represents the weight of the powder on the surface before heating. The column marked "2" represents the increase in weight of the specimen and the powder during the heat cycle. Note that the blank specimen without powder gained 0.14 grams due to oxidation. The only large variation from this value is with WO₃. This may be due to the spalling of the reaction film. All surfaces were scrubbed with a stiff brush and hot water. Column 3 shows the loss of weight due to scrubbing and may be compared to Column 1, the original amount of powder on the surface. Column 4 shows the gain in weight of the specimen due to the heating process.

This information is given for reference purposes. No significant conclusions are drawn from the data, since some

spalling has occurred with certain specimens and some vaporization (CO_2 from BaCO_3) can take place at these temperatures.

TABLE 5 OXIDE DIFFUSION DATA								
OXIDE	SPECIMEN WEIGHT g	SPECIMEN + OXIDE g	1 ▲ g	AFTER HEATING g	2 ▲ g	AFTER SCRUBBING g	3 ▲ g	4 AMOUNT INTER- ACTING g
BaCO_3	64.47	64.56	.11	64.70	.14	64.68	-.02	.21
CeO_2	64.18	64.22	.04	64.38	.16	64.34	-.04	.16
TiO_2	64.22	64.23	.01	64.37	.14	64.35	-.02	.13
ZrO_2	64.26	64.36	.10	64.52	.16	64.43	-.09	.17
MgO	63.99	64.02	.03	64.13	.11	64.11	-.02	.12
CaO	63.97	64.01	.04	64.11	.10	64.08	-.03	.11
ZnO	63.96	63.99	.03	64.14	.15	64.13	-.01	.17
WO_3	64.66	64.75	.09	64.77	.02	64.75	-.02	.09
BLANK	63.26	-	.00	63.40	.14	63.39	-.01	.13
Column 1: Weight of oxide originally on surface Column 2: Change in weight of the specimen after heating with oxide to 1000°C Column 3: Change in weight after removing loose oxide by scrubbing Column 4: Amount of combined oxide on surface								

A microscopic description of the surfaces is given in Table 6. In all cases except BaCO_3 and WO_3 , a considerable amount of loose powder remained on the surface which could be scrubbed away. Beneath the loose powder there was some interaction film in every case. This was a strongly adherent film which was difficult to remove. In some cases (WO_3 , ZnO , MgO), it seemed obvious that a reaction had occurred to produce a molten film at temperature. In all cases, the interaction film looked considerably different from the naturally occurring oxide film which was black, smooth and shiny.

Friction tests were run on the interaction film without further surface finishing.

TABLE 6 SURFACE OBSERVATIONS - OXIDE DIFFUSION SPECIMENS	
OXIDE	OBSERVATIONS
BaCO ₃	Very little loose powder on surface. Surface appears to have been molten and crystallized into small regular grains on surface. Surface rough and black. Appears to have reacted strongly with surface.
CeO ₂	Most powder remains on surface. Dull black film beneath powder. Smooth very fine-grained surface. Some NiO spalling away from interaction zone.
TiO ₂	Most of the powder could be brushed away. Green two-layered film under powder. Top layer cracked and spalled. Bottom layer black, shiny and patchy. NiO film spalled out of interaction area.
ZrO ₂	Most powder remains and could be brushed away. No powder sintering. Black fine-grained film remains on surface.
MgO	Most powder could be brushed away. Green film below loose powder spalled in some places. Apparently there is a strong reaction with the surface. Film consists of large shiny grains.
CaO	Most of powder remains on surface. Black mottled film below powder. No texture to film. NiO film spalled out of contact area.
ZnO	Powder strongly adherent. Very little could be brushed away. Thick black film remains with large grainy structure; some surface spalling in interaction areas. Apparent previously molten reaction film below black surface film.
WO ₃	No powder left on surface; apparent strong reaction with surface. Layered surface with large-grained surface film and apparent previously molten film below. Film is greenish and below the level of NiO film.
BLANK	Shiny, smooth, strongly adherent black film on surface. No spalling.

TABLE 7
COMMERCIAL ALLOYS¹

	Ni	Cr	Fe	Co	Ti	W	Al	Mo	Ce	Y	B	Zr
Inconel X750	70 ²	15	7	1.0	2.5	-	-	-	-	-	-	-
Nimonic 86	65	25						10	0.03			
MA 6000	75	15			2.5	4		2		1.1		
MAR-M200	62	9		10	2	12	5					
Nimonic 115	60	14		13	4		5				0.16	
Nimonic PE 11a	40	18	35		2.3		0.8	5				0.2

1. High-temperature nickel-base alloys made by Inco International
2. Weight percent

4.3 IMPLANTATION

Implantation is probably the best method to prepare surfaces to produce the desired oxide-stabilization effect. Only a small amount of the additive element is needed and only at the surface. Upon heating, the interacting element will diffuse to the metal oxide interface where it is needed or will interact to form the ductile film. Implantation also should not affect the alloy properties as do alloying additions or high-temperature diffusion.

Surface implantation was carried out by Prof. Paul J. Wilbur of Colorado State University. The process used was high current density ion implantation. Specimens of Ni200 and Inconel 750X were implanted with Ti, Mg, Va, B, Zr and W at 50 KeV with ion doses of approximately 5×10^{17} atoms/cm². Current densities were 200-500 microamps/cm² for several minutes. This process implants atoms to a depth of several hundred angstroms. At this dosage the surfaces were probably

saturated, according to Prof. Wilbur. For tungsten, a different process was used (ion beam mixing). This process consisted of sputter coating the samples with 600Å of tungsten and then bombarding it with 75keV Ar to knock atoms from the coating into the substrate material. All specimens were maintained at temperatures between 100°C and 200°C during processing.

4.4 COMMERCIAL ALLOYS

Several commercial alloys contain metals which are under consideration in this study. These are shown in Table 7. Original plans called for an evaluation of such alloys to determine which alloy best stabilized the oxide film. However, these tests were not run in the current investigation since it was felt that the other alloy ingredients would also exert considerable influence over their tribological behavior. Comparative tests can be run at a later date if necessary.

4.5 PITTED SURFACES

This approach was evaluated even though it had nothing to do with oxide stabilization. Essentially, the technique involves the chemical pitting of nickel-alloy surfaces and filling the pits with high-temperature solid lubricants (fig. 3)(53). The nickel-alloy specimen used was Inconel 601, which has a composition of 60Ni23Cr13Fe1.5Al.

To prepare the surface, two specimens (stainless steel anode and Inconel 601 cathode) were cleaned and placed on either side of a 100 ml beaker. These specimens had a ground finish ($0.1\mu\text{m CLA}$) and a total surface area of 15 cm^2 . They were immersed in an FeCl_3 solution ($2.5\text{g}/100\text{ml}$). The beaker temperature was maintained at 30°C by means of a water bath. A current of 1 A was carried at 12 volts for a period of 5 minutes. This procedure introduced pits which were about $50\mu\text{m}$ deep with various widths. The density was approximately 20-40 pits/ mm^2 .

After cleaning, the pits were filled with solid lubricants by placing the powder on the surface and burnishing it into the surface. The materials used in this investigation were nickel tungstate, lead oxide, cerium oxide, zinc oxide and silver.

5.0 FRICTION-TEMPERATURE RESULTS

A series of friction-temperature tests were run to evaluate the previously discussed concepts. The results are discussed in the following paragraphs.

5.1 APPARATUS & PROCEDURE

A schematic diagram of the reciprocating test apparatus used in these experiments is shown in Figure 4. The test specimens consisted of an Al_2O_3 ball (1.27cm diameter) sliding back and forth, in a single track, on a flat plate. The ball is mounted in a special holder and is affixed to the arm which is moved back and forth with an air cylinder. A strain gauge mounted between the cylinder and the arm allows the friction force to be measured. The flat specimen is mounted on a stationary table. Cartridge heaters are mounted in this table and in the ball-specimen holder. The ball moves back and forth in a track 2.5 cm in length and at an average rate of 5 cm/sec. The load is usually 145N or 50N and the temperature range is room temperature to 900°C.

In operation, the specimens were cleaned and placed in the apparatus. The temperature was increased to the highest temperature, usually 900°C, and the test started. The test was run for approximately 5 minutes. The temperature was then decreased to a lower level and the specimen and the ball were moved to a new track. Another 5 minute test was run. This procedure was followed to the lowest temperature (usually that

temperature where friction increased significantly). In some cases, the same procedure was followed but with increasing temperature, starting at room temperature.

For most of these experiments, an Al_2O_3 ball was used sliding against the following flats:

Binary Ni alloys

Nickel alloys

Nickel 200 with diffused oxides

Pitted Inconel 601 surfaces filled with a solid lubricant

Implanted Ni 200

Implanted Inconel 750X

In some cases, materials and procedures varied; these are described in conjunction with the tests involved.

An Al_2O_3 ball was used because the regenerator core will probably be made of ceramic and because the Al_2O_3 would not be expected to be significantly changed or worn during the course of the experiment.

5.2 BINARY NICKEL ALLOYS

The friction-temperature behavior for nickel alloys containing Ti, Ta, W, and B are shown in Figures 5-8.

Two tests were run for each alloy: one with increasing temperatures, and one with decreasing temperatures. A new ball was used for each temperature, but sliding was on a single track at all temperatures.

The results for Ni₂₀Ti and Ni₂Ti are shown in Figure 5. For Ni₂₀Ti, friction decreased from room temperature to 400°C and then increased slightly as the temperature was raised to 900°C. For Ni₂Ti, friction remained high to 500°C and then decreased to 0.38 at 900°C. In the cooling test, friction remained low until 100°C. The lower friction at higher temperatures is undoubtedly due to the formation of an effective oxide film.

Thus, it can be seen that the Ni₂Ti alloy retains its oxide to the lowest temperature; this meets the objectives of the study. Both alloys are an improvement over unalloyed Ni₂₀₀ as shown in Figure 9. For unalloyed nickel, friction ranges from 0.50 to 0.60 at low temperatures, and increases to 1.00 at 400°C; thereafter, friction decreases. The large increase in friction at 400°C is probably due to the transfer of nickel to the Al₂O₃; friction is then essentially nickel on nickel. Above 400°C, some oxidation occurs, but the amount is insufficient to lower friction into the 0.30 to 0.40 range. A test started at 800°C gives low friction until somewhat below 500°C, where it increases significantly.

The friction-temperature trends for nickel-tungsten alloys are very similar to those for titanium alloys (Fig. 6) except that friction is lower at 900°C for tungsten and friction increases to 0.44 at lower temperatures. This is also an acceptable result: a friction coefficient of 0.44 is acceptable in many applications as long as there is no surface

damage. An examination of the Ni2W surface from the 100°C run showed that the oxide film had been retained during the cooling cycle.

Good results were also obtained with Ni20Ta. Friction coefficients ranged between 0.30 and 0.40 from 100°C to 900°C. Although these friction values would be acceptable in many applications, the addition of 20 wt percent tantalum would completely change the properties of an alloy. It would be much better to keep additions as low as possible. The Ni-2Ta gave friction coefficients which show little improvement over nickel 200.

The results for Ni-1.5B are shown in Figure 8. The results are similar with increasing and decreasing temperature, except with decreasing temperature there is much more variation at a single temperature. This variation usually means that the protective film is being removed. Again, there is little improvement over nickel alone.

A direct comparison of the different alloys with both increasing and decreasing temperature is shown in Figures 10 and 11. With increasing temperatures, the high alloying contents gave the lowest friction coefficients and the lowest temperatures of friction reduction. This is probably due to the properties of the alloy rather than any affect on its oxidation characteristics. With low alloying contents, high friction persisted to higher temperature; however, tungsten gave the lowest drop-off temperature (300°C). The reason for

this is not understood at the present time. With decreasing temperatures, friction began to increase with all the alloys except Ni2Ti. This increase corresponds to the removal of the oxide film as determined by surface examination.

The results of this phase of the investigation suggest the use of titanium and tungsten to stabilize the oxide film during sliding contact. Based on this work, some special alloys were prepared and evaluated. These results are described in a later section.

5.3 OXIDE-DIFFUSION COATINGS

The results for the oxide-diffusion coatings are shown in Figure 12. In this series, only decreasing-temperatures tests were conducted. In addition, the load was reduced to 50N (5kg) because it was found that the use of an Al_2O_3 ball resulted in a very severe test. The Al_2O_3 ball does not wear to reduce the nominal pressure. Each data point in Figure 12 represents a separate 5-minute test with a different track.

The results for many of the diffusion treatments gave approximately the same behavior as Nickel 200 (MgO , TiO_2 , CaO , ZnO), indicating little benefit. Friction increased at approximately the same temperature as Nickel 200 for all specimens. At the lower temperatures, less oxide was removed from the lower friction specimens.

ZrO_2 , CeO_2 , WO_3 , and BaO gave much lower friction at 800°C . This is understandable since all but ZrO_2 are

effective solid lubricants at high temperatures. However, no reason could be postulated for the low friction of 0.06 with ZrO_2 . A literature review (1977 to 1993) to determine the nature of ZrO_2/NiO reactions revealed no phase diagram and no low-melting-point eutectics. In fact, there appears to be no reaction between NiO and ZrO_2 at such temperatures. There is, however, a ZrNi eutectic consisting of 22 atomic percent nickel which melts at 961°C , but there is no obvious reason why this would form.

Sliding tests using NiZrO_3 as a lubricant showed that it was a poor lubricant at 800°C , yielding a friction coefficient of 0.46. Thus, the only explanation for the low friction was that a NiZr eutectic was formed. Examination of the surface showed that recrystallization had taken place but a surface analysis was not carried out.

Based on these results, further work was carried out with CeO_2 , WO_3 , ZrO_2 , and TiO_2 . These were diffused into Inconel 750X specimens under the same conditions used for Nickel 200. Inconel 750X was chosen since it contains 2.5 percent titanium (Table 7) and was found to be beneficial for oxide stabilization (Figure 5).

Friction-temperature tests were run with these specimens under the same conditions as for the Nickel 200 tests. The results are shown in Figure 13. The line drawn in this figure represents the friction value for Al_2O_3 sliding against an Inconel 750X specimen which had been preoxidized for 10 hours

at 1000°C (the same as the oxide-diffused specimens). It can be seen that none of the oxides, except ZrO_2 , provided any benefit over the oxidized Inconel 750X. However, the ZrO_2 gave much higher friction (0.45) at higher temperatures.

Since none of the diffusion treatments appeared to have any significant benefit on oxide stabilization, further efforts using this approach were abandoned.

5.4 IMPLANTATION

A nickel specimen was obtained which was implanted with titanium. This specimen was run in the temperature cycle test at 145N load. The results for Ti-implanted nickel are shown in Figure 14. Friction was high even at 800°C. As mentioned previously, this test was probably too severe. The Al_2O_3 ball does not wear, so high nominal pressure persisted and a deep groove was worn in the nickel flat. Based on this result, Inconel 750X plates were obtained which had been implanted with tungsten. This sample was run with a nickel 200 slider and at a reduced load of 50N. These results are also shown in Figure 14. It can be seen that much lower friction coefficients were obtained. However, friction still increased at about 500°C and there were metallic wear particles and surface damage at lower temperatures.

Since Argonne was conducting a much more thorough evaluation with the same specimens, no further work was carried out.

3.5 PITTED SURFACES

Temperature-cycle tests were run with pitted Inconel 601 surfaces filled with NiWO_4 , PbO , ZnO , CeO_2 , and Ag . The results are shown in Figure 15. In these tests, an Al_2O_3 ball was used. The same ball-and-flat track was used at each temperature as the temperature was lowered from 900°C to room temperature. The results showed that both silver and lead oxide provided substantial improvement over the unfilled I601. Although there was a worn groove on the flat, the track was still covered with a lubricating film. However, the nickel tungstate showed no benefit over the unfilled specimen. This is surprising, since NiWO_4 is an effective lubricant at 750°C , yielding a friction coefficient of 0.22. It was noted during the 900°C test that the I601 surfaces "broke up" due to the weakened pitted surface. This was attributed to the high pressure generated by the non-wearing Al_2O_3 ball. Accordingly, a second test was run with a Nickel 200 slider. This test showed slightly lower friction, but still did not achieve the beneficial results hoped for. In this test, the Nickel 200 slider wore considerably during the 900°C test; this may have obliterated the NiWO_4 film.

With the ZnO -filled specimen, a slight lowering of friction was found at 900°C ; however, as the temperature was lowered, failure occurred and friction rose to 0.64. Almost the same result was found for CeO_2 . Again, it appeared as if the surfaces were too weak to support the load.

The results with silver and lead oxide show that the approach will work, but more effort is needed to optimize the system.

6.0 SUMMARY OF RESULTS

Figure 16 is a friction-temperature plot of the best materials developed in this study. It shows that adequate friction behavior can be achieved over a broad temperature range with appropriate choice of materials. These tests were run under very severe conditions of nominal pressure; thus they can apply to a wide range of applications, including the regenerator-crossarm seal.

Figure 15 contains the friction results for two alloys not previously discussed: 80Ni15Cr2Ti3Zr and 73Ni15Cr2Ti10W. These alloys were prepared based on the beneficial results of the binary alloys Ni2W and Ni2Ti and the low friction found with ZrO₂ diffusion in nickel 200. Both of the alloys give low friction at temperatures above 500°C and reasonable values (0.32 to 0.42) at lower temperatures. The important point is that the surfaces were not damaged nor was there metal transfer at low temperatures. With both alloys, the track was oxide-covered with little evidence of metallic contact. The Al₂O₃ ball had a transfer film of oxide.

Although the binary alloys Ni2W and Ni2Ti showed similar friction behavior, they could not be used at 900°C because of strength and oxidation characteristics. The nickel alloys contained chromium so they may be more practical. The important point is that the addition of the chromium did not change the friction-temperature behavior.

A comparison can be made between the results shown in Figure 16 and those for the commercial alloy Inconel 750X which had been preoxidized (Fig. 13). As shown in Table 7, the primary composition is 70Ni15Cr7Fe2.5Ti, which is very similar to the alloys of Figure 16. The primary difference in friction behavior is at low temperatures where Inconel 750X gave more surface damage and metallic contact.

These results demonstrated some success; however, much more work remains before such materials can be used. First of all, wear rates must be determined at high temperatures to see if they approach those required in regenerator applications. If they do not, selective approaches can be taken to improve them. If they do, then additional work will be required to determine the mechanical properties of the modified alloys.

7.0 CONCLUSIONS

A series of friction-temperature tests were run with alloys and surface-modified nickel 200 specimens to determine whether the naturally occurring oxide film could be stabilized to permit effective sliding over a broad temperature range from room temperature to 900°C. Based on these results, the following conclusions were drawn:

1. The addition of certain elements, titanium, tungsten and tantalum, have a beneficial effect when added to nickel and nickel alloys in stabilizing the oxide film and allowing effective sliding over a broader temperature range than found for nickel alone.
2. With alloys 80Ni15Cr2Ti3Zr, 73Ni15Cr2Ti10W, and 70Ni15Cr7Fe2.5Ti, friction coefficients when sliding against Al_2O_3 were 0.33 to 0.44 for temperatures from 100°C to 400°C, and were 0.22 to 0.32 for temperatures from 400°C to 900°C. These friction coefficients and the resulting surface damage are acceptable for many applications, including the regenerator seals.
3. Pitted surfaces filled with silver and lead oxide proved to be equally effective; however, other solid lubricants like ZnO, CeO₂, and NiWO₄ were ineffective. Further work is necessary to optimize this approach.
4. Diffusing oxides into the surfaces proved to be ineffective in stabilizing the oxide film, but did lower

friction at high temperatures with CeO_2 , WO_3 , ZrO_2 and BaO in nickel 200.

5. Further work is necessary to evaluate the implantation approach and to consider the performance of commercial alloys containing the desired elements.

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Fig. 1 Effect of temperature on the coefficient of friction for Inconel 750X sliding against itself. Load 20 N; average velocity 0.76 cm./sec.

Fig. 2 Effect of time on the wear of Inconel 750X sliding against itself at different temperatures. Load 20N; average velocity 0.76 cm./sec.

Fig. 3 Photograph of etched and unetched surfaces of a nickel alloy.

Fig. 4 Schematic diagram of the friction apparatus.

Fig. 5 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against two nickel-titanium alloys. Load 145N; average velocity 5 cm./sec.

Fig. 6 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against two nickel-tantalum alloys. Load 145N; average velocity 5 cm./sec.

Fig. 7 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against two nickel-tungsten alloys. Load 145N; average velocity 5 cm./sec.

Fig. 8 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against a nickel-boron alloy. Load 145N; average velocity 5 cm./sec.

Fig. 9 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against Ni200. Load 145N; average velocity 5 cm./sec.

Fig. 10 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against various binary alloys. Tests run with increasing temperature levels. Load 145N; average velocity 5 cm./sec.

Fig. 11 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against various binary alloys. Tests run with decreasing temperature levels. Load 145N; average velocity 5 cm./sec.

Fig. 12 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against Nickel 200 with various oxide treatments. Tests were run with decreasing temperature levels. Load 50 N; average velocity 5 cm./sec.

Fig. 13 Effect of temperature on the coefficient of friction for Al₂O₃ sliding against Inconel 750X with various oxide treatments. Tests were run with decreasing temperature levels. Load 50 N; average velocity 5 cm./sec.

Fig. 14 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against implanted alloys. Load 145N; average velocity 5 cm./sec.

Fig. 15 Effect of temperature on the coefficient of friction for Al₂O₃ sliding against pitted Inconel 601 surfaces filled with various solid lubricants. Tests were run at decreasing temperature levels. Load 50 N; velocity 5 cm./sec.

Fig. 16 Effect of temperature on the coefficient of friction for Al_2O_3 sliding against various materials. Load 145 N; average velocity 5 cm./sec.

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