# NASA TN D-295



# TECHNICAL NOTE

D-295

HALOGEN-CONTAINING GASES AS LUBRICANTS FOR CRYSTALLIZED-

GLASS-CERAMIC - METAL COMBINATIONS AT

TEMPERATURES TO 1500° F

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HALOGEN-CONTAINING GASES AS LUBRICANTS FOR CRYSTALLIZED-GLASS-CERAMIC -

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#### SUMMARY

Pyroceram 9608 (a crystallized glass ceramic) has been considered for use in high-temperature bearing and seal applications. One of the problems encountered with Pyroceram is the lack of availability of lubricants for the temperature range in which this material becomes practical. Experiments were conducted with Pyroceram sliding on various nickel- and cobalt-base alloys using reactive halogen-containing gases as lubricants. Friction and wear data were obtained as a function of sliding velocity and temperature.

Studies were made with a hemispherical rider (3/16-in. rad., Pyroceram 9608) sliding in a circumferential path on the flat surface of a rotating disk ( $2\frac{1}{2}$ -in. diam., nickel- or cobalt-base alloys). The specimens were run in an atmosphere of the various gases with a load of 1200 grams, a sliding velocity of 3200 feet per minute, and temperatures from 75° to 1500° F.

The gas  $CF_2Br$ - $CF_2Br$  was found to be an effective lubricant for Pyroceram 9608 sliding on Hastelloy R-235 and Inconel X up to 1400° F. The gas  $CF_2Cl$ - $CF_2Cl$  provided effective lubrication for Pyroceram sliding on various cobalt-base alloys at  $1000^\circ$  F.

### INTRODUCTION

High temperatures and corrosive environments require the consideration of some unconventional materials for bearing and seal construction. One novel material that has been considered for such applications is the crystallized glass ceramic Pyroceram (refs. l to 3). It has good high-temperature physical and chemical properties. It has the distinct advantage over tool steels (used in bearing construction) of retaining its mechanical properties over a broad temperature range, whereas the tool steels lose hardness and oxidize readily at elevated temperatures (above  $1000^{\circ}$  F). Further, Pyroceram possesses the high-temperature corrosion resistance characteristic of ceramic materials.

An anticipated problem in the use of Pyroceram as a bearing material is the lack of lubricants for the temperature range at which the use of Pyroceram might be practical (above  $1000^{\circ}$  F). Lubrication with reactive gases such as symmetrical dibromotetrafluoroethane (CF<sub>2</sub>Br-CF<sub>2</sub>Br) or dichlorotetrafluoroethane (CF<sub>2</sub>Cl-CF<sub>2</sub>Cl) might be employed in systems involving the use of Pyroceram.

The halogen-containing gases CF<sub>2</sub>Cl-CF<sub>2</sub>Cl and CF<sub>2</sub>Br-CF<sub>2</sub>Br possess very stable molecular structures at elevated temperatures and have provided effective lubrication at these temperatures with various material combinations (ref. 4). The mechanism underlying reactive-gas lubrication is fundamentally the same as that in extreme-pressure lubrication. Halogen-containing gas molecules are normally stable in contact with metal surfaces at ambient temperatures of 1000° F. In lubrication systems where materials are in sliding contact, however, the frictional heat generated at contacting metal asperities (ref. 5) is sufficient to cause localized decomposition of gas molecules. This molecular decomposition results in liberation of free halogen (chlorine or bromine) atoms, which react with the hot metal surface to form metal halides. The metal halides with their associated low-shear-strength properties then function as solid lubricants. In gas-lubricated systems, at least one of the surfaces in sliding contact should therefore be a metal.

In general, cobalt-base alloys (e.g., Stellite Star J and Rexalloy 33) are effectively lubricated by chlorine-containing gases, such as CF<sub>2</sub>Cl<sub>2</sub> and CF<sub>2</sub>Cl-CF<sub>2</sub>Cl. The reaction of the cobalt-base alloys with the chlorine-containing gas generally results in the formation of cobalt chloride (CoCl<sub>2</sub>) on the metal surface as well as chlorides of other metals present in the alloy. Cobalt chloride, because of its low shear strength, acts as a solid lubricant. The nickel-base alloys (e.g., Hastelloy C, Hastelloy R-235, and Inconel X) are in general most effectively lubricated by the bromine-containing gases (e.g., CF<sub>3</sub>Br, CF<sub>2</sub>Br<sub>2</sub>, and CF<sub>2</sub>Br-CF<sub>2</sub>Br). The reaction of the nickel-base alloy with the bromine-containing gases generally results in the formation of nickel bromide (NiBr<sub>2</sub>) on the metal surface as well as bromides of other metals present in the alloy. Nickel bromide has low-shear-strength properties similar to cobalt chloride and also functions as a solid lubricant.

The object of this investigation was to study the lubricating properties of the gases  $\mathrm{CF_2Cl}$ - $\mathrm{CF_2Cl}$  and  $\mathrm{CF_2Br}$ - $\mathrm{CF_2Br}$  with Pyroceram 9608 sliding on various nickel- and cobalt-base alloys at temperatures from 75° to 1500° F. In friction and wear experiments, a 3/16-inch-radius (Pyroceram 9608) hemisphere contacted the flat surface of a rotating  $2\frac{1}{2}$ -inch disk (of nickel- or cobalt-base alloy). The load employed was 1200 grams, and sliding velocities were from 75 to 8000 feet per minute. Friction, wear, and corrosion characteristics were noted.

#### MATERIALS

The gases used in this investigation were symmetrical dichlorotetra-fluoroethane ( $CF_2Cl-CF_2Cl$ ) and symmetrical dibromotetra-fluoroethane ( $CF_2Br-CF_2Br$ ). These gases were selected because of (1) good lubricating properties over a broad temperature range, and (2) high chemical and thermal stability.

The material used for the rider specimens in all experiments was the crystallized glass ceramic Pyroceram 9608. (This material has a Knoop hardness of 588, which is equivalent to a Rockwell hardness of C-53.) The disk specimens were various nickel and cobalt alloys. The composition and hardness of each of the disk materials are given in the following table:

Material					Compo	sitio	n, p	ercen	t				Room tem- perature
	Fe	Ni	Co	Cr	С	Si	Mn	Мо	W	Ti	Nb	Other	hardness, Rockwell
Stellite Star J	3.0	2.5	43.0	31.0	2.4	1.0			17.0				57
Stellite 98M2	3.0	3.5	40.0	30.5	2.0	1.0			18.5			B, V	52 to 57
Stellite 21	.7	2.8	62.2	27.4	.22	.53	$\operatorname{Tr}^{\mathbf{a}}$	1	5.5				30
Rexalloy 33	2.0		44.0	33.0	2.25	.75			17.0				60
70WC-30Co			30.0		4.3				65.7	İ			
Hastelloy R-235	10.0	66.0	2.5	14.0 to	.16	1.0	Tr	5.5		Tr		Al	28
Hastelloy C	6.0	52.0		17.0		Tr	Tr	19.0	5			ļ	23
Inconel X	5 <b>to</b> 9	70.0		15.0	.08	.50	Tr		1		Tr	Al,S	29
$7\frac{1}{2}\%$ Si-Ni		92.0				<b>7.</b> 5							33
K-175B		41.0			7.2			15.0		28.0	4.5		

a<sub>Trace</sub>.

## APPARATUS AND PROCEDURE

The apparatus used in this investigation is shown schematically in figure 1. The basic elements of the apparatus consist of a rotating disk specimen ( $2\frac{1}{2}$ -in. diam.) and a hemispherically tipped rider specimen (3/16-in. rad.).

The rider specimen is stationary and in sliding contact with the rotating disk. The disk specimen is rotated on a drive shaft by means of a variable-drive motor unit through a gear box and spindle assembly. The drive shaft enters the housing of the apparatus through an interlocking labyrinth seal. The portion of the drive shaft inside the apparatus housing contains a heat shield and the disk specimen which is held on the shaft by a lock nut. A magnetic speed pickup is used to

monitor rotative speeds. The disk specimens were rotated at angular velocities that produced surface speeds of 75 to 8000 contact feet per minute.

The rider specimen is loaded against the disk surface by means of a retaining arm, which is gimbal-mounted and bellows-sealed to the apparatus. The load is applied to the arm by means of dead weights. At a right angle to the cable is a linkage connecting the arm with a strain-gage assembly for measuring frictional force.

The test specimens are heated by 12 650-watt cartridge heaters located in an Inconel housing that fits around the circumferential surface of the disk specimen. The heaters are controlled by a Variac unit and a temperature controller. The controller is operated by a thermocouple located near the point of disk and rider contact. The apparatus was operated at temperatures from  $75^{\circ}$  to  $1500^{\circ}$  F.

The experimental gaseous lubricants were supplied by means of an Inconel tube at a rate of 1.0 liter per minute to an Inconel test chamber (0.7-liter volume) which housed the specimens and heater assembly. A gas exhaust tube was used to remove effluent gases. Prior to each run a 15-minute purge period was employed to expel air from the chamber. A face plate containing a quartz window for experimental observation was bolted to the outer apparatus housing.

The disk and rider specimen used in the investigation were surface ground to 4 to 8 microinches. Before each experiment the disk and rider were given the same preparatory treatment: (1) a thorough rinsing with acetone to remove oil and grease, (2) polishing with moist levigated alumina on a soft cloth, (3) a thorough rinsing in tap water followed by distilled water, (4) a thorough rinsing of the specimens with absolute ethyl alcohol to remove the water. The specimens were then stored in a desiccator until used.

As a result of some previous work with  $\mathrm{CF}_2\mathrm{Cl}_2$ , a run-in procedure was found necessary under all experimental test conditions in this study. The results obtained with  $\mathrm{CF}_2\mathrm{Cl}_2$  showed that, if the run was started with high loads and speeds, surface failure of the specimen was apt to occur. High initial friction and wear can sometimes be attributed to lack of sufficient time for the formation of a reaction film. By use of the run-in procedure, it was found that a reaction film could form that markedly reduced the initial high friction and wear. The run-in procedure included the following steps: an initial load of 650 grams for a period of 1 minute, 850 and 1050 grams for intervals of 1 minute, and finally the full 1200-gram load.

#### RESULTS AND DISCUSSION

The results obtained in friction and wear experiments conducted at  $1000^{\circ}$  F with Pyroceram 9608 sliding on various cobalt-base alloys and a cobalt bonded cermet in air and with the gaseous lubricant CF<sub>2</sub>Cl-CF<sub>2</sub>Cl are presented in figure 2. The friction coefficient with CF<sub>2</sub>Cl-CF<sub>2</sub>Cl varied from values appreciably lower than obtained in air (Pyroceram on Stellite Star J) to values which were actually higher than those obtained in air (Pyroceram on Stellite 21). The wear of Pyroceram was considerably lower in CF<sub>2</sub>Cl-CF<sub>2</sub>Cl than in air with all alloy compositions.

The friction and wear results obtained in the preliminary experiments of figure 2 indicated that Pyroceram sliding on Stellite Star J might be a good combination for lubrication with CF2Cl-CF2Cl. This particular combination was therefore run at temperatures from  $75^{\circ}$  to  $1400^{\circ}$ F to determine the influence of CF2Cl-CF2Cl on the friction and wear properties of Pyrocerum sliding on Stellite Star J over a broad temperature range. The results obtained in these experiments are presented in figure 3. The data indicate considerable scatter for both friction and wear over the temperature range explored. The variation in friction coefficient over the temperature range is represented by a band (variation of approx. 0.1). The relatively high friction and wear values as well as the data scatter shown in figure 3 indicated that CF2Cl-CF2Cl was not a good lubricant for Pyroceram 9608 sliding on Stellite Star J. The results for Stellite Star J do not necessarily indicate that Pyroceram 9608 in combination with other cobalt-base alloys would not be effectively lubricated by chlorine-containing gases.

Since bromine-containing gases have been shown to provide good lubrication for nickel-base alloys (ref. 4), friction and wear experiments were made with Pyroceram 9608 sliding on various nickel-base alloys at  $1000^{\circ}$  F in air and with the gaseous lubricant CF<sub>2</sub>Br-CF<sub>2</sub>Br. The results of these experiments are presented in figure 4. In every combination of Pyroceram 9608 with a nickel-base alloy presented in figure 4, a considerable improvement in friction and wear was obtained using CF<sub>2</sub>Br-CF<sub>2</sub>Br as a lubricant over similar runs made in air. The results indicate the effectiveness of bromide films in reducing friction and wear.

The results presented in figure 4 show only the influence of  $CF_2Br$ - $CF_2Br$  on the friction and wear of Pyroceram 9608 against nickel-base alloys at  $1000^{\circ}$  F. In order to explore the influence of  $CF_2Br$ - $CF_2Br$  on the friction and wear properties of Pyroceram sliding on a nickel-base alloy over a broad temperature range, experiments were conducted from  $75^{\circ}$  to  $1400^{\circ}$  F. The results obtained with Pyroceram 9608 sliding on Hastelloy R-235 in a gaseous environment of  $CF_2Br$ - $CF_2Br$  up to  $1400^{\circ}$  F are presented in figure 5. The friction coefficient was low, and

effective boundary lubrication was obtained over the greater portion of the temperature range. The wear of the Pyroceram rider was low at temperatures below  $1000^{\circ}$  F. Above  $1000^{\circ}$  F, the rider wear increased markedly. Although wear in these experiments was measured on the rider specimen, the wear of the disk specimen (as qualitatively indicated by surface profile tracings) was also extremely low. In experiments run in air and with  $CF_2Br-CF_2Br$  the Pyroceram seemed to do little more than polish the disk surface.

To explore the influence of sliding velocity on the wear of Pyroceram, the sliding velocity employed in figure 5 was doubled to 6400 feet per minute and the wear on Pyroceram was measured over the temperature range  $75^{\rm O}$  to  $1400^{\rm O}$  F. The results obtained, together with the wear values at 3200 feet per minute from figure 5, are presented in figure 6. It is of interest to note that doubling the sliding velocity increased the wear of Pyroceram by a factor of better than ten. The friction coefficient at 6400 feet per minute was, however, nearly the same as obtained at 3200 feet per minute. The upward trend of the wear curve obtained in figure 5 at 3200 feet per minute reoccurred at 6400 feet per minute and in the same temperature region. With metal-metal combinations sliding in halogen-containing gaseous lubricants, similar wear trends were observed (ref. 4). The increase in the metallic wear of the reference studies was attributed to corrosive wear from excessive surface reactivity. Since, in general, increasing sliding velocity increases surface temperature and ultimately surface activity, the upward trend of the wear curve at 6400 feet per minute should occur at a lower ambient temperature. The data of figure 6 indicate that the increasing wear trend of Pyroceram at elevated temperatures seems to be independent of surface activity or corrosive wear. The Pyroceram used in these experiments represents one of the first commercial formulations of crystallized glass ceramics. The development of this type material is in its infancy, and it is expected that subsequent formulations will have improved mechanical properties at high temperatures.

Experiments were conducted to explore the influence of sliding velocity on friction coefficient for Pyroceram sliding against Hastelloy R-235 in CF<sub>2</sub>Br-CF<sub>2</sub>Br. The results obtained for three ambient temperatures,  $75^{\circ}$ ,  $600^{\circ}$ , and  $1000^{\circ}$  F, are presented in figure 7. The friction coefficients indicate the effectiveness of CF<sub>2</sub>Br-CF<sub>2</sub>Br in providing protective surface films over a range of sliding velocities at the three temperatures. In general very little difference exists in friction values obtained at  $600^{\circ}$  and  $1000^{\circ}$  F.

The increase in wear at elevated temperatures (above  $1000^{\circ}$  F) observed in figures 3, 5, and 6 with gas lubrication prompted experiments with Pyroceram in air to determine whether the increase at these

temperatures was due to corrosive wear or simply an inherent characteristic of the Pyroceram. The results of experiments with Pyroceram sliding on Hastelloy R-235 in air up to  $1500^{\circ}$  F are presented in figure 8. The wear gradually decreases from  $75^{\circ}$  to  $800^{\circ}$  F with increasing oxide formation. The wear is relatively constant to  $1200^{\circ}$  F, where it begins to increase. The result is the same as observed with gas lubrication in figures 3, 5, and 6. The wear trends observed at elevated temperatures for Pyroceram in air seem to indicate that the values or trends observed with gas lubrication did not result from corrosive wear.

Comparison of the wear data of figure 8 with those of figure 5 shows that, in air, wear was seventy times greater at  $75^{\circ}$  F and seven times greater at  $1400^{\circ}$  F than that obtained in  $CF_2Br-CF_2Br$ . These data were obtained with Pyroceram riders on Hastelloy R-235 disks. The relative differences in wear data obtained in air and  $CF_2Br-CF_2Br$  at  $1400^{\circ}$  F are appreciably less than those obtained at  $75^{\circ}$  F. The reasons for the differences in the wear curves are not known. Reference 6 indicates some evidence of deterioration in mechanical properties (e.g., Young's modulus) at temperatures above  $1300^{\circ}$  F. It is quite possible that a relation exists between the wear results obtained and the change in the mechanical properties of Pyroceram 9608 above  $1300^{\circ}$  F. The relative differences in wear of Pyroceram riders run at  $1000^{\circ}$  F in air and  $CF_2Br-CF_2Br$  can be seen in the photomicrographs presented in figure 9.

Friction and wear experiments were also made with Pyroceram 9608 sliding on Inconel X over the temperature range  $75^{\circ}$  to  $1500^{\circ}$  F in an atmosphere of  $CF_2Br-CF_2Br$ . The results of these runs can be seen in figure 10. The coefficient of friction was 0.1 or less from  $75^{\circ}$  to  $1400^{\circ}$  F and only slightly higher at  $1500^{\circ}$  F. These values are within the region obtained with some oils in effective boundary lubrication. The rider wear was extremely low over the entire temperature range from  $75^{\circ}$  to  $1400^{\circ}$  F. At  $1500^{\circ}$  F the wear increased, as was expected from the results obtained in figure 8. The rider and disk specimen of a  $1200^{\circ}$  F run in  $CF_2Br-CF_2Br$  are shown in figure 11.

# SUMMARY OF RESULTS

The experimental results obtained with Pyroceram 9608 sliding on nickel- and cobalt-base alloys with  $CF_2Cl-CF_2Cl$  and  $CF_2Br-CF_2Br$  as lubricants can be summarized as follows:

l. The material combinations of Pyroceram with Hastelloy R-235 and Inconel X were effectively lubricated with the bromine-containing gas  $CF_2Br-CF_2Br$  at temperatures to  $1400^\circ$  F.

- 2. Pyroceram in combination with various alloys was effectively lubricated by both halogen-containing gases at temperatures from  $75^{\circ}$  to  $1200^{\circ}$  F. At temperatures higher than  $1200^{\circ}$  F a trend of increasing wear with temperature was observed.
- 3. The material combination of Pyroceram with Stellite Star J in a chlorine-containing atmosphere (CF<sub>2</sub>Cl-CF<sub>2</sub>Cl) was not effectively lubricated. Although friction and wear properties improved over runs made in air, the values were still higher than normally associated with effective boundary lubrication, and the data over a broad temperature range were erratic. These results do not necessarily indicate that chlorine-containing gases would not lubricate Pyroceram sliding on cobalt-base alloys other than Stellite Star J.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, August 1, 1960

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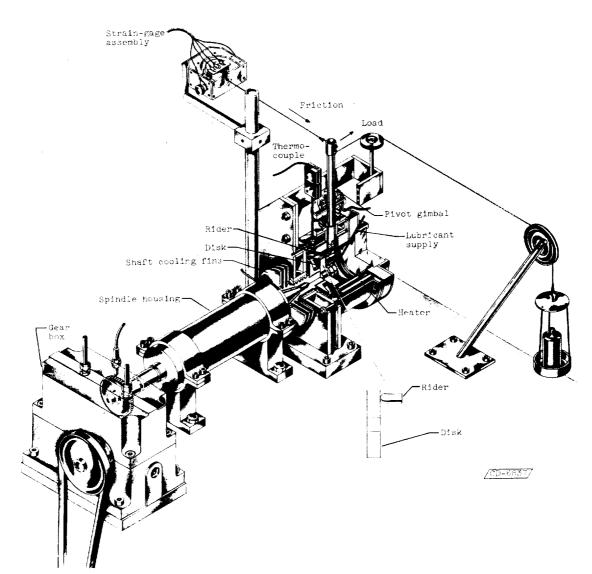


Figure 1. - High-temperature friction apparatus.

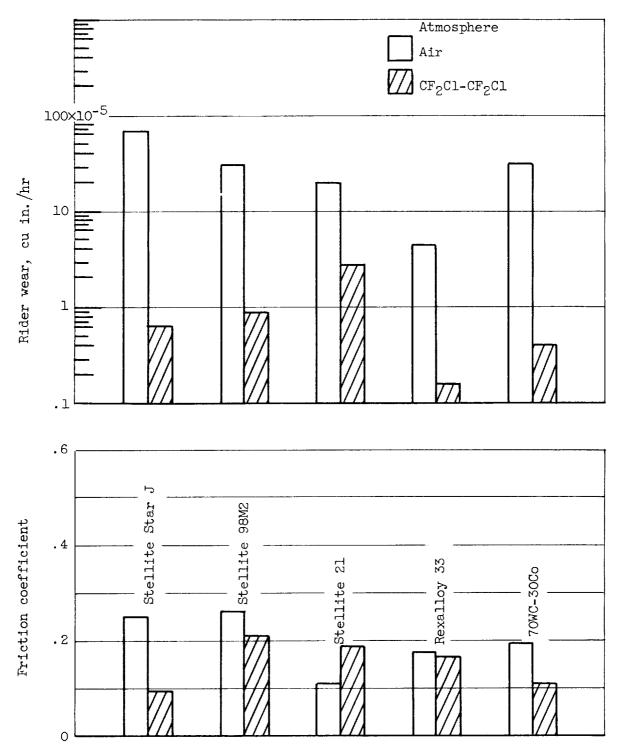
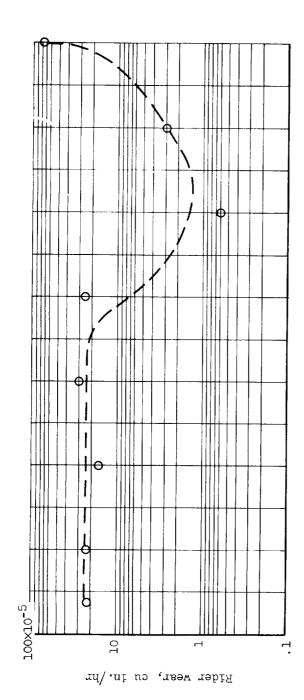


Figure 2. - Friction and wear of Pyroceram 9608 sliding on various cobalt-base alloys at 1000° F. Sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour.

CX-2 back



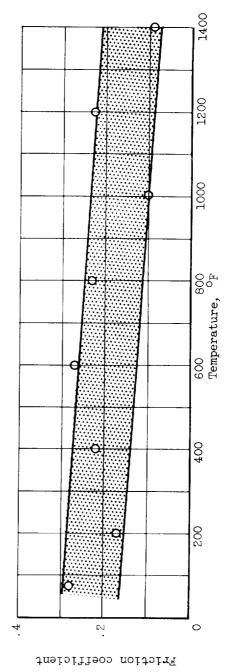


Figure 3. - Friction and wear of Pyroceram 9608 sliding on Stellite Star J at various temperatures. Atmosphere, CF2Cl-CF2Cl; sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour.

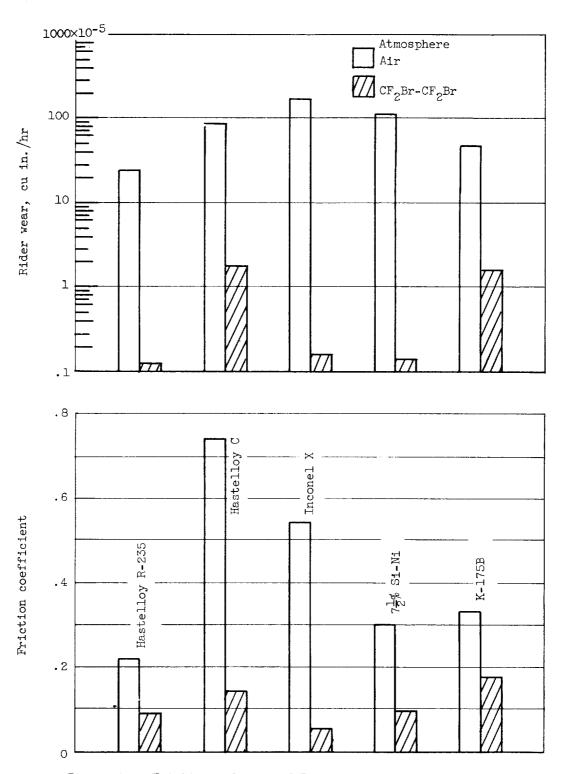
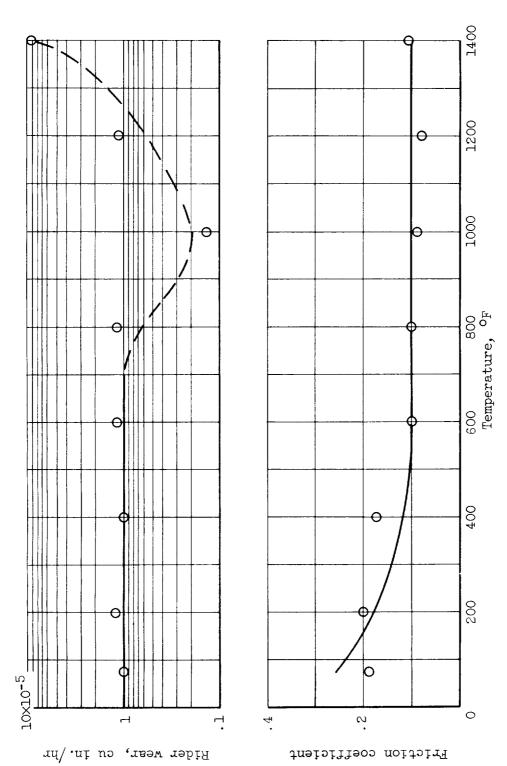


Figure 4. - Friction and wear of Pyroceram 9608 sliding on various nickel-base alloys at 1000° F. Sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour.



temperatures. Atmosphere,  $\mathrm{CF}_2\mathrm{Br}\text{-}\mathrm{CF}_2\mathrm{Br}$ ; sliding velocity, 3200 feet per minute; load, Figure 5. - Friction and wear of Pyroceram 9608 sliding on Hastelloy R-235 at various 1200 grams; duration, 1 hour.

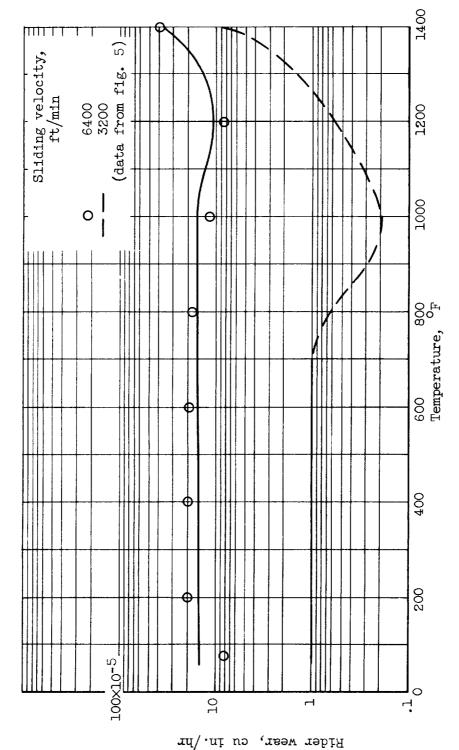


Figure 6. - Wear of Pyroceram 9608 rider sliding on Hastelloy R-235 disk at various temperatures and sliding velocities. Atmosphere, CF2Br-CF2Br; load, 1200 grams; duration, 1 hour.

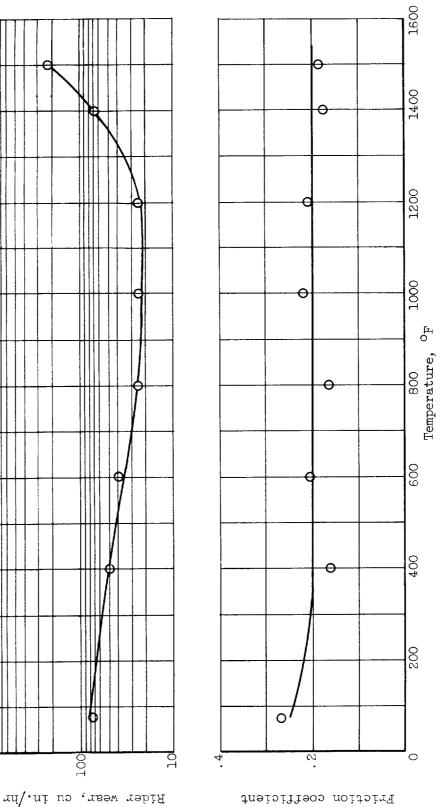
Friction coefficient

Figure 7. - Friction coefficient of Pyroceram 9608 rider sliding on Hastelloy R-235 disk at various temperatures and sliding velocities. Atmosphere,  ${\rm CF_2Br}$ - ${\rm CF_2Br}$ ; load, 1200 grams; duration, 1 hour.

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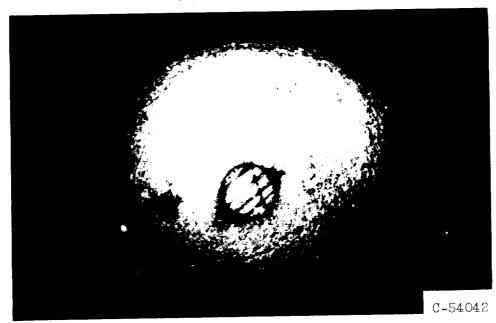
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- Friction and wear of Pyroceram 9608 sliding on Hastelloy R-235 at various temperatures Sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour. Figure 8. in air.

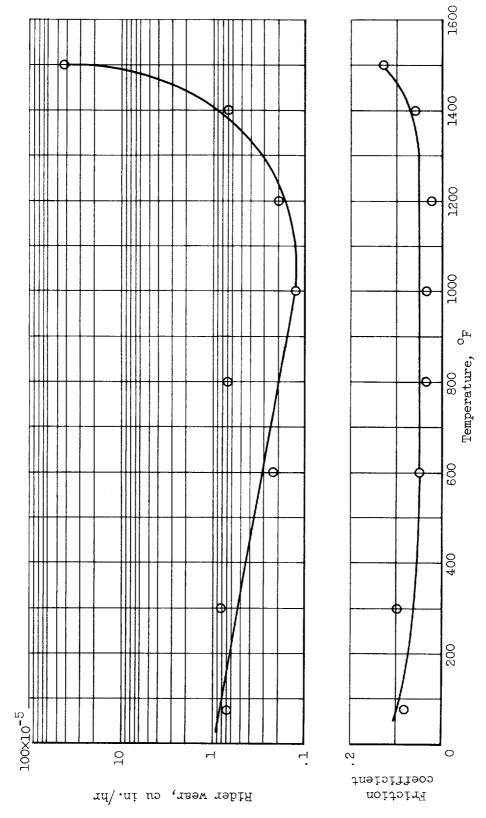


(a) Air atmosphere.

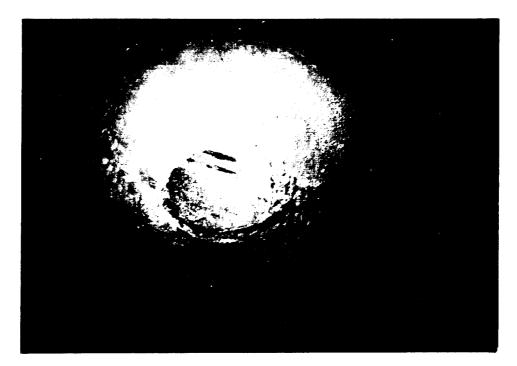


(b)  $CF_2Br-CF_2Br$  atmosphere.

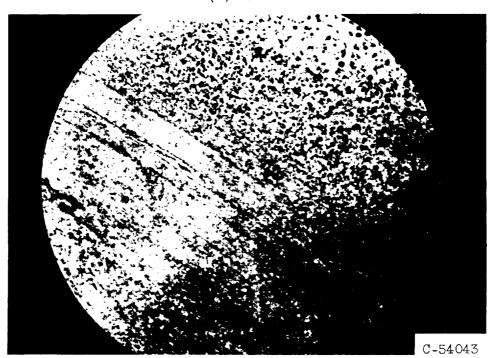
Figure 9. - Photomicrographs of wear areas on Pyroceram 9608 rider specimens. Disk specimen, Hastelloy R-235; sliding velocity, 3200 feet per minute; load, 1200 grams; duration, l hour; temperature, 1000° F. X15.



Atmosphere, CF2Br-CF2Br; sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour. Figure 10. - Friction and wear of Pyroceram 9608 sliding on Inconel X at various temperatures.



(a) Rider.



(b) Disk.

Figure 11. - Photomicrographs of Pyroceram 9608 rider and Inconel X disk specimens. Atmosphere, CF<sub>2</sub>Br-CF<sub>2</sub>Br; sliding velocity, 3200 feet per minute; load, 1200 grams; duration, 1 hour; temperature, 1200° F. X15.

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NASA TN D-295  National Aeronautics and Space Administration.  HALOGEN-CONTAINING GASES AS LUBRICANTS FOR CRYSTALLIZED-GLASS-CERAMIC - METAL COMBINATIONS AT TEMPERATURES TO 1500° F.  Donald H. Buckley and Robert L. Johnson. October 1960. 19p. OTS price, \$0.50.  (NASA TECHNICAL NOTE D-295)	The gases CF2C1-CF2C1 and CF2Br-CF2Br were used to provide lubrication for Pyroceram 9608 sliding on various nickel- and cobalt-base alloys. The use of Pyroceram 9608 minimized the corrosive wear encountered with metal-metal combinations using halogen-containing gases reported in earlier research. In the friction and wear experiments a hemispherically tipped rider (Pyroceram 9608) under a 1200-gram load slid on a disk (nickel- or cobalt-base alloy) rotating at 3200 ft/min. The gas CF2Br-CF2Br was an effective lubricating agent for Pyroceram 9608 sliding on Hastelloy R-235 and Inconel X at temperatophers obtainable from NASA, Washington (over)	NASA TN D-295  National Aeronautics and Space Administration.  HALOGEN-CONTAINING GASES AS LUBRICANTS FOR CRYSTALLIZED-GLASS-CERAMIC - METAL COMBINATIONS AT TEMPERATURES TO 1500° F. Donald H. Buckley and Robert L. Johnson. October 1960. 19p. OTS price, \$0.50. (NASA TECHNICAL NOTE D-295)	The gases CF2CI-CF2CI and CF2Br-CF2Br were used to provide lubrication for Pyroceram 9608 sliding on various nickel- and cobalt-base alloys. The use of Pyroceram 9608 minimized the corrosive wear encountered with metal-metal combinations using halogen-containing gases reported in earlier research. In the friction and wear experiments a hemispherically tipped rider (Pyroceram 9608) under a 1200-gram load slid on a disk (nickel- or cobalt-base alloy) rotating at 3200 ft/min. The gas CF2Br-CF2Br was an effective lubricating agent for Pyroceram 9608 sliding on Hastelloy R-235 and Inconel X at tempera-	Copies obtainable from NASA, Washington (OVer)
I. Buckley, Donald H. II. Johnson, Robert L. III. NASA TN D-295 (Initial NASA distribution: 25, Materials, engineering; 26, Materials, other.)	MASA	I. Buckley, Donald H. II. Johnson, Robert L. III. NASA TN D-295 (Initial NASA distribution: 25, Materials, engineering; 26, Materials, other.)		NASA
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