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PROPERTIES OF EXTRUDED PS-212 TYPE SELF-LUBRICATING MATERIALS

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ABSTRACT

Research has been underway at the NASA Lewis Research Center since the 1960's to develop high temperature, self-lubricating materials. The bulk of the research has been done "in-house" by a team of researchers from the Materials Division.

A series of self-lubricating solid material systems has been developed over the years. One of the most promising is the composite material system referred to as PS-212 or PM-212. This material is a powder metallurgy product composed of metal bonded chromium carbide and two solid lubricating materials known to be self-lubricating over a wide temperature range.

NASA feels this material has a wide potential in industrial applications. Simplified processing of this material will enhance its commercial potential. Processing changes have the potential to reduce processing costs, but these must not adversely effect tribological and physical properties.

Extrusion processing has been employed in this investigation as a consolidation process for PM-212/PS-212. It has been successful in that high density bars of EX-212 (extruded PM-212) can readily be fabricated. Friction and strength data indicate these properties have been maintained or improved over the P.M. version. A range of extrusion temperatures have been investigated and tensile, friction, wear and microstructural data have been obtained. Results indicate extrusion temperatures are not critical from a densification standpoint, but other properties are temperature dependent.

INTRODUCTION

As advances in engine technology have proceeded over the years, a corresponding need has been generated to develop lubricating systems capable of performing in extreme temperature environments. These temperatures exceed the useful range of normal lubricating systems. Liquid and dry lubricants cannot operate at temperatures where the organic components break down or the solids oxidize. New methods include gas bearings and self-lubricating/load bearing composites such materials are needed to fulfill the promise of improved high temperature system performance.

A series of self-lubricating composite materials, unique in their chemistries, has been developed at NASA Lewis. The latest and most promising in this series is referred to as PS-212 or PM-212. This material is composed of powders of chromium carbide with a nickel base alloy binder combined with silver and eutectic fluorides. Both PS-212 and PM-212 have the same composition, only fabrication methods differ. The first version was PS-212, a plasma sprayed material that is described in detail in refs. 1, 2, 3, and 4. Plasma spraying of the material, while relatively simple, has some inherent drawbacks including the uneven buildup of the sprayed surface, compositional variation, entrapped porosity and oxidation, and the inability to coat many internal surfaces. PM-212 was the next version and it was developed using standard powder metallurgy techniques including cold or hot pressing, cold isostactic pressing and sintering or hot isostatic pressing. This data is reported in refs. 5 and 6. Reference 5 deals with powder metal processing and resultant tribological properties. In ref. 6 strength properties of PM-212 are reported for various processing conditions. Thermal conductivity and thermal expansion are also included in this reference.

A new material must be cost effective to be accepted as a viable commercial product. Raw material costs and processing are major considerations in this acceptance process. Extrusion has the potential for reduced processing costs and is a commonly accepted processing procedure.

Optimum extruded properties should only be expected if the variables are optimized. These include: (1) starting powder sizes and distribution, (2) extrusion temperatures, (3) reduction ratio and extrusion speed. This investigation was limited and only looked at varying extrusion temperatures over a range from 1400°F to 1800°F. The results reported are therefore not necessarily optimum for EX-212 .

The advantages for using extrusion include the following: Commercial extrusion is done over a range of temperatures and is used on a variety of materials including both metallics and non-metallics. The extrusion process both consolidates and shapes. Shapes and sizes can vary greatly depending upon tooling. Resultant products are consistent in properties and reduced in cost.

MATERIALS AND PROCEDURE

PS-212, PM-212 and EX-212 are three process versions of the same basic material. They are composed of 70% of a metal bonded, chromium carbide powder (i.e. Metco 430 NS), 15% silver, and 15% barium fluoride/calcium fluoride. The chemical compositions of these components are listed in Table I. Metco 430 NS powder gives the hardness, wear resistance and load bearing capacity while silver lubricates from cryogenic temperatures to 900°F and the fluorides lubricate from 900°F to 1600°F.

MATERIAL PREPARATION

All components were obtained from a commercial source that processed and blended the powders under the direction of NASA personnel. As indicated in Table I, Metco 430 NS powder is from -200 to +400 mesh, silver powder is -100 to +325 mesh and the eutectic fluorides are -200 to +325 mesh. These powders are mixed in a "V-blender" for 45 minutes.

The blended powders are processed in air and are gravity filled into the extrusion cans. These cans are fabricated from mild steel and have a finish size of 3" O.D. x 2" I.D. x 6" long. After filling the cans, the contents are degassed prior to welding on the top. After sealing the cans were furnace heated for one hour at the extrusion temperature and then rapidly transferred to the extrusion press. Area reductions for the extrusions were held constant at 16:1. This resulted in an EX-212 bar diameter of about .4 inch and a length of 5 feet. The extrusions were done in a 1020 ton vertical press. Maximum punch pressure is less than 190,000 PSI. Extrusion temperatures were 1400°F, 1500°F, 1600°F, 1700°F, and 1800°F.

After extrusion, the bars were inspected by both x-ray and surface fluorescence (Zyglo) and then fabricated into test samples. Two types of test samples were used in this investigation. They are shown in fig. 1 and include tensile test bars (ref. 7) and friction and wear bars. Test specimens were fabricated by diamond grinding for the friction wear bars and by silicon carbide grinding for the tensile bars. Both methods worked, but the diamond ground surface was smoother.

TENSILE AND TENSILE TESTING

Tensile tests were run at room temperature, 800°F, 1200°F, 1400°F and 1600°F. They were run, in most cases, as single data points. Tests were run in a controlled strain rate test machine and temperatures were measured with thermocouples attached to the test section.

Friction and wear specimens were scrubbed with an alumina/water paste and rinsed with deionized water prior to testing. These specimens were slid against a Rene '41 (nickel base alloy) counterface disk. Testing was done in a pin-on-disk tribometer. The hemispherically tipped (3/16" radius of curvature) specimen (pin) was loaded against the flat surface of the rotating Rene '41 disk. Sliding speed was 9 ft/sec with a deadweight load of 1.1 lb. Tests were run in an air atmosphere (relative humidity 35% at room temperature) at 70°F, 660°F and 1400°F.

Nine double-ended EX-212 specimens were tested for a total of 36 half hour tests by running two tests on each pin end. Of the nine specimens, three each were from the 1400°F, 1600°F and 1800°F extrusions. The Rene '41 disks were heated by induction and were generally run at 1000 rpm (9.2 ft/sec.). (A velocity survey was conducted by running successive five minute tests at 10, 100, 500, 1000, 2000, and 3500 rpm.)

After each half-hour test, wear measurements were made. The pin tips were photomicrographed to determine wear scar diameters. A surface profile meter was used to measure the cross sectional area of the disk wear track. These measurements were used to calculate wear volumes and wear factors for the pins and disks.

Microstructures of the extruded EX-212 material were examined in both the etched and the unetched conditions. All microstructural examination was done with optical microscopes on longitudional sections. It should be noted that optical viewing shows the fluorides as black, due to their low reflectivity. They should not be mistaken for voids which have no reflectivity.

HARDNESS

Hardness tests were run on an automatic readout Rockwell Hardness Tester. All tests were run on cross sectional segments cut from the extrusions. Tests were run on the R "C" scale and are listed in Table IV.

RESULTS AND DISCUSSION

Extrusions of PS-212 blended powder were run at temperatures of 1400°F, 1500°F, 1600°F, 1700°F and 1800°F. This was done to determine the feasibility ofof using a low cost commercial process to consolidate the composite material into near net shape. Starting powder sizes were those used in the earlier studies of PS/PM-212. Reduction ratios were similar to those used in earlier studies with high temperature alloy systems. (Ref. 7)

All of the extrusions were successful in that they did consolidate into a bar configuration. X-rays of the bars indicated a relatively uniform, continuous extrusion with no bursts or discontinuities. Test samples were examined by X-ray and Zyglo and found to be generally porous. The porosity was the greatest in the lower temperature extrusions. Less porosity was evident as extrusion temperature increased and was a minimum at 1800°F.

TENSILE

Tensile data is shown in Table II and plotted in fig. 2. Generally the lower temperature extrusions had better strength at test temperatures from room to 1200°F and the higher temperature extrusions had better strengths at 1400°F and 1600°F. Elongation in all cases were less than 3%. The low temperature extrusions had the least ductility as measured by tensile elongation. The brittle fluoride eutetic encapsulates the metallic phases and limit both the strength and ductility. Both the strengths and the ductility of the EX-212 compare favorably with the PM-212. It should also be noted that refinements in the particular size of the components may have a strenghtening effect on the resultant extrusion, especially at the lower extrusion temperatures.

FRICTION AND WEAR

The friction and wear results are shown in Table III and figs. 3-8. The friction and wear behavior for the EX-212 samples are shown to be similar to results previously found for sintered PM-212.

The friction values for the EX-212 at a sliding velocity of 9.2 ft/sec ranges from a low of 0.26 at 1400°F for the 1400°F extrusion to a high of 0.50 at 660°F for the 1600°F extrusion. The pin wear factors for all the extruded tests were determined to fall within the moderate to low range within the 1400°F extrusion showing the least wear at 1400°F. The disk wear factors were found to fall within the moderate to low range for all the extruded tests except two. The wear factor for the 1600°F and 1800°F extrusions were negative, denoting material transfer from pin to disk. This transfer was reflected in the wear factors of the corresponding pins, which had the two highest pin wear volumes of the extruded tests.

In general for the EX-212, increasing the disk speed was found to decrease the friction coefficient, with less of an effect at higher temperatures. This behavior was also displayed by the sintered PM-212 previously studied.

Overall the EX-212 samples were determined to display friction and wear behavior to sintered PM-212. Varying the extrusion temperature was found to have little effect on the tribobehavior of EX-212, although the 1400°F extrusion has the best overall performance.

MICROSTRUCTURE

Fig. 9 shows 25X unetched photomicrographs of all extrusion temperature bars. Stringers appear most evident in the lower temperature bars. Some voids are present in all bars but again they are the most evident in the low temperature extrusions. Fig. 10 shows the etched bars at 100X. Particles are deformed and elongated (cold worked) in the 1400°F extruded bars. This deformation decreases as the extrusion temperature is increased. At 1800°F the extruded microstructure is equiaxed. Fig. 11 shows the etched microstructure at 1000X. The 1400°F microstructure shows three major components. Fluorides are shown as dark phases, the white phase is silver and the grey phase is the Metco 430NS. Note that there are very thin bands of silver between grey phases. As extrusion temperature is increased, a second phase appears in the matrix of the Metco 430NS. The carbides remain clear. Silver appears to coalesce as the extrusion temperature increases.

It should be emphasized that the interconnected dark grey areas are fluorides and not voids. Density measurements of the extrusions indicate near theoretical density in all extrusions.

HARDNESS

The hardness tests were taken on the Rockwell "C" scale and hence represent an average hardness over a range of phases. Micro hardness studies might be appropriate for further studies on the effect of extrusion parameters. The hardness values obtained are listed in Table IV and generally indicate an increasing hardness with increasing extrusion temperatures. This is unexpected since lower temperature working conditions would be expected to result in increased work hardening and hardness. One explanation might be the increased evidence of carbide growth in the higher temperature extrusions and the development of the second phase precipitate in the nickel matrix.

CONCLUSIONS

The PS-212 chemistry and the components lend themselves to fabrication by the extrusion process. Large reduction ratios (16:1) of the canned powders result in solid bars with minimal porosity. Component particle size is probably a factor in resulting physical properties. Properties of the extruded EX-212 compare favorably with the prior versions of the PM-212.

Extrusion offers a low cost process to fabricate near net shape components for bearing applications. Further work might be done to evaluate finer particulate sizes and their effect on resultant physical properties.

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7. Waters, W. J. and Freche, J. C.: Strength Enhancement Process for Prealloyed Powder Superalloys, NASA TM 78834.

TABLE I. - COMPONENTS OF EX-212

Component	Composition, wt %	Composition, vol %	Particle size U.S. Sieve No. (μm)	MP[1] °C	Hardness, Hv kg/mm^2
Component A: Bonded Chromium Carbide: 70 wt % of PM212					
Cr_3C_2	45	47	-200 + 400 (35 to 74)	1895	[a]1300
Ni	28	22		1455	[a]570
Co	12	10		----	-----
Cr	9	9		----	-----
Mo	2	1		----	-----
Al	2	5		----	-----
B	1	3		----	-----
Si	1	3		----	-----
Component B: Silver Metal: 15 wt % of PM212					
Ag	100	100	-100 + 325 (44 to 150)	961	[c]25
Component C: Prefused Eutectic: 15 wt % of PM212					
BaF_2	62	52	-200 + 325	1050 1280	[b]150 [b]110
CaF_2	38	48	(44 to 74)	1423	[b]145

(a)Handbook of Physics and Chemistry, 70th ed., 1990, CRC Press Inc., Boca Raton, FL.

(b)Deadmore, D.L. and Sliney, H.E.: "Hardness of CaF_2 and BaF_2 Solid Lubricants at 25 to 670 °C," NASA TM-88979, March 1987.

(c)Lozinskii, M.G.: "High Temperature Metallography," Pergamon Press, 1961.

TABLE II TENSILE STRENGTHS OF EX-212

Extrusion Temp, Deg. F	Test Temp., Deg. F	Ultimate Tensile Strength, KSI
1400	70	21.4, 23.4
	800	20.0
	1200	17.5
	1400	7.0
	1600	1.6
1500	70	25.5
	800	22.0
	1200	17.0
	1400	7.1
	1600	2.2
1600	70	24.4, 24.0
	800	20.0
	1200	13.0
	1400	7.0
	1600	1.6
1700	70	19.0, 19.5
	800	16.5
	1200	12.0
	1400	5.6
	1600	1.3, 1.2
1800	70	24.2, 26.5
	800	19.0
	1200	16.0
	1400	10.5
	1600	2.4

TABLE III - Friction and Wear Comparison of EX-212 Pins Against René 41 Disks With Sliding Velocity of 9.2 ft/sec at 1000 RPM

Processing Method	Temperature [°C]	Friction Coefficient	Kpin 10^{-5} mm^3/N-m	Kdisk 10^{-5} mm^3/N-m	Number of Tests
Extruded at 1400F	25 (77F)	0.33±0.01	2.1±0.5	0.55±0.4	3
	350 (662F)	0.46±0.07	1.5±1.3	0.23±0.8	3
	760 (1400F)	0.26±0.03	0.39±0.1	0.15±0.5	3
Extruded at 1600F	25	0.37±0.04	3.2±0.3	3.5±4.3	3
	350	0.50±0.05	1.6±0.6	0.18±0.2	3
	760	0.35±0.01	6.8±3.7	-3.0±2.1	3
Extruded at 1800F	25	0.35±0.04	3.1±2.3	1.4±3.2	3
	350	0.44±0.08	1.2±0.6	0.23±1.1	3
	760	0.33±0.03	3.5±9.4	-1.1±3.9	3
Sintered	25	0.35±0.05	3.2±1.5	7.0±2.0	2
	350	0.38±0.02	3.9±1.8	0.35±0.1	3
	760	0.35±0.06	0.36±0.09	1.0±6.0	4
HIPped	25	0.37±0.04	1.8±0.4	0.45±0.1	≥4
	350	0.32±0.07	2.5±0.3	0.85±0.4	≥4
	760	0.31±0.04	0.07	2.2±0.8	≥4

Note: Uncertainties represent one standard deviation from the mean for the friction coefficients and the data scatter range for the wear factors.

TABLE IV

ROOM TEMPERATURE HARDNESS DATA FOR EX-212

EXTRUSION TEMP, DEG F	AVERAGE HARDNESS, R"C"
1400	21.5
1500	29.0
1600	31.5
1700	24.5
1800	32.0

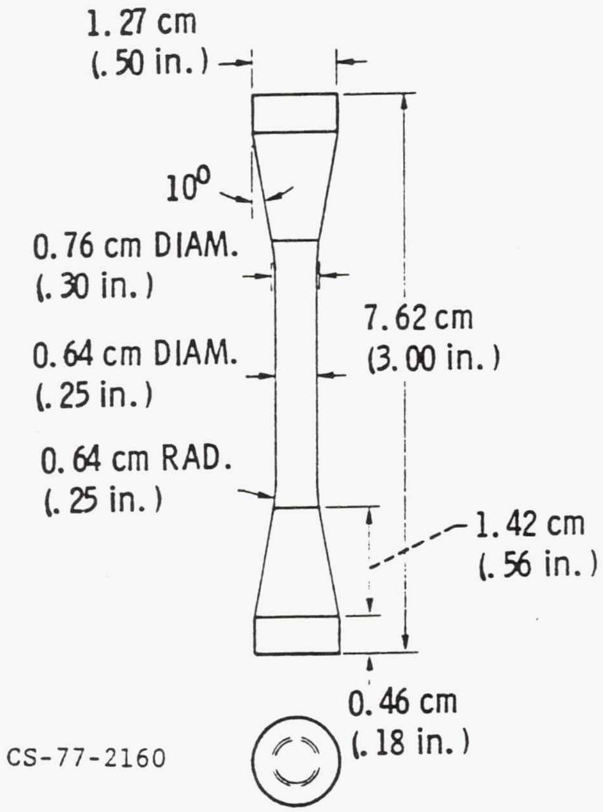


FIGURE 1.A - Tensile/Stress Rupture Specimen Ground From Extruded Bar Stock

Tribometer Pin Specimens

Finishing of Hemispherical Tips

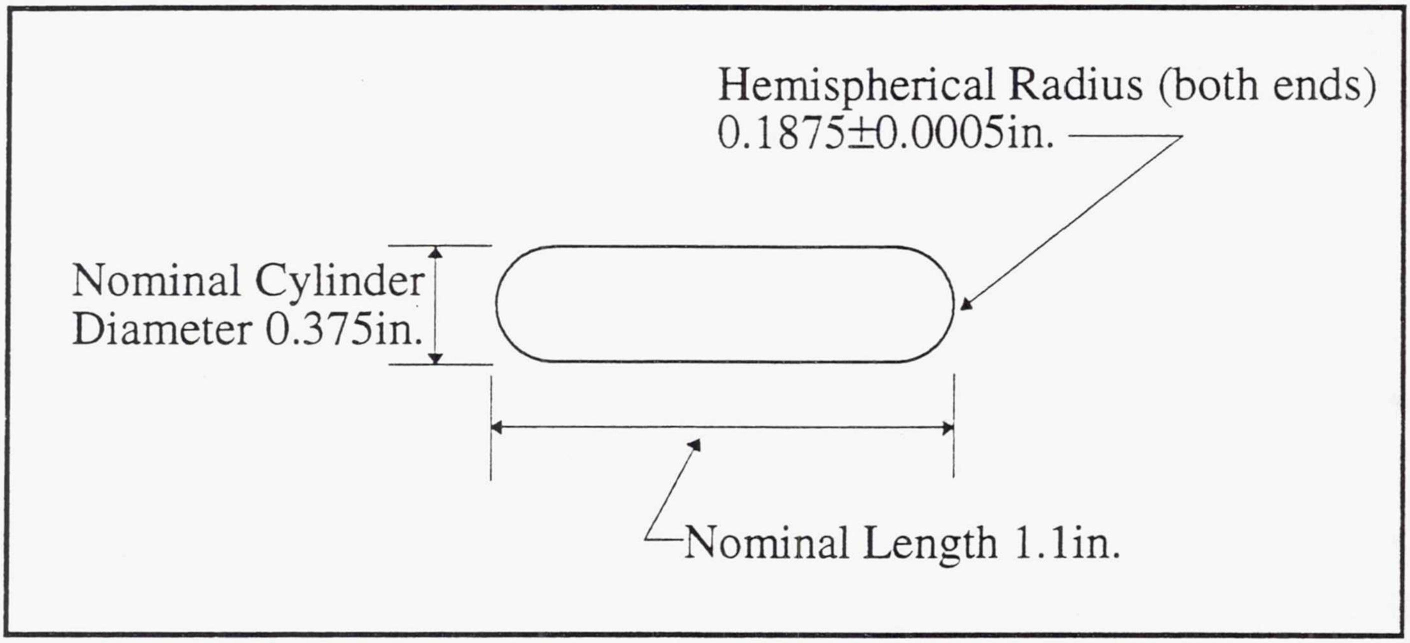
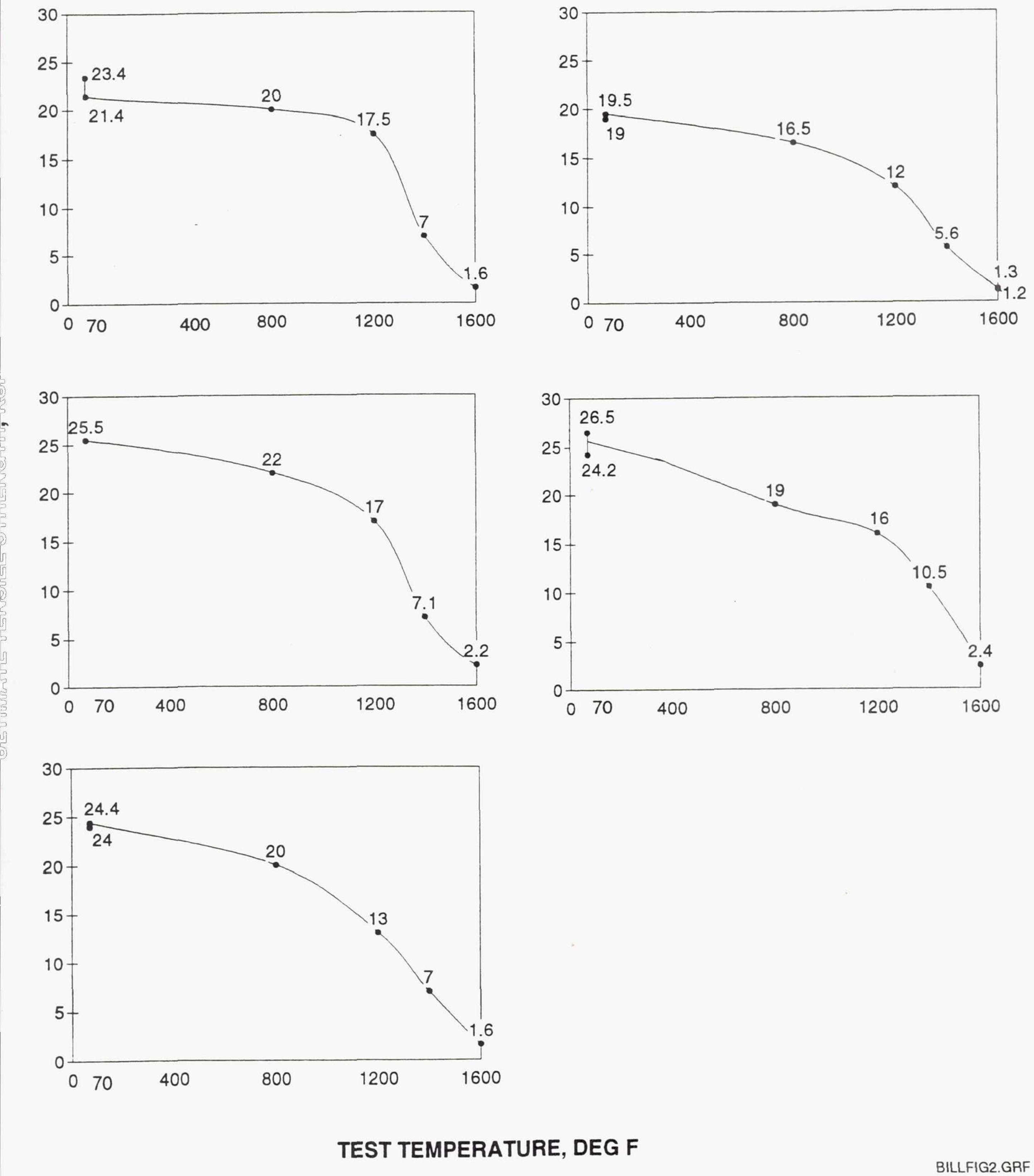


Figure 1.B

FIG. 2 ULTIMATE TENSILE STRENGTH OF EX-212 AT VARIOUS TEMPERATURES



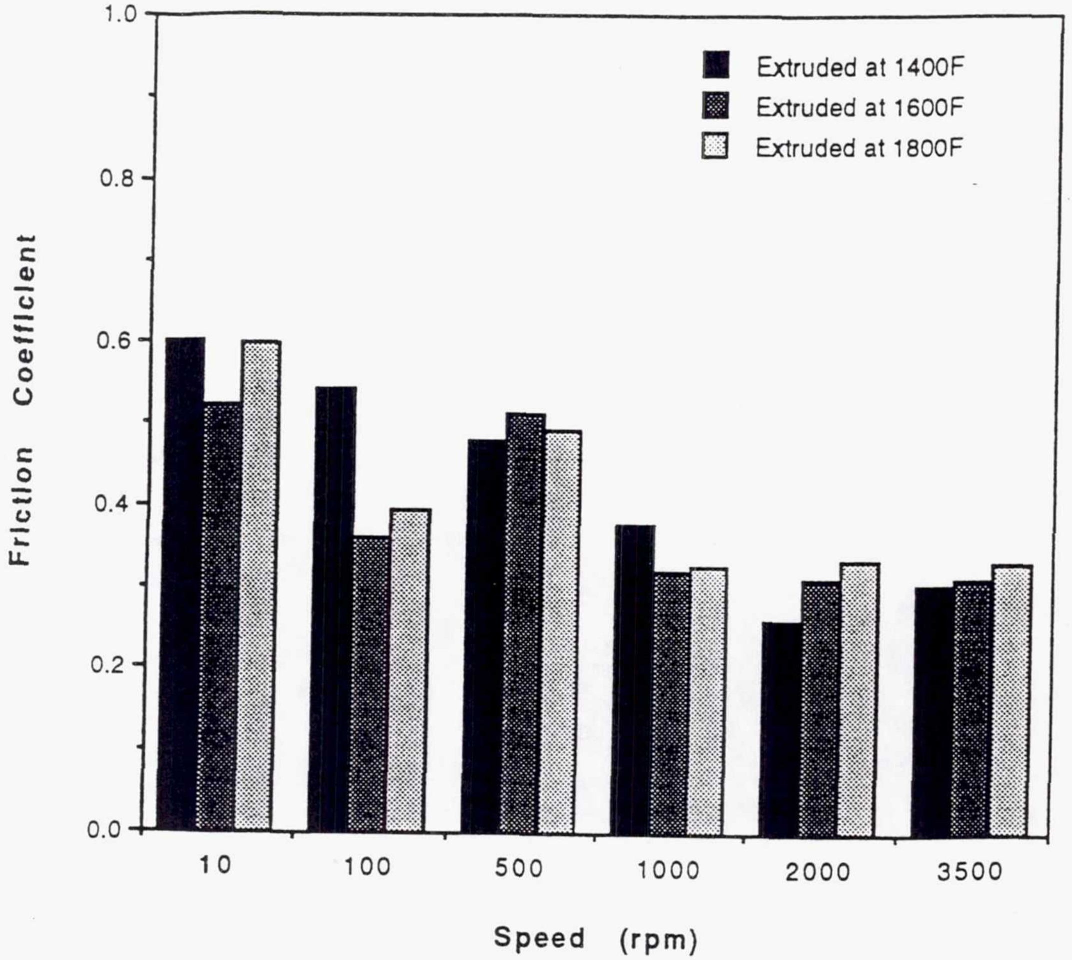


Figure 3 - Velocity Survey for Extruded PM212 Test Temperature at 25C (77F)

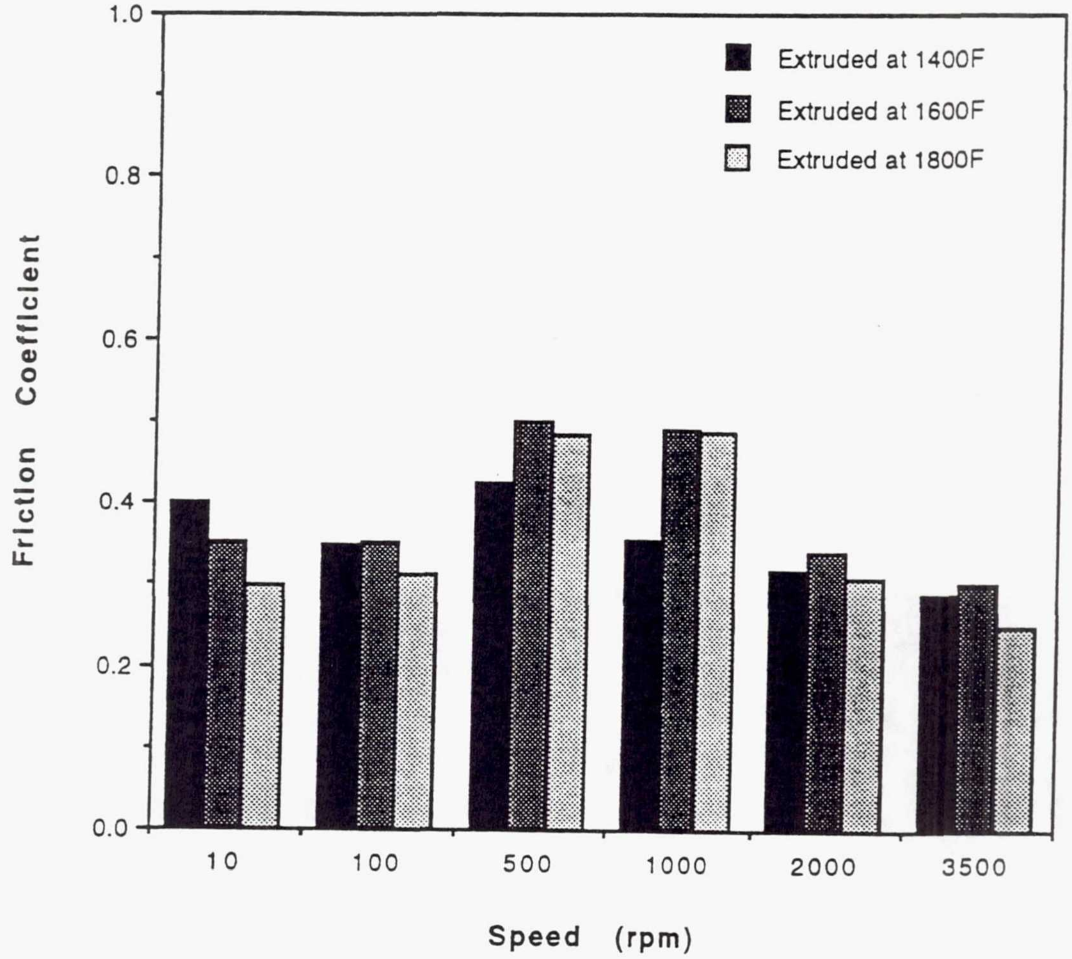


Figure 4 - Velocity Survey for Extruded PM212
Test Temperature at 350C (662F)

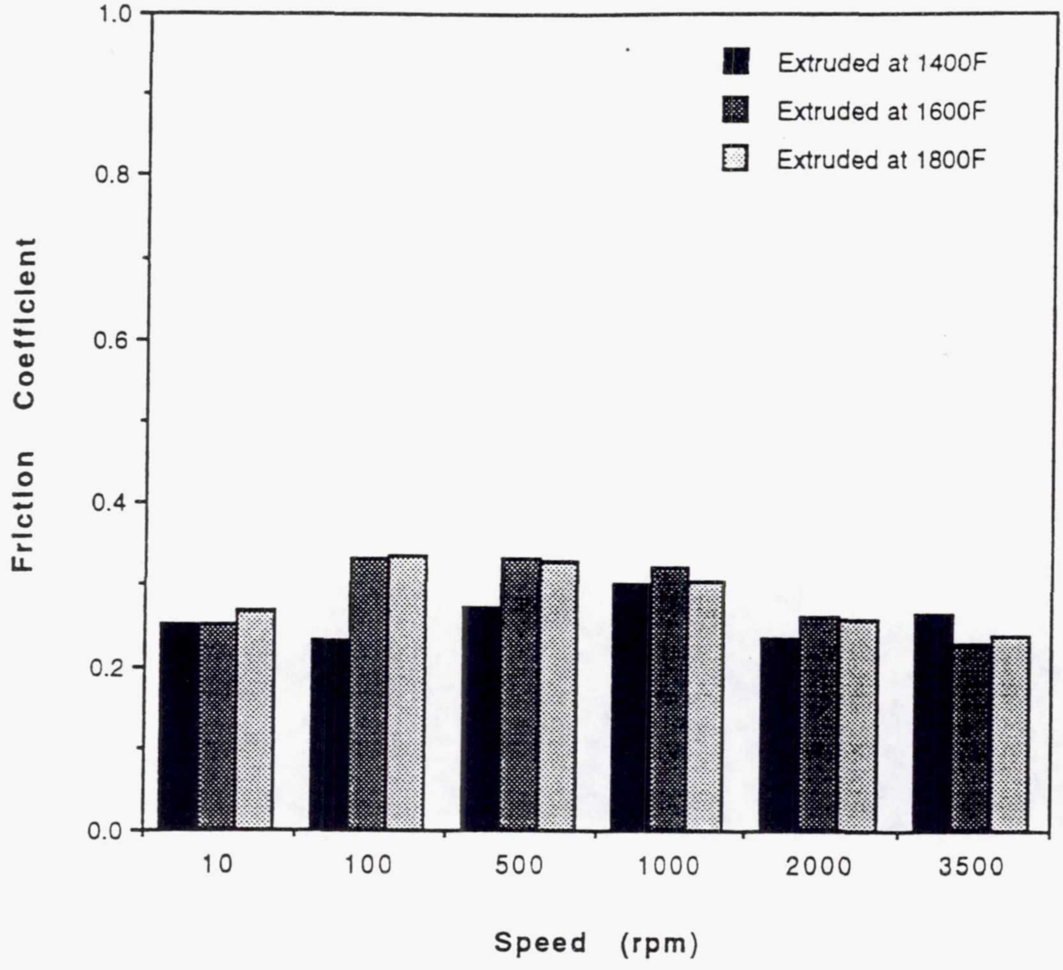


Figure 5 - Velocity Survey for Extruded PM212
Test Temperature at 760C (1400F)

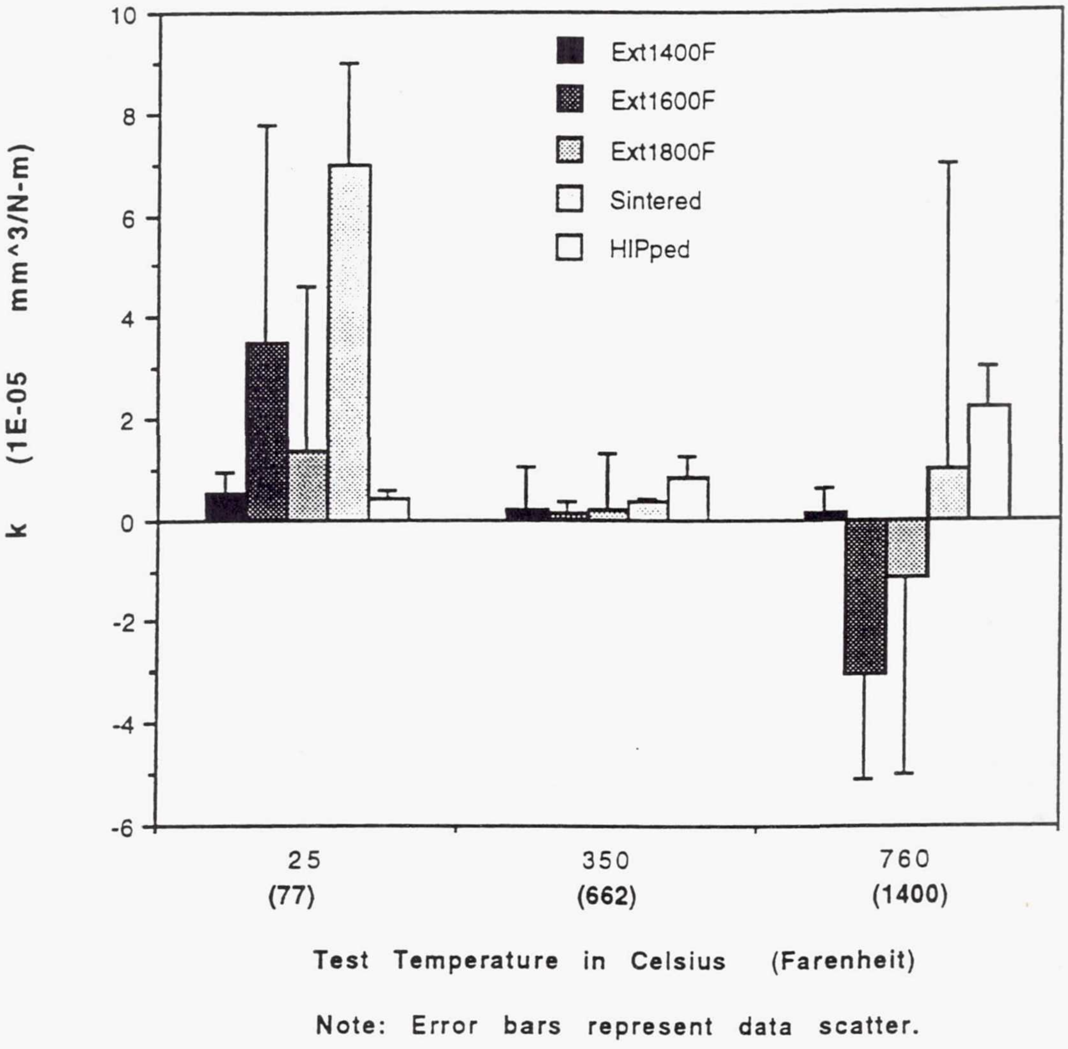


Figure 6 - Disk Wear Factors Rene 41 Disks vs. PM212 Pins

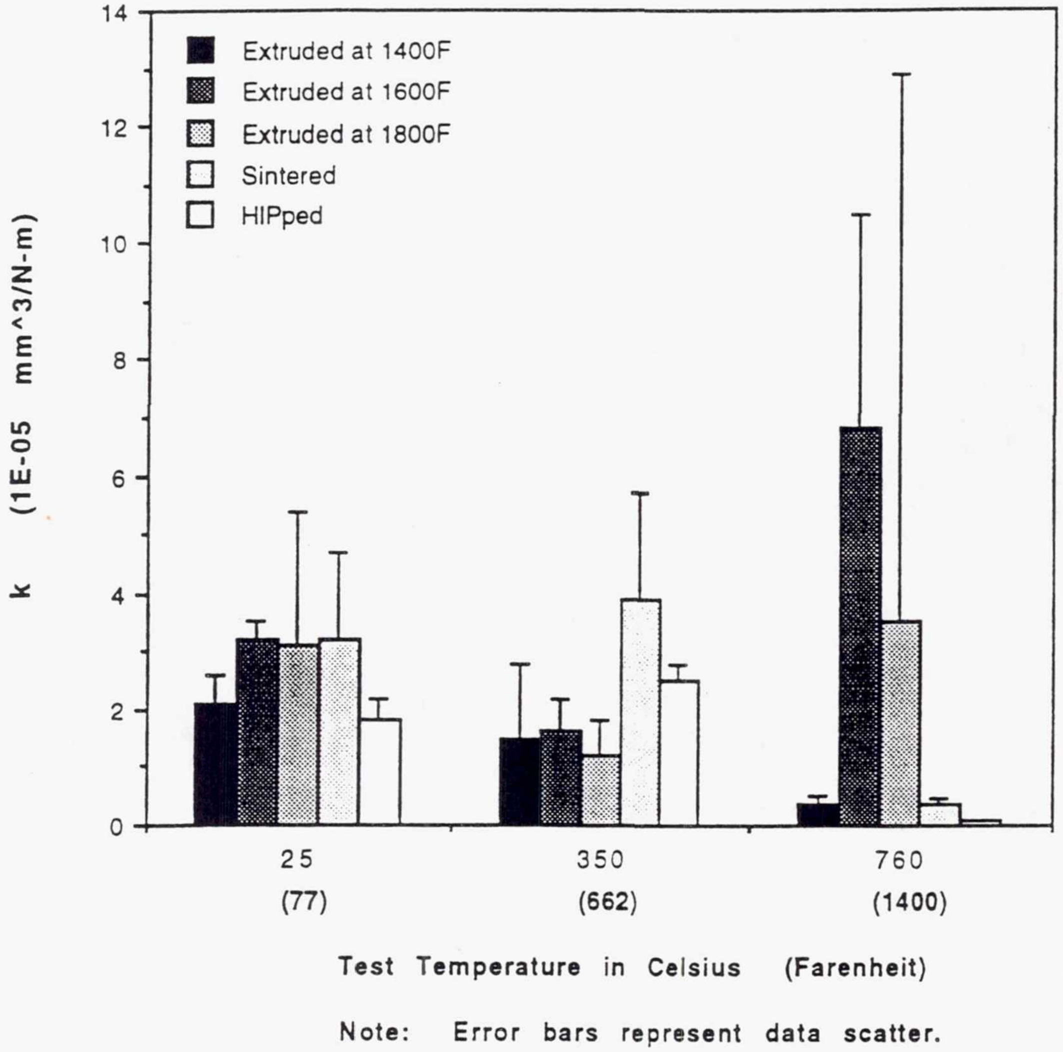
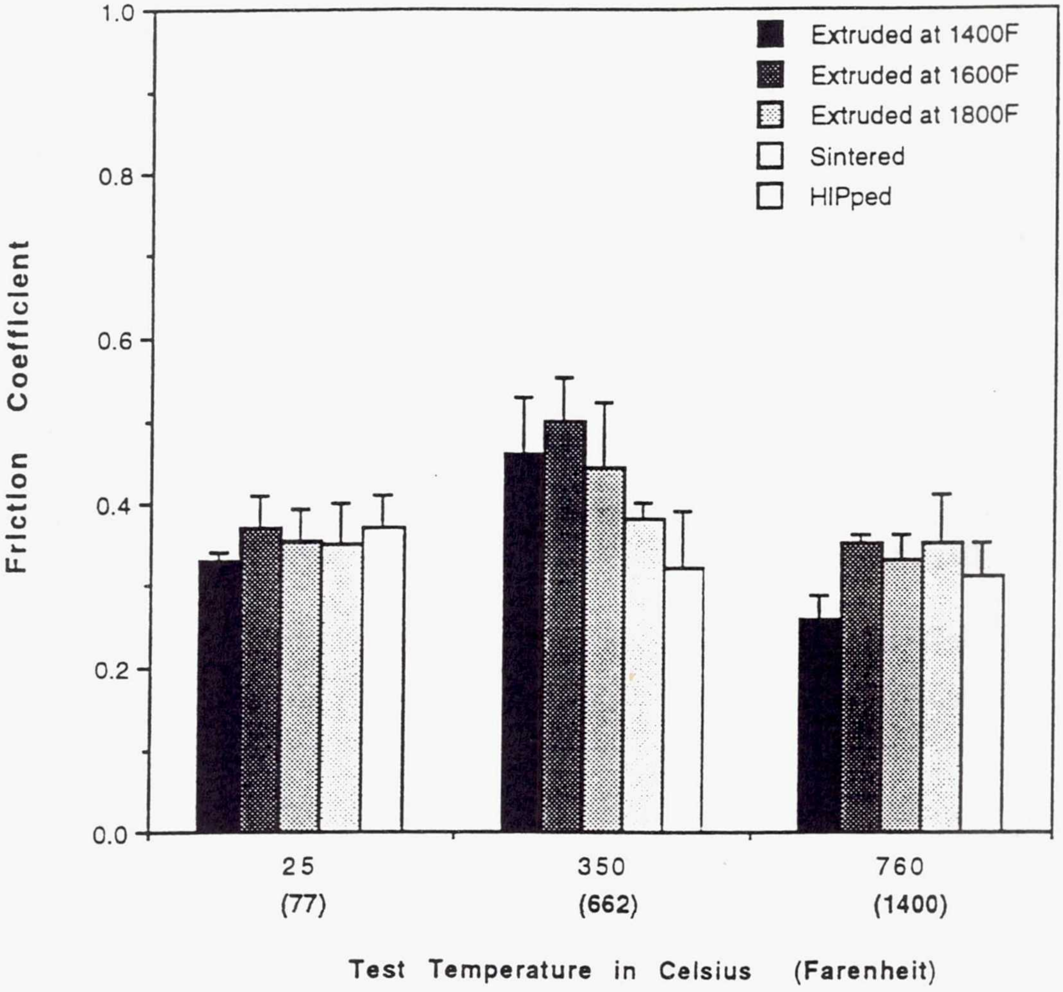


Figure 7 - Pin Wear Factors PM212 Comparison



Note: Error bars represent one standard deviation from the mean.

Figure 8 - Friction Comparison for PM212

(a) Ext. at 1400°F

(b) Ext. at 1500°F

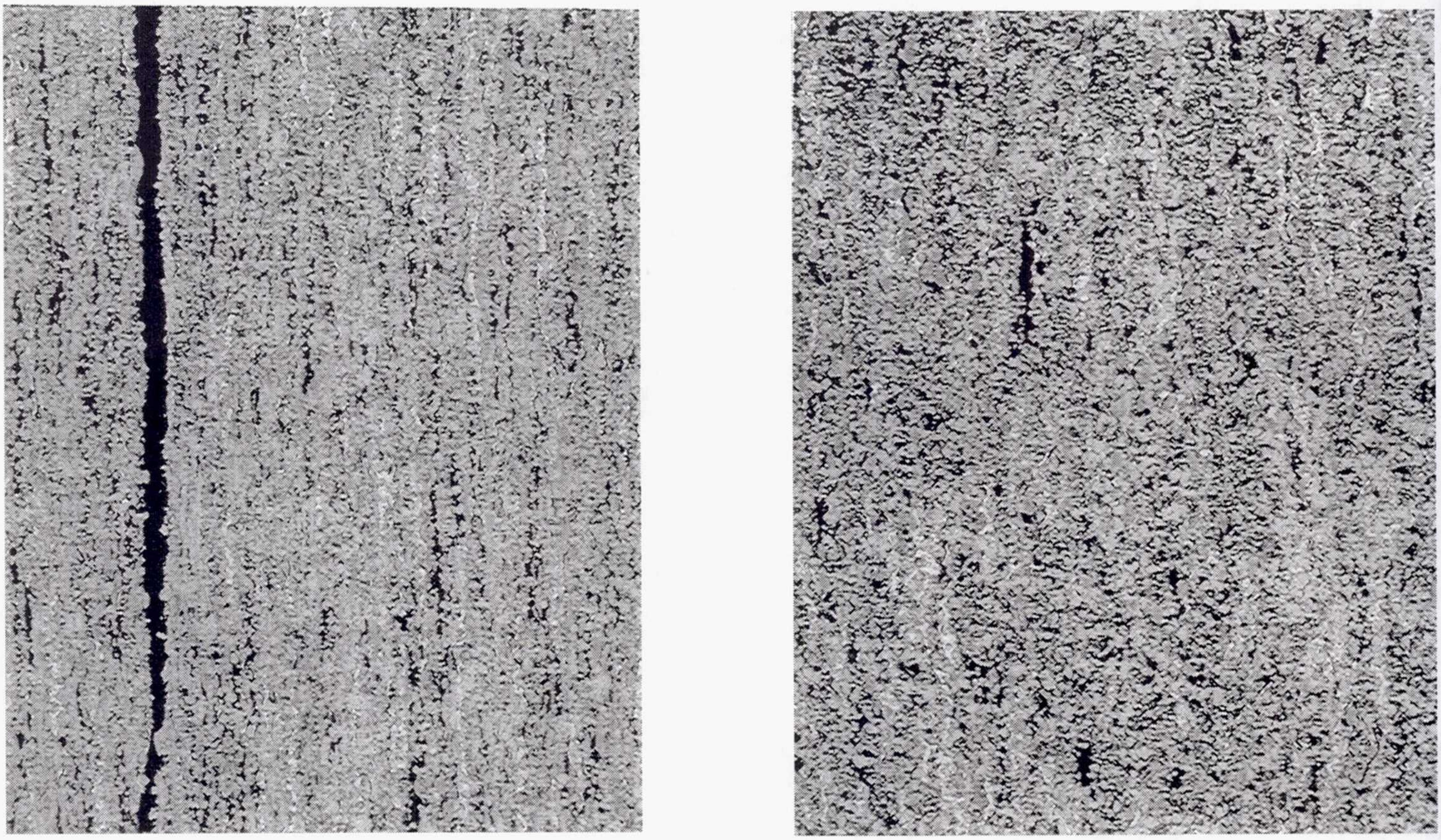


FIG. 9 EX-212 Unetched, 25x

(c) Ext. at 1600°F

(d) Ext. at 1700°F

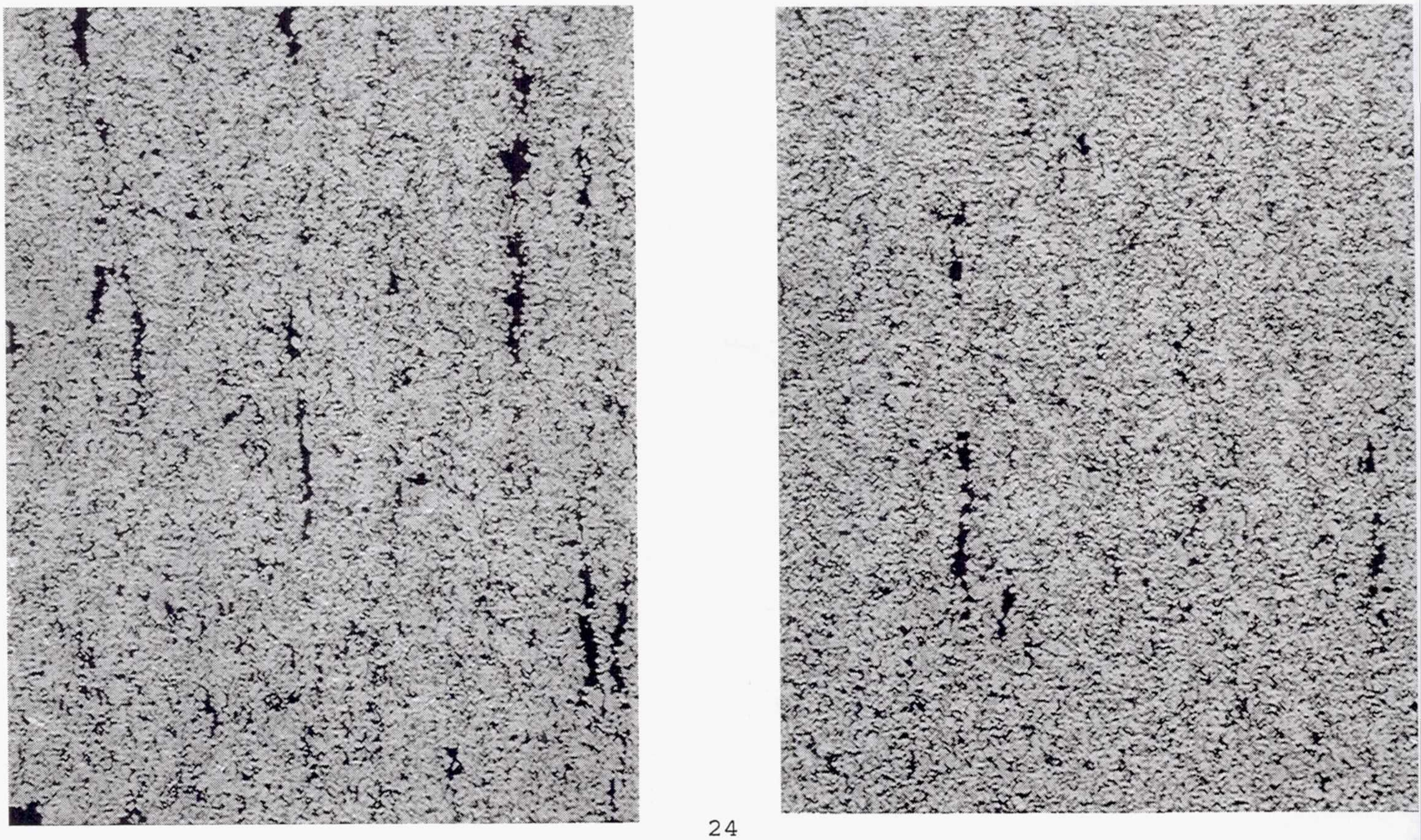
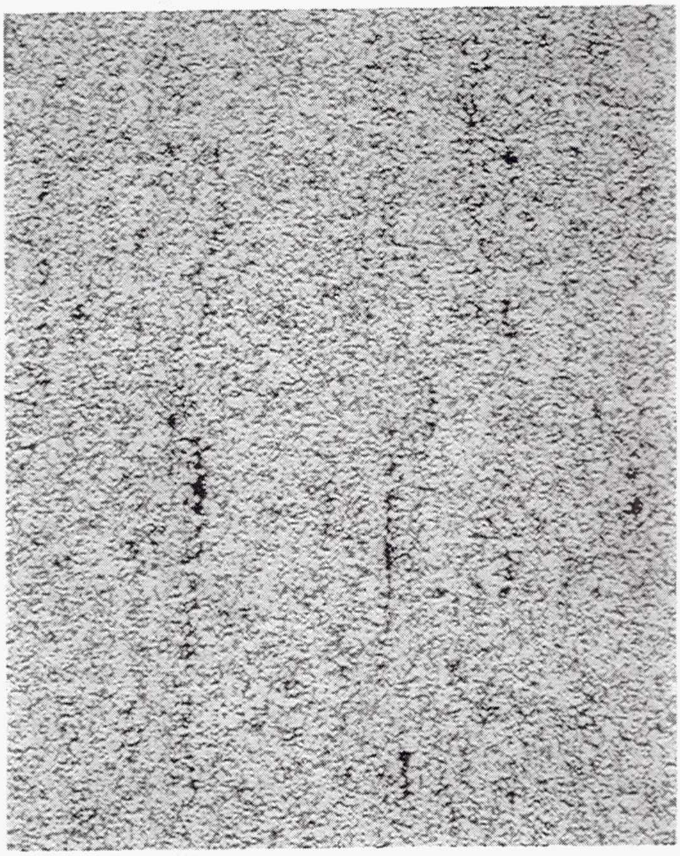
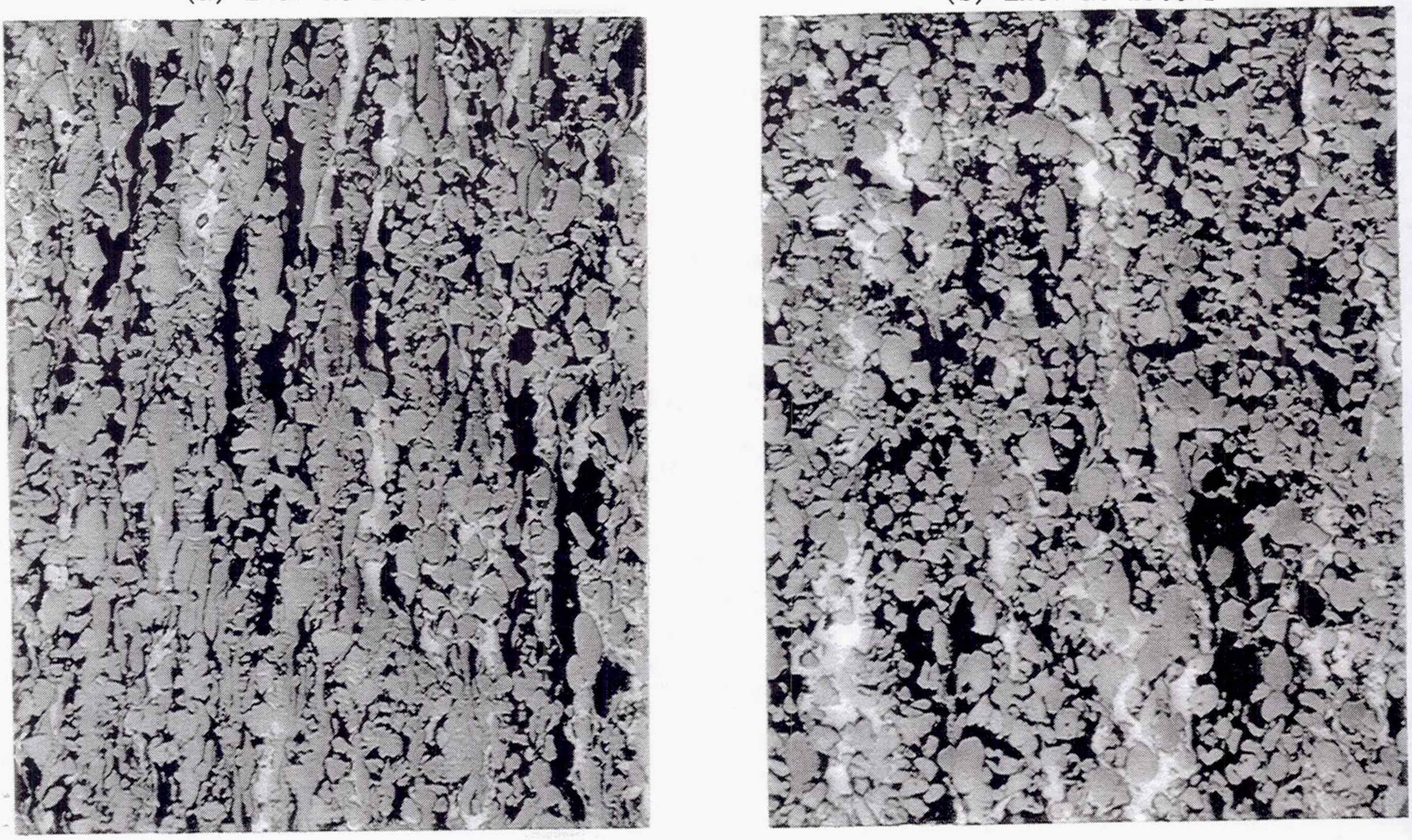


FIG. 9
(e) Ext. at $1800^{\circ}F$

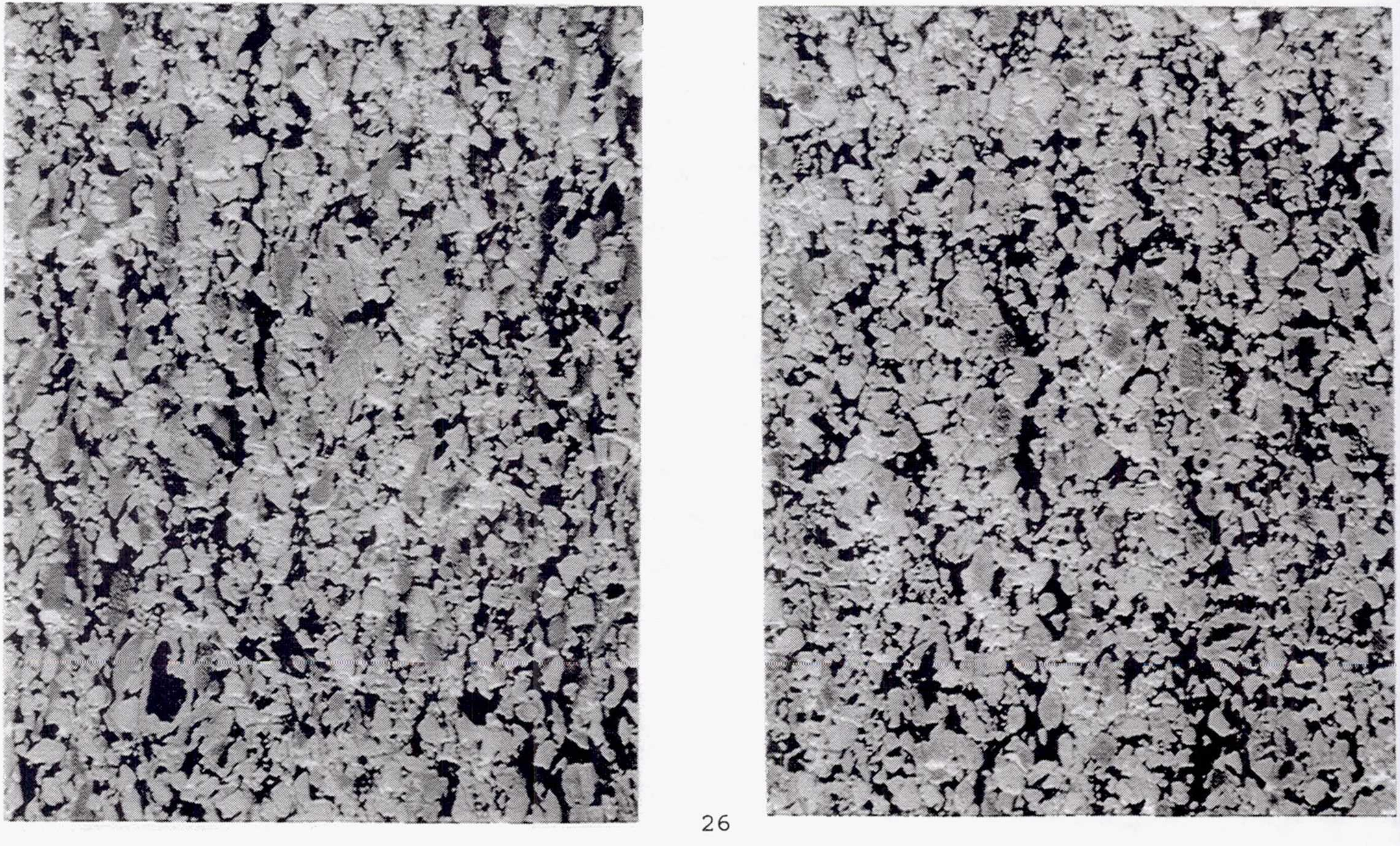




(a) Ext. at 1400°F

(b) Ext. at 1500°F

FIG 10. EX-212 Etched, 100x

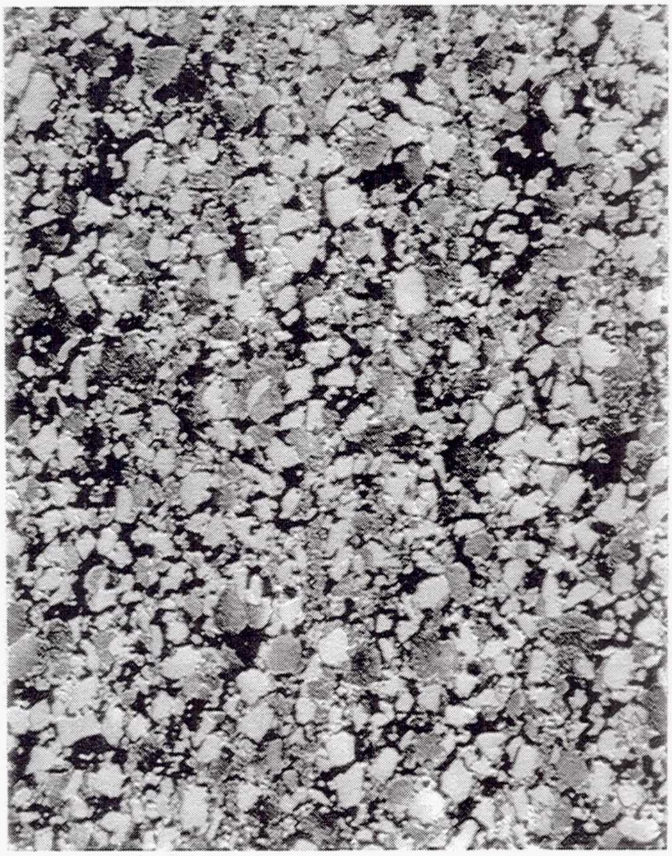


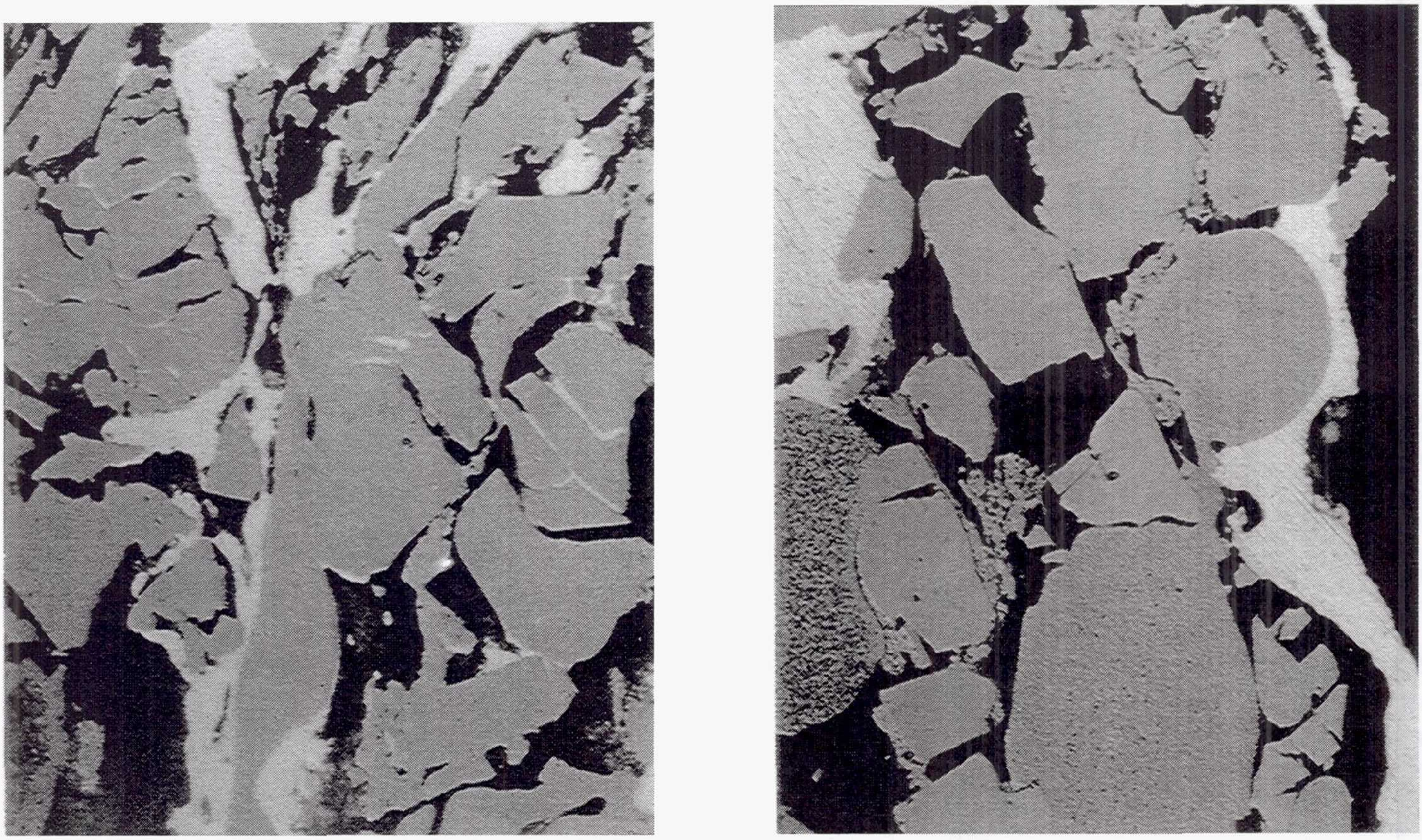
(c) Ext. at 1600°F

(d) Ext. at 1700°F

FIG. 10

(e) Ext. at 1800^{o}F

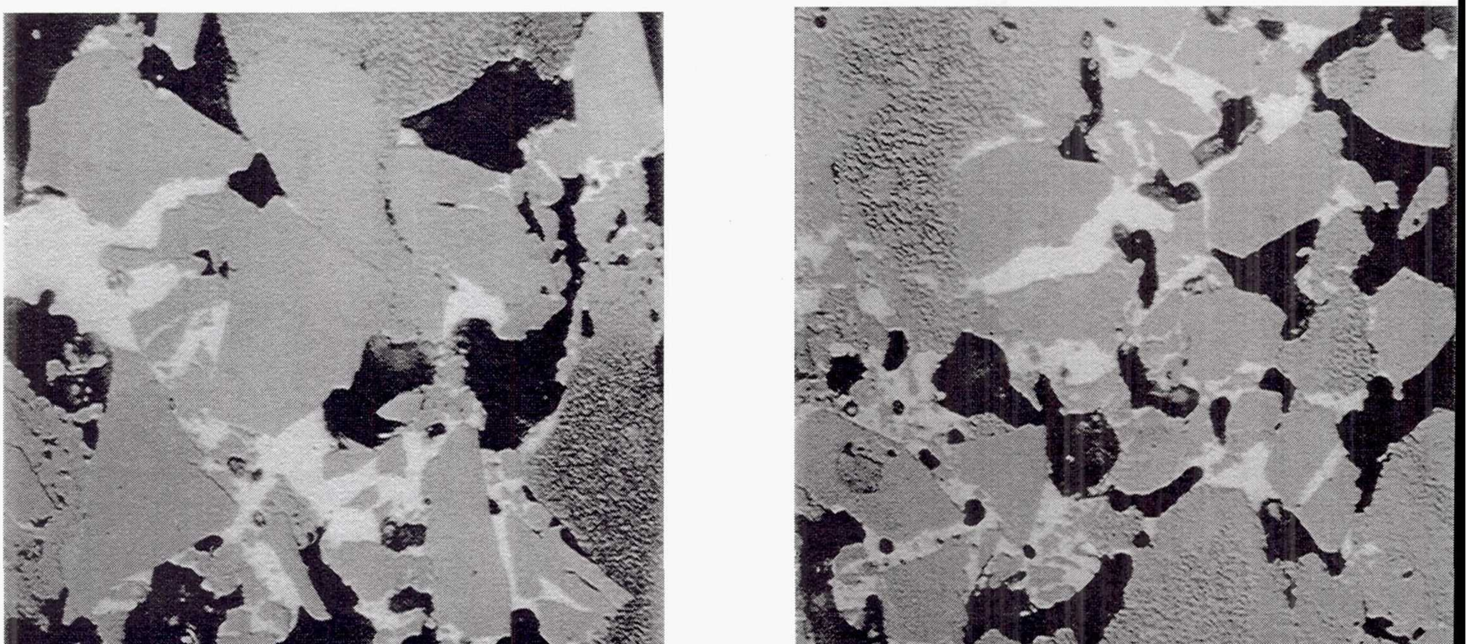




(a) Ext. at 1400°F

(b) Ext. at 1500°F

FIG. 11 EX-212 Etched, 1000x



(c) Ext. at 1600°F

(d) Ext. at 1700°F

FIG. 11

(e) Ext. at 1800°F

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