/N-27 1282 830

DOE/NASA/50162-5 NASA TM-103694

Mechanical Strength and Thermophysical Properties of PM212: A High Temperature Self-Lubricating Powder Metallurgy Composite

Phillip M. Edwards Case Western Reserve University Cleveland, Ohio

Harold E. Sliney, Christopher DellaCorte, J. Daniel Whittenberger, and Robert R. Martineau National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio

December 1990

Prepared for

U.S. DEPARTMENT OF ENERGY Conservation and Renewable Energy Office of Vehicle and Engine R&D

(NASA-TM-103694) MECHANICAL STRENGTH AND
THERMOPHYSICAL PROPERTIES OF PM212: A HIGH
TEMPERATURE SFLF-LUBRICATING POWDER
METALLURGY COMPOSITE Final Report (NASA) Unclas
30 p CSCL 11C G3/27 0001282

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Printed in the United States of America

Available from

National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price codes¹
Printed copy:
Microfiche copy: A03

¹Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: Energy Research Abstracts (ERA); Government Reports Announcements and Index (GRA and I); Scientific and Technical Abstract Reports (STAR); and publication, NTIS-PR-360 available from NTIS at the above address.

Mechanical Strength and Thermophysical Properties of PM212: A High Temperature Self-Lubricating Powder Metallurgy Composite

Phillip M. Edwards Case Western Reserve University Cleveland, Ohio 44106

Harold E. Sliney, Christopher DellaCorte, J. Daniel Whittenberger, and Robert R. Martineau National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135

December 1990

Work performed for U.S. DEPARTMENT OF ENERGY Conservation and Renewable Energy Office of Vehicle and Engine R&D Washington, D.C. 20545 Under Interagency Agreement DE-AI01-85CE50162

	• •	
	•	

MECHANICAL STRENGTH AND THERMOPHYSICAL PROPERTIES OF PM212:

A HIGH TEMPERATURE SELF-LUBRICATING POWDER METALLURGY COMPOSITE

Phillip M. Edwards*

Case Western Reserve University

Cleveland, Ohio

and

Harold E. Sliney, Christopher DellaCorte,
J. Daniel Whittenberger, and Robert R. Martineau
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

SUMMARY

A powder metallurgy composite, (PM212), composed of metal-bonded chromium carbide and solid lubricants is known to be self-lubricating to a maximum application temperature of 900 °C. In this paper, the high temperature compressive strength, tensile strength, thermal expansion and thermal conductivity data needed to design PM212 sliding contact bearings and seals are reported for sintered and isostatically pressed (HIPed) versions of PM212. Other properties presented are room temperature density, hardness, and elastic modulus. In general, both versions appear to have adequate strength to be considered as sliding contact bearing materials, but the HIPed version, which is fully dense, is much stronger than the sintered version which contains about 20 percent pore volume. The sintered material is less costly to make, but the HIPed version is better where high compressive strength is important.

INTRODUCTION

In recent years, the field of solid lubrication has received considerable attention for application in advanced technology systems. Developments in high-efficiency heat engines and advanced aircraft propulsion systems have identified numerous high temperature lubrication problems. The extreme conditions encountered in these advanced areas of technology preclude the use of conventional liquid lubrication methods and require alternative approaches such as the use of gas bearings, vapor phase lubrication, or new solid lubricants and self-lubricating materials.

A promising solid lubricant system intended for use from 25 to 900 °C has been invented at NASA Lewis Research Center (refs. 1 and 2). Plasma sprayed coatings and powder metallurgy composites, known as PS212 and PM212, respectively, are composed of a metal-bonded chromium carbide matrix with silver and BaF₂/CaF₂ eutectic solid lubricant additives. Previous work has largely dealt with the tribological evaluation of this material in the form of a plasma-sprayed coating. Low friction coefficients and wear have been observed in laboratory evaluations of PS212 (refs. 3 and 4). However, not all surfaces can be coated by the plasma spraying process. For example, it is very difficult to spray the internal surfaces of cylindrical bearings and bushings.

NASA Resident Research Associate.

A logical next step therefore is to use this material to fabricate free-standing parts by powder metallurgy processes. Previous experimental work with PM212 has been solely concerned with the tribological evaluation of this composite (ref. 5). This paper presents the results of a test program to evaluate the mechanical and thermophysical properties of PM212 needed for bearing and seal design. PM212 was fabricated into test specimens using two different powder metallurgy processing techniques: sintering and hot isostatic processing. A series of tests were then conducted to assess the properties of compressive and tensile strength, strain rate sensitivity, Young's modulus, coefficient of thermal expansion, and thermal conductivity of these two powder metallurgy forms of PM212.

MATERIALS AND PROCEDURES

Abbreviations of Process Terms

CIP = cold isostatically pressed

HIP = hot isostatically pressed

(CS) = CIPed, then sintered composite

(CH) = CIPed, then HIPed composite

Example: PM212 (CS) - A composite prepared by cold isostatically pressing followed by sintering of PM212 powders.

PM212 is composed of 70 wt % metal-bonded chromium carbide (Metco 430NS), 15 wt % silver, and 15 wt % barium-fluoride/calcium fluoride eutectic. Table I lists the composition of PM212. The metal-bonded chromium carbide, serves as a hard and wear-resistant matrix for the solid lubricant additives. Silver provides lubrication from low temperatures to 500 °C (ref. 6) while the fluoride eutectic is lubricous from 500 to 900 °C (ref. 7). All components in PM212 are chemically stable up to 900 °C or higher.

Material Preparation

The two forms of PM212 evaluated in this study were produced either by sintering or hot isostatic pressing of powder compacts. The silver and the metal-bonded chromium carbide are commercially procured as -100 and -200 mesh powders. The fluoride eutectic is fabricated by first prefusing the eutectic mixture of BaF₂ and CaF₂ at 1100 °C. The cooled melt is then mechanically crushed followed by further size reduction in a ball mill. The resulting eutectic powder is then sieved to the desired -200 + 325 mesh size. The metal-bonded chromium carbide silver, and fluoride eutectic are combined in the appropriate PM212 proportions and blended in a vee blender for one-half hour. The blended powder can either be mechanically cold pressed in a die or cold isostatically pressed. In this work the powder is CIPed at 414 MPa (60 000 psi) for 5 min in a rectangular rubber bar mold. The cold pressed bar is then either sintered or HIPed.

Sintering Procedure

The cold pressed (green) compact is pressureless sintered in a tube furnace with a dry hydrogen atmosphere. The furnace is heated at a rate of 10 °C/min to 1100 °C, held at that temperature for 30 min, then cooled at a rate of 10 °C/min.

Hot Isostatic Pressing Procedure

The cold pressed bars are vacuum sealed in a stainless steel can which is lined with graphite to prevent interfacial chemical reactions between the bar and the steel envelope. HIPing is performed in argon pressurized to 138 MPA (20 000 psi) at 1100 °C for 20 min. Heating and cooling rates are 6 and 12 °C/min, respectively. The rate of pressurization and depressurization are 0.75 and 1.5 MPa/min (109 and 218 psi/min), respectively.

Machining

Wire electrodischarge machining, with water as the cutting fluid, is used to fabricate test specimens from the sintered or HIPed bars. Parts intended for tribological applications are final machined by diamond grinding. Some parts made by this procedure are shown in figure 1. Considerably less machining of sintered parts is possible by cold mechanically pressing the powders to near final dimensions in a die prior to sintering. Allowances for any shrinkage that may occur during sintering is made in the design of the die.

Mechanical Strength Measurements

Compressive strength. - Compression tests were conducted on cylindrical specimens of the two forms of 430 NS and PM212. All tests were performed in triplicate. The specimens were 10 mm long and 5 mm in diameter in conformance to the standard ratio of 2:1 commonly used in compressive strength determinations. Tests were conducted at 25, 350, 760 and 900 °C in air on an universal test machine at a constant crosshead velocity calculated to yield an initial strain rate of $2.1 \times 10^{-4} \text{ s}^{-1}$. Stress and strain data were calculated from the load-time charts. A series of tests at varying strain rates were also conducted at 760 °C to assess the strain rate sensitivity of the sintered and hipped PM212.

Tensile Strength

The first tensile tests were of PM212 (CS). The specimens were threaded at both ends for assembly into the test machine; the gage sections were 13 mm long and 3 mm in diameter. This geometry was difficult to machine with this material. Subsequent PM212 (CH) specimens substituted a button head design for the threaded ends, a longer gage length of 32 mm to accommodate the grips, and retained the 3 mm gage diameter. All tensile tests were conducted at an initial strain rate of 2.1×10^{-4} s⁻¹ in air at 25, 550, and 760 °C until tensile failure occurred.

Elastic Modulus

An ultrasonic technique known as the pulse-echo method was used to evaluate the room temperature elastic modulus of PM212 (CS) and PM212 (CH). This test was performed on 25 mm square and 2.5 mm thick samples with the faces machined parallel to ± 0.025 mm. In this technique, the piezoelectric transducer emits a sound wave into the sample and monitors the reflections off the front and back sample faces as displayed in figure 2. These reflections, in conjunction with a mathematical algorithm known as the cross-correlation method (ref. 8), lead to the determination of the velocity of sound through the sample. The elastic modulus for each material is then calculated from the following formula (ref. 9): $E = v^2 \rho$.

Where E is the elastic modulus, v is the sonic velocity, and ρ is the density of the material.

Thermophysical Properties

Various thermophysical properties of the two forms of PM212 were determined as a function of temperature. The linear coefficient of thermal expansion was measured using a dilatometer at temperatures from 25 to 850 °C in a nitrogen atmosphere. These measurements were conducted on 12 mm diameter by 50 mm long cylindrical samples. Dilatometer measurements were also conducted on sintered and HIPed samples of the 430NS bonded carbide matrix material for comparative purposes.

Thermal conductivity (λ) was calculated as a product of specific heat (Cp) thermal diffusivity (α) and bulk density (ρ), i.e., $\lambda = \text{Cp}\alpha\rho$. The specific heat of PM212 was evaluated from 25 to 600 °C using a differential scanning calorimeter and sapphire as the reference material. These tests were conducted in an argon atmosphere on cylindrical samples with diameter and thickness equal to 5.6 and 16.5 mm, respectively. The thermal diffusivity was evaluated using a laser flash method in which the front face of a sample is subjected to a short laser pulse while the temperature rise is monitored on the back face. The heating rate is then used to calculate the thermal diffusivity at the set temperature. Bulk densities were computed from sample geometries and mass. Further details concerning the experimental procedure for these techniques can be found in references 10 and 11.

RESULTS

Microstructure

Optical photomicrographs in figure 3(a) and (b) illustrate the microstructures of sintered metal-bonded chromium carbide and PM212. In both materials, the hard chromium carbide particles can be seen surrounded by a matrix of nickel alloy binder. The brightest areas in figure 3(b) are silver. Both materials contain significant amounts of porosity which appear as dark areas in the photomicrographs. Some of the dark areas in PM212 are the fluorides which are transparent under veritical illumination. The average density, of the sintered bonded carbide, based upon weight and volume measurements, is 50 percent of theoretical while that of sintered PM212 is 80 percent of theoretical. Clearly, porosity constitutes a significant volume fraction of the sintered materials.

HIPed bonded carbide and PM212, on the other hand, are essentially fully-consolidated with densities 100 percent of theoretical, $(7.01~g/cm^3~for~430NS~and~6.60~g/cm^3~for~PM212)$. One effect of the differences in density of PM212 (CS) and PM212 (CH) is quantifiable by room temperature hardness, where PM212 (CS) has a Rockwell hardness of $R_{\rm p}45$ while that of PM212 (CH) is $R_{\rm c}33$.

The microstructure of sintered and PM212 (CH) are compared in the optical photomicrographs in figure 4(a) and (b). The much coarser microstructure of the sintered material illustrates the incomplete consolidation of the sintered compact. However, the fluorides cannot be distinguished from porosity in these pictures. Therefore, backscatter scanning electron micrographs of PM212 are presented in figure 5(a) to (c). All of the components can be distinguished according to the atomic number of the elements present; the brighter the area, the higher the atomic numbers of the elements in that area. All of the major components of PM212 (CS) including pore space are labelled in figure 5(a), and higher magnification pictures

of selected areas are shown in figure 5(b) and (c). The bright fields are silver, light gray fields are the nickel alloy binder, carbides are a little darker shade of gray, and pore spaces are black. The fluoride areas (fig. 5(c)) have the highly lamellar structure characteristic of eutectics, where the darker bands are calcium fluoride and the lighter ones are barium fluoride.

Compression Tests

The results of the compression strength study are given in table II and figures 7 to 12. The various mechanical properties given in table II are illustrated on the idealized stress/strain curve in figure 6. The weakest of the four materials was 430NS-CS which displayed essentially brittle behavior at 25 and 350 °C but exhibited ductile behavior at 760 and 900 °C. The 0.2 percent yield stress and maximum stress of this sintered carbide matrix material remained relatively constant up to 760 °C followed by a significant reduction at 900 °C (fig. 7).

PM212 (CS) was approximately seven times stronger than the sintered carbide matrix material at the three lower temperatures (table II). It displayed very little plasticity, and almost identical, stress-strain behavior at 25 and 350 °C. At 760 and 900 °C, a substantial decrease in strength coupled with an increase in ductility was observed as illustrated in figure 8.

PM212 (CH) was approximately three times stronger than PM212 (CS) at all temperatures. As in the case of the sintered materials, PM212 (CH) had little ductility at 25 and 350 °C (table II). As can be seen in figure 9, the strength of PM212 (CH) at 350 °C was slightly less than the room temperature strength; however, a substantial reduction in strength and increase in ductility was evident at 760 and 900 °C.

The HIPed carbide matrix material was the strongest of the four materials, but there was a decrease in strength coupled with an increase in ductility at 760 and 900 °C. The 0.2 percent yield strength as a function of temperature is illustrated in figure 10. In figure 11, the strengths of the four test materials evaluated in this study are compared as a function of temperature. Clearly, the HIPed form are much stronger than the sintered materials.

The results of compression tests conducted on PM212 (CS) and PM212 (CH) at 760 °C and at various strain rates are listed in table III and illustrated in figure 12. Significant strength reductions were observed with decreasing strain rate for both materials. The amount of permanent deformation prior to maximum stress increased with decreasing strain rate.

Tensile Tests

The averaged results of duplicate tensile tests conducted on PM212 (CS) and PM212 (CH) are listed in table IV. At all three temperatures, PM212 (CS) displayed relatively weak and brittle tensile behavior. The two room temperature tests failed at an average stress of 45 MPa (6.4 ksi) after 0.1% elastic strain with no measurable permanent elongation. The tests at 550 and 760 °C produced similar results experiencing brittle failure at an average of 25 MPa (3.6 psi). The 760 °C tests displayed a small, almost negligible, amount of permanent elongation.

PM212 (CH) was significantly stronger than PM212 (CS) while also displaying brittle failure at the test temperatures (table IV). The failure strength of PM212 (CH) at 25 and 550 $^{\circ}$ C was fairly constant at approximately 118 MPa (17 ksi).

As in the case of PM212 (CS), failure occurred in the elastic regime. At 760 $^{\circ}$ C, the average tensile failure strength of the material declined to 84 MPa (12 ksi) with no permanent elongation in the specimen.

Acoustic Evaluation of Young's Modulus

The results of the acoustic evaluation of Young's modulus for PM212 (CS) and PM212 (CH) are listed in table V. The average modulus for PM212 (CS) and PM212 (CH) at room temperature were 95 GPa (14x10⁶ psi) and 175 GPa (25x10⁶ psi), respectively. The average sound velocities for PM212 (CS) and PM212 (CH) were 4290 m/s and 5162 m/s, respectively. Sonic velocity measurements made at three different locations on each sample varied by less than 1 percent. All sonic velocity measurements were made using the first and second reflections of the sound pulse off the back surface of the sample plate. Attenuation of the sound wave and interference from the relatively rough sample surface prevented viewing of any subsequent reflections.

Linear Coefficient of Thermal Expansion

The thermal expansion coefficients of PM212 and the carbide matrix material are given in table VI. The average thermal expansion coefficient of PM212 from 25 to 850 °C is $(14.4\pm0.05)\times10^{-6}$ °C for the HIPed material and $(13.8\pm0.05)\times10^{-6}$ °C for the sintered material. The thermal expansion coefficient for the HIPed carbide matrix material over the same temperature range is $(13.3\pm0.05)\times10^{-6}$ °C. Curves of thermal expansion versus temperature are given in figure 13(a) and (b). The slopes gradually increase with temperature. Above 500 °C PM212 expands more than 430NS. Both materials have an average expansion coefficient of 12.2x10⁻⁶/°C from 25 to 550 °C, but the coefficient for PM212 is about $18 \times 10^{-6}/^{\circ} \text{C}$ from 550 to 850 °C while that of 430NS is only about 14×10^{-6} /°C in the same temperature range (table VI). The higher expansion coefficient of PM212 is attributable to the very high expansion coefficients of CaF, BaF, and silver which are about twice that of Cr,C,. The lower expansion coefficient of sintered versus HIPed PM212 is possibly due to the 20 percent porosity in the sintered material which may provide partial relief of the internal strain that is caused by the higher expansion of the fluorides and silver. Residual strain existed in HIPed material prior to the first heating cycle of the thermal expansion determination. This is evidenced by the thermal expansion hysteresis shown in figure 13(b).

Thermal Conductivity

Thermal conductivity was calculated from the values of density, specific heat, and thermal diffusivity given in table VII. Conductivity as a function of temperature is plotted in figure 14. The thermal conductivities of both materials steadily increased with temperature. PM212 (CH) was consistently more conductive than sintered PM212 (CS) at all temperatures. The values of specific heat used in the calculations above 600 °C where obtained by extrapolation due to temperature limitations of the differential scanning calorimeter used in this study. This introduces a small amount of uncertainty into the thermal conductivity results from 600 to 900 °C.

DISCUSSION

Materials Processing

The results of the sintering operation showed a substantial density difference between the sintered carbide (50 percent porosity) and PM212 (20 percent porosity). Minimal density gains were observed during the pressureless sintering of the PM212 and carbide powder compacts. Consequently, the density differences between the sintered carbide and PM212 can be attributed to density differences in the presintered compacts of the two materials. It is believed that the solid lubricants in PM212 may have aided the compaction by reducing the interparticle friction. All HIPed specimens were fully-dense.

Compression Tests

The difference in porosity, was largely responsible for the strength difference observed between HIPed and sintered materials. The strongest material was the HIPed carbide followed by PM212 (CH), PM212 (CS), and sintered carbide. The respective densities of these materials were 100, 100, 78 and 54 percent of their theoretical values. In the case of 430NS, the 46 percent higher density of the HIPed carbide relative to the sintered carbide resulted in a 30-fold increase in the 0.2 percent yield strength at 25 and 350 °C. For PM212, the 22 percent higher density of hot isostatically pressed relative to the sintered composites produced an approximately 3-fold increase in strength at 25 and 350 °C. These results clearly illustrate the benefits of the full density processing.

Strength differences in the HIPed materials were due to compositional differences between the carbide matrix material and PM212. When silver and fluoride eutectic are added to the carbide matrix material to formulate PM212, these weaker solid lubricants dilute the stronger matrix material. This results in an overall reduction in strength as compared to the carbide matrix material.

In both the sintered and HIPed PM212, a significant reduction in strength was observed when increasing the temperature to 760 and 900 °C. It is believed that the fluorides are influential in this reduction. Previously reported hardness measurements on the fluoride eutectic have demonstrated the existence of a brittle to ductile transition temperature at approximately 550 °C (ref. 12). Additional factors are the softening of silver and the nickel-alloy binder at elevated temperatures. The combined softening of the two solid lubricant materials and the binder in the matrix material leads to a decline in the strength of PM212 at elevated temperatures.

The variable strain rate tests conducted at 760 °C displayed an approximately linear relationship between the strength properties and the log of the strain rate for both materials as illustrated in figure 12, a form of creep behavior that is typical of high temperature materials.

Tensile Tests

PM212 (CS) and PM212 (CH) were much weaker in tension (table IV) than in compression which is typical for a brittle material. The dominant factors in the tensile fracture of PM212 were the material porosity and possibly weak interfacial bond among the various components. Excessive porosity in the sintered material created stress concentration sites which led to rapid fracture across the sintered

interface. As in the compression tests, the fully-dense PM212 (CH) was approximately three times as strong in tension as PM212 (CS). However, the results indicate that neither material is suitable for use under appreciable tensile loading conditions.

Acoustic Evaluation of Young's Modulus

As can be seen in table V, the acoustically measured elastic modulus of PM212 (CH) at room temperature (175 GPa) was approximately twice that of PM212 (CS) (94 GPa). The porosity in sintered PM212 is believed to be responsible for the reduction in the elastic modulus of PM212 (CS). Sonic velocities follow a well known relationship between density and speed of sound in a material. The ratio of the sonic velocities for the two materials approximately equaled the ratio of their densities. This relationship between speed of sound and density in materials has also been reported in past work by NASA personnel on density measurements in porous ceramic materials (ref. 13). The average sonic velocity for PM212 (CH) (5160 m/s) is about 8 percent below the speed of sound through nickel (5630 m/s) which is a major constituent of the matrix material.

Linear Coefficient of Thermal Expansion

The results given in table VI and figure 13(a) indicate that the thermal expansion coefficients for both forms of PM212, especially below 550 °C, are largely determined by the metal bonded chromium carbide content. Above 550 °C, the higher expansion fluorides and silver contribute to increased thermal expansion. The expansion curves for PM212 and the carbide matrix material superimpose on figure 13(a) up to about 550 °C, but diverge at higher temperatures as the high expansion fluorides and silver begin to influence the thermal expansion of the composites.

A significant amount of compressive residual stress was indicated for the HIPed materials by hysteresis loops in the expansion/contraction curves during the first heating and cooling cycle of the materials. As shown for PM212 (CH), (fig. 13(b)), residual stress was relived by the first thermal cycle and little or no hysteresis was observed during subsequent thermal cycling. A much narrower hysteresis loop was observed for the bonded carbide during the first heating/cooling cycle. No hysteresis was noted during heating and cooling of the sintered materials, indicating that as expected, there was no residual stress in these materials after sintering at 1100 °C. In the compression tests, preheating of the HIPed materials removed residual stress prior to compression.

Since, residual stresses in fabricated parts could cause problems with dimensional stability HIPed parts should be annealed prior to finishing to final dimensions.

Thermal Conductivity

In multiphase systems, a continuous phase will often dominate the thermal conductivity mechanism by providing a continuous path for heat flow through the material. The continuous phases in PM212 are the carbide matrix material and the fluoride eutectic. These are relatively low conductivity materials. The fact that the thermal conductivity of PM212 (CH) is greater than it is in PM212 (CS) can be explained by the difference in the porosity of the two materials. Porosity in the sintered material results in reduced thermal diffusivity and thereby reduced thermal conductivity.

Discussion of Comparative Properties

PM212 must have adequate strength to be considered as a candidate material for self-lubricating bearings, bushings and seals. The material comparison data in table VIII(a) and figure 15 shows that the room temperature compressive strength of PM212 (CH), which is 937 MPa (136 ksi) and of PM212 (CS) which is 346 MPa (50 ksi) considerably exceed those of such common bearing materials as typical grades of mechanical carbon graphite and bearing bronze. Appreciable compressive strength is retained up to about 760 °C: 318 MPa, (50 ksi), for HIPed PM212 and 95 MPa, (14 ksi), for sintered PM212. These high temperature values are as high as the room temperature compressive strength of some bearing bronzes. This suggests that PM212 may have bearing load capacity at high temperature equivalent to that of bearing bronze at much lower temperature. This however, remains to be demonstrated in actual bearing tests of these new materials. Both modifications have a compressive strength of about 70 MPa (10 ksi) at 900 °C.

Table VIII(b) lists thermal expansion coefficients of various materials over two temperature spans. The thermal expansion of fully-dense PM212 (CH) is about 15 percent lower than the nickel base alloys listed. A rule of mixtures calculation was made in which the products of the volume fractions and the expansion coefficients of the components of PM212 were summed to estimate the expansion coefficient of the composite material. The calculated value was approximately the same as the published values for the super alloys. Therefore, the experimentally determined expansion coefficient of PM212 appears to be disproportionately influenced by the lower expansion materials such as chromium carbide, especially at temperatures below 500 °C.

The compressive strength and temperature capabilities of PM212 are compared to some common bearing grades of carbon and bronze in figure 15. This figure shows that the compressive strength of PM212 to at least $700\,^{\circ}\mathrm{C}$ is comparable to the room temperature strength of typical, conventional sliding contact bearing materials, and in addition, can be used to much higher temperatures.

Thermal conductivity is also an important property of bearing materials. Table VIII(c) compares the thermal conductivity temperature of PM212 to those of other materials. At room temperature, the thermal conductivities of both modifications of PM212 are comparable to those of materials such as boron carbide and austenitic stainless steel, somewhat lower than for metals such as cast iron and bearing bronze, and much lower than for highly conductive metals such as silver, copper, and aluminum. Unlike most of the other materials listed in table VIII, the thermal conductivity of PM212 increases with temperature. At about 900 °C, its thermal conductivity is somewhat higher than the values for aluminum oxide and some grades of silicon carbide.

SUMMARY OF RESULTS

- 1. The compressive strength of both forms of PM212 remains fairly constant between 25 and 350 °C. The strength of these materials is of course reduced at high temperature, but HIPed PM212 retains a compressive strength of 318 MPa at 760 °C and 67 MPa (10 ksi) at 900 °C.
- 2. HIPed PM212 is approximately three times stronger in compression than sintered PM212. The porosity in sintered PM212 is largely responsible for this strength difference.

- 3. Sintered PM212 is less porous than sintered bonded carbide baseline material. The silver and fluoride eutectic in PM212 may increase the density by reducing interparticle friction during compaction.
- 4. HIPed PM212 and HIPed 430NS are fully dense. In these fully dense products PM212 is weaker than the baseline bonded carbide because the silver and fluoride eutectic reduce the strength of the bonded carbide matrix.
- 5. In general, PM212 displays low to moderate thermal conductivity. The thermal conductivity of PM212 is comparable to the values for austenitic stainless steels.
- 6. The thermal expansion of PM212 displays two approximately linear regions of 25 to 550 °C and 550 to 850 °C. The high expansion coefficients of silver and the fluoride eutectic may be responsible for this behavior. The avereage expansion coefficients for PM212 over these two temperature ranges are 12.5 and 18.0×10^{-6} °C; respectively. The average expansion coefficient from 25 to 850 °C is 14.1×10^{-6} °C.
- 7. The tensile strength of both forms of PM212 is much lower than their compressive strength. Sintered PM212 in particular, is not suitable for supporting appreciable tensile loads.
- 8. The acoustically measured room temperature elastic moduli of sintered PM212 and HIPed PM212 are 95 GPa (14 ksi) and 175 GPa (25 ksi) respectively.

CONCLUSIONS

The results of this study show that both hot isostatically pressed and sintered PM212 have sufficient compressive strength and acceptable thermophysical properties for their serious consideration as sliding contact bearing and seal materials for use to at least 760 °C. The HIPed version may be suitable to 900 °C, especially for lightly loaded bearings and seals.

ACKNOWLEDGMENTS

The authors wish to acknowledge the indispensable support of a number of NASA personnel in accomplishing this project. Dr. John Gayda and Darrell Gaydosh measured and evaluated the tensile properties, Dr. Edward Generazio employed the ultrasonic method to determine the elastic moduli, and Anna Palczer was responsible for the sintering operation and measurements of linear thermal expansion.

REFERENCES

- 1. Sliney, H.E.: Carbide/Fluoride/Silver Self-Lubricating Composite; U.S. Patent 4,728,448, Useful at Low and High Temperatures. Mar. 1, 1988.
- Sliney, H.E.; and DellaCorte, C.: Method of Making Carbide/Fluoride/ Silver Composites. U.S. Patent Application 571,058, Aug. 1990.
- 3. DellaCorte, C.; and Sliney, H.E.: Composition Optimization of Self-Lubricating Chromium-Carbide-Based Composite Coatings for Use to 760 °C. ASLE Trans., vol. 30, no. 1, Jan. 1987, pp. 77-83.
- 4. Sliney, H.E.: Hot Piston Ring/Cylinder Liner Materials Selection and Evaluation. SAE Paper 880544, 1989 (also, NASA TM-100276).

- DellaCorte, C.; and Sliney, H.E.: Tribological Properties of PM212: A High Temperature, Self-Lubricating, Powder Metallurgy Composite. NASA TM-102355, 1989.
- Sliney, H.E.: The Use of Silver in Self-Lubricating Coatings for Extreme Temperatures. ASLE Trans., vol. 29, no. 3, July 1985, pp. 370-376.
- 7. Sliney, H.E.; Strom, T.N.; and Allen, G.P.: Fluoride Solid Lubricants for Extreme Temperatures and Corrosive Environments. ASLE Trans., vol. 8, no. 4, Oct. 1965, pp. 307-322.
- 8. Generazio, E.R.; Roth, D.J.; and Baaklini, G.Y.: Acoustic Imaging of Subtle Porosity Variations in Ceramics. Mater. Eval. vol. 46, no. 10, Sept. 1988, pp. 1338-1343.
- 9. Hinsley, J.F.: Non-Destructive Testing. MacDonald & Evans, Ltd., London, 1959.
- 10. Standard Test Method for Specific Heat of Aircraft Turbine Lubricants by Thermal Analysis. ASTM Standard D3947-86, ASTM, Philadelphia, PA, 1990.
- 11. Parker, W.J., et al.: Flash Method of Determining Thermal Diffusivity, Heat Capacity, and Thermal Conductivity. J. Appl. Phys., vol. 32, no. 9, Sept. 1961, pp. 1679-1684.
- 12. Deadmore, D.L.; and Sliney, H.E.: Hardness of CaF₂ and BaF₂ Solid Lubricants at 25 to 670 °C. NASA TM-88979, 1987.
- 13. Generazio, E.R.; Stang, D.B.; and Roth, D.J.: Dynamic Porosity Variations in Ceramics. NASA TM-101340, 1988.

TABLE I. - COMPONENTS OF PM212

Compo- nent	Compo- sition, wt %	Composi- tion, vol %	Particle size U.S. Sieve No. (#m)	MP ¹ °C	Hardness, Hv kg/mm ²
Comp	onent A:	Bonded Chro	mium Carbide: 70	wt % of	PM212
Cr ₃ C ₂	45	47		1895	^a 1300
Ni	28	22		1455	^a 570
Со	12	10			
Cr	9	9			
Мо	2	1	-200 + 400 (35 to 74)		
Al	2	5	, i		
В	1	3			
Si	1	3			
Component B: Silver Metal: 15 wt % of P					2
Ag	100	100	-100 + 325 (44 to 150)	961	^c 25
C	omponent C	: Prefused	Eutectic: 15 wt	% of PM	212
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 CaF₂ and BaF₂ Solid Lubricants at 25 to 670 °C," NASA TM-88979, March 1987.

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

TABLE II. - COMPRESSIVE STRENGTH DATA FOR PM212 AND 430NS 2.1x10⁻⁴ s⁻¹ STANDARD STRAIN RATE

Property	Tempera- ture, °C	PM212-CS	PM212-CH	430NS-CS	430NS-CH
Proportional limit, MPa	25 350 760 900	324±7 320±12 77±6 13±7	807±26 730±64 304±24 34±15	42±3 44±3 37±2 16±1	1288±251 817±12 331±3
0.2 percent yield stress, MPa	25 350 760 900	346±3 334±13 95±4 20±8	937±21 840±16 318±29 67±25	46±2 50±3 44±2 19±1	1961±85 905±28 361±4
Maximum stress, MPa	25 350 760 900	346±3 334±13 109±6 35±2	967±23 851±19 321±31 107±38	47±1 52±1 55±3 39±1	2022±88 1048±9 386±5
Plastic strain at maximum stress	25 350 760 900	0.002±.0003 .002±0 .010±0 .027±.009	0.004±.0007 .003±.0003 .003±.0013 .018±.0034	0.004±.003 .006±.004 .027±.007 .086±.005	0.003±.001 .012±0 .011±.002

TABLE III. - COMPRESSSIVE TESTS CONDUCTED AT VARYING STRAIN RATES AT 760 °C

Strain rate, s ⁻¹	0.2 Percent yield stress, MPa		1	Maximum stress, MPa		Strain at Maximum stress	
	PM212-CS	PM212-CH	PM212-CS	PM212-CH	PM212-CS	PM212-CH	
2.1x10 ⁻³	131	399	142	409	0.008	0.006	
2.1x10 ⁻⁴	95	318	109	321	0.010	0.003	
2.1x10 ⁻⁵	77	215	95	240	0.013	0.012	
2.1x10 ⁻⁶	54	104	71	126	0.019	0.013	
2.1x10 ⁻⁷		64		85		0.029	

TABLE IV. - TENSILE TEST DATA FOR PM212

Test tem- perature,		strength, MPa	Elongat perce	
°c	cs	СН	CS	СН
25	45	118	0	0
550	26	118	0	0
760	25	84	0.1	0

CS: PM212-CS CH: PM212-CH

TABLE V. - ACOUSTIC MEASUREMENT
OF YOUNG'S MODULUS

[Test temperature: 25 °C.]

Material	Velocity, m/s	Young	's modulus
		GPa	Psi
PM212-CS	4282	94	13.6×10 ⁶
	4257	93	13.5
	4331	96	13.9
PM212-CH	5144	174	25.2×10 ⁶
	5142	174	25.2
	5200	178	25.8

TABLE VI. - LINEAR COEFFICIENT OF THERMAL EXPANSION OF PM212 AND COMPONENTS

(Test conditions: nitrogen atmosphere)

Material	Coefficient of thermal expansion, °C ⁻¹	Temperature range, °C
PM212 (CH)	12.2×10 ⁻⁶ 18.7 14.4	25 to 550 550 to 850 25 to 850
PM212 (CS)	12.7 17.2 13.8	25 to 500 550 to 850 25 to 850
430NS (CH)	12.2 14.3 13.3	25 to 550 550 to 850 25 to 850
Ag CaF ₂ BaF ₂ Cr ₃ C ₂ Ni	28.4 36.6 25.8 12.1 18.3	^a 25 to 900 ^a 25 to 627 ^a 25 to 577 ^a 25 to 927 ^a 25 to 927

Thermophysical Properties of Matter, Vol. 12, Thermal Expansion - Metallic elements and Alloys, Touloukain, Y.S., et al. IFI/Plenum, NY 1975.

TABLE VII. - THERMAL CONDUCTIVITY CALCULATIONS

		IAI	IABLE VII IMERMAL CONDUCTIVITI CALCULATIONS	L CONDUCTIVILI	CALCULATIONS		
Sample	Temper- ature, °C	Density, gm, cm ⁻³	Specific Heat, W, s, gm ⁻¹ , K ⁻¹	Diffusivity, cm², sec ⁻¹	Conductivity W, cm ⁻¹ , K ¹	Conductivity, BTU units ^a	Temper- ature, °F
	23.0	5.141	0.4780	0.04050	0.09952	69.01	73.4
	100.0		.5150	.04030	.10670	73.98	212.0
	200.0		.5410	.04070	.11320	78.49	392.0
	300.0		.5570	.04140	.11855	82.20	572.0
PM212-CS	400.0		.5750	.04300	.12711	88.13	752.0
	500.0		.6020	.04350	.13463	93.34	932.0
	0.009		.6280	.04560	.14722	102.08	1112.0
	700.0		.6570	.04670	.15774	109.37	1292.0
	800.0		0569.	.04670	.16686	115.69	1472.0
	0.006	>	.7480	.04430	.17035	118.11	1652.0
	23.0	6.566	0.4870	0.04240	0.13558	94.00	73.4
	100.0		.5270	.04240	.14672	101.73	212.0
	200.0		.5570	.04310	.15763	109.29	392.0
	300.0		.5730	.04470	.16818	116.60	572.0
PM212-CH	400.0		.5870	.04670	.17999	124.80	752.0
	500.0		.6130	.04830	.19441	134.79	932.0
	0.009		.6490	.04930	.21008	145.66	1112.0
	700.0	-	.6820	.05050	.22614	156.79	1292.0
	800.0		.7190	.05120	.24171	167.59	1472.0
	0.006	٨	.7600	.04860	.24252	168.15	1652.0

^aBTU in hr^{-1} , ft⁻², F^{-1} .

TABLE VIII(a). - COMPARATIVE PROPERTIES OF SELECTED MATERIALS

ູບ MECHANICAL PROPERTIES at 25

Material	Compressiv	Compressive strength	Tensile	strength	Modulus of	elasticity
	MPa	ksi	MPa	ksi	10 ⁴ MPa	10 ⁶ psi
ິວະມວ	3100	450	248	36		
Alumina	1800	261	170	25	36.5	^a 53
Inco 718	i 	 	1241	180	20.0	62 _q
Inco x -750			1200	174	21.4	^b 31
Stellite 6B	1 1 1	1	1034	150	21.4	°31
PM212 (CH)	937	136	118	17	17.5	° 25
440 SS		1	1966	285	20.0	6Z _q
304 SS		!	009	87	19.3	^b 28
Tic	752	109	1	1	31.0	^a 45
PM212 (CS)	346	20	45	6.5	9.7	*14
Bearing bronze	172	25	241	3.5	!	g P
Carbon/graphite	241	35	21	3.0	6.0	a _{1.3}
4 53						

Kingery, W.D., Bowery, H.K., and Uhlmann: "Introduction to Ceramics," John

Wiley & Sons, NY 1976.

^bMaterials Selector 1990, Materials Engineering, Penton Publ., Cleveland, Ohio, December 1989.

Manufacturing Data, "Haynes Alloys 6B and 6K," Bulletin H 3043.

Cast Bearing Bronze Design Manual, Rippel, H.C., Cast Bronze Bearing Institute, Inc., Cleveland, OH, 1960. This work.

TABLE VIII.(b) - THERMAL LINEAR EXPANSION COEFFICIENTS

Materials	Expansion co	efficient °C ⁻¹
	20 to 627 °C	20 to 927 °C
CaF ₂ BaF ₂ Ag Cu Ni Ni-Cr Alloys PM212 (CH) PM212 (CS) Diamond Cr ₃ C ₂ TiN Al ₂ O ₃ TiC ZrO ₂ B ₄ C αSiC Si ₃ N ₄	36.6×10 ⁻⁶ 25.8 24.8 21.3 17.1 14 to 16 12.2 (20 to 550 °C) 12.7 (20 to 550 °C) 10.8 9.7 8.8 7.9 6.4 5.5 5.1 3.4	28.4x10 ⁻⁶ 24.9 18.3 ^a 16 to 18 ^b 14.4 (20 to 850 °C) ^b 13.8 (20 to 850 °C) 12.1 10.3 9.6 8.6 8.9 6.3 5.6 3.6

From: Thermophysical Properties of Matter, Vol. 12
Thermal Expansion - Metallic Elements and Alloys,
Touloukian, Y.S., Kirby, R.K., Taylor, R.E., and
Desai, P.D., IFI/Plenum, NY, 1975.

And, Vol. 13, Thermal Expansion Nonmetallic Solids, Touloukian, Kirby, Taylor and Lee, T.Y.R., 1977.

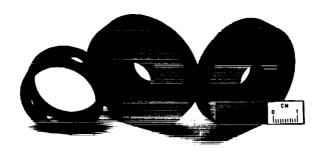
Except: ^aManufacturing data.
^bThis work.

TABLE VIII.(c) - THERMAL CONDUCTIVITY

Materials	Thermal con	nductivity, wcm ⁻¹ k ⁻¹
	25 °C	927 °C
Diamond	9.0	
Ag	4.3	3.6
Cu	4.0	3.4
Au	3.2	2.6
Al	2.4	
SiC	0.3 to 2.0	0.14 to 0.6
Carbon/graphite	1.3	.55
Cr	0.91	.62
Ni	.90	.76
Gray cast iron	.80	
Bearing bronze	.80	
Die steel	.50	
Al ₂ O ₃	.36	.07
TiĆ 3	.30	.50
TiN	.25	.25
BC	.20	
304ss	.16	a.21 (500 °C)
PM212 (CH)	.13	b.24
Ni-Cr Superalloy	.11 to .12	a.24 to .27
PM212 (CS)	.10	b.17
BaF ₂	.07	
CaF ₂	.07	.02
Si_N_	.05	.07
Zro ₂ 4	.018	.02

From: Thermophysical Properties of Matter, Vol. 1 and 2.
Thermal Conductivity, Touloukian, Y.S., Powell, R.W.,
Ho, C.Y., and Kleinens, P.G., IFI/Plenum, N.Y., 1970.

Except: ^aManufacturing data.
^bThis work.



C-90-00100

Figure 1.—PM212 parts fabricated by electrical discharge machining (EDM) with rub surfaces final machined by diamond grinding.

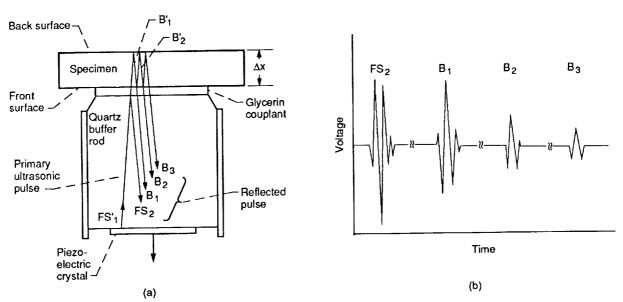
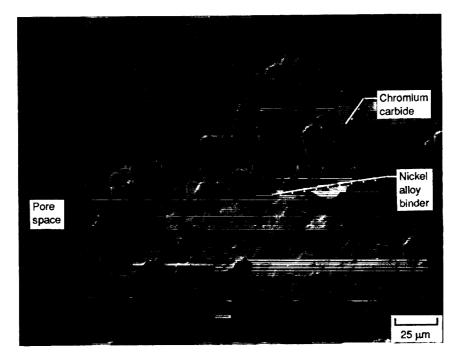
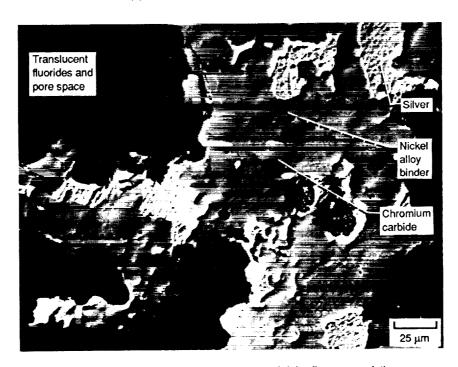


Figure 2.—Ultrasonic apparatus for Young's Modulus determination.

ORIGINAL PAGE BLACK AND WHITE PHOTOGRAPH



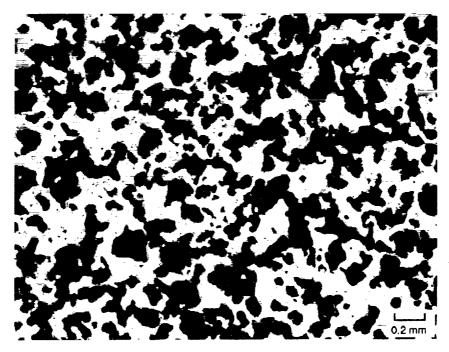
(a) Sintered 430NS. (50 percent porostiy)



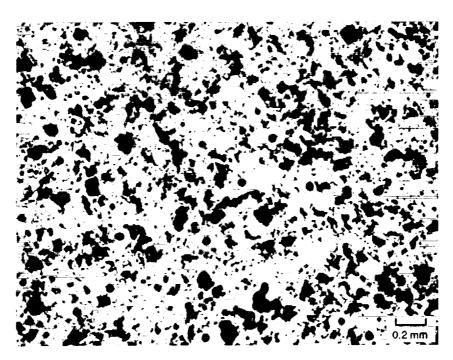
(b) Sintered PM212. (20 percent porosity) Note bright silver accumulations.

Figure 3.—Optical micrographs.

ORIGINAL PAGE BLACK AND WHITE PHOTOGRAPH

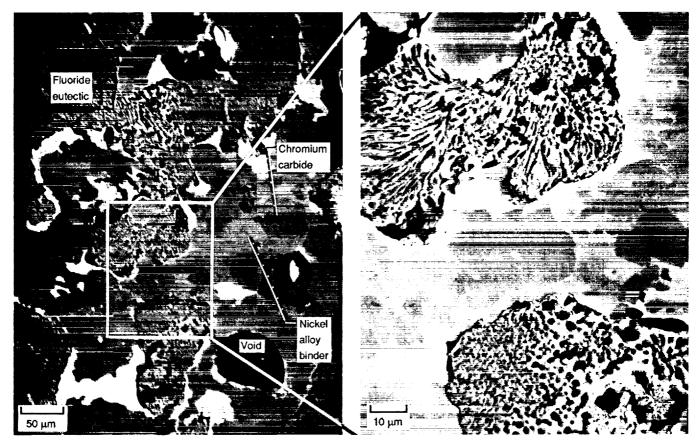


(a) Sintered PM212-CS.



(b) HIPed PM212-CH.

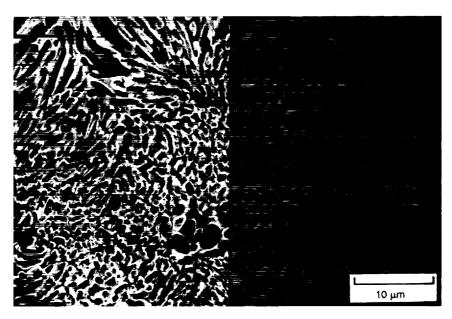
Figure 4.—Illustration of density difference.



(a) Distribution of components

(b) Distribution of components.

ORIGINAL PAGE BLACK AND WHITE PHOTOGRAPH



(c) Lamellar structures of ${\rm CaF_2/BaF_2}$ eutectic.

Figure 5.—Microstructure of sintered PM212 by back scatter scanning electron microscopy.

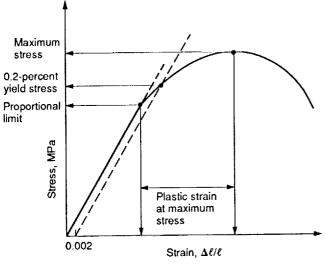


Figure 6.—Idealized stress/strain curve.

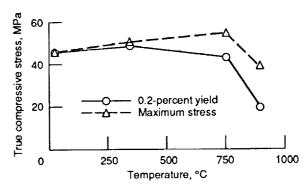


Figure 7.—43ONS-CS ∞mpressive strength versus temperature.

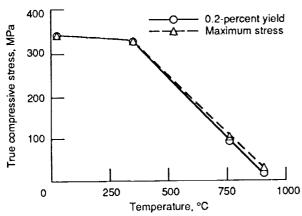


Figure 8.—PM212-CS compressive strength versus temperature.

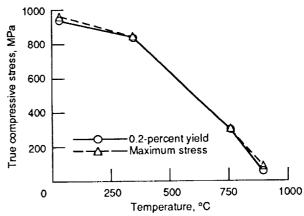


Figure 9.—PM212-CH compressive strength versus temperature.

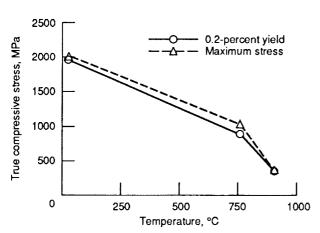


Figure 10.—43ONS-CH compressive strength versus temperature.

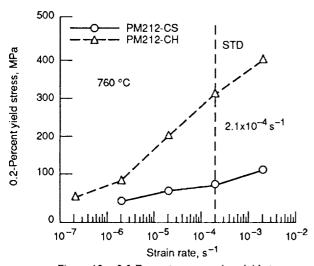


Figure 12.—0.2-Percent compressive yield stress versus strain rate for PM212-CS and PM212-CH at 760 °C.

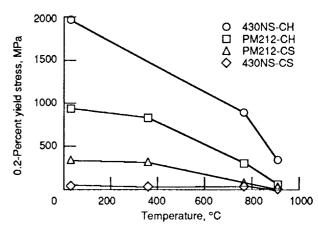


Figure 11.—0.2-Percent compressive yield strength comparison of test materials.

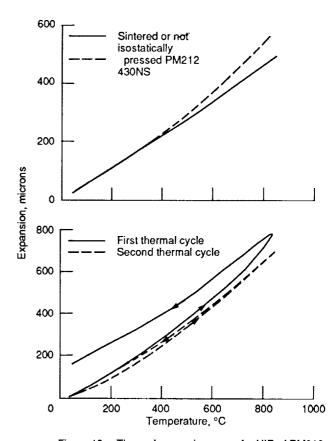


Figure 13.—Thermal expansion curve for HIPed PM212 showing presence of residual stress during first thermal cycle and essentially strain-relieved condition during subsequent thermal cycle.

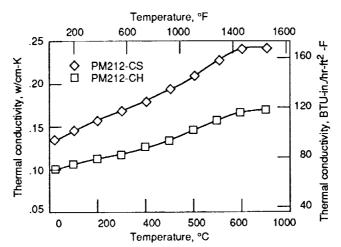


Figure 14.—Thermal conductivity versus temperature: PM212-CS and PM212-CH.

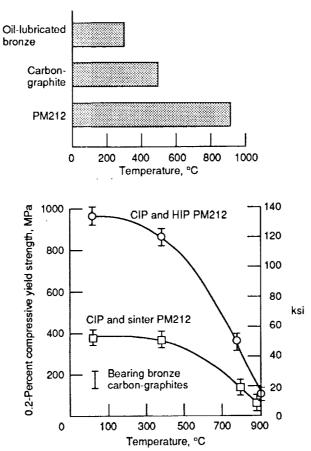


Figure 15.—Maximum service temperature and compressive strength comparison of self-lubricating bearing materials.

National Aeronautics and Space Administration	R	eport Documenta	ation Page	9	
1. Report No. NASA TM-103694 DOE/NASA/50162-5	5	2. Government Accession I	No.	3. Recipient's Catalo	g No.
Title and Subtitle				5. Report Date	
Mechanical Strength and Therm	ophysi	cal Properties of PM212	A High	December 1990)
Temperature Self-Lubricating P	owder	Metallurgy Composite		6. Performing Organi	zation Code
7. Author(s)				Performing Organi	zation Report No.
Phillip M. Edwards, Harold E.			, J. Daniel	E-5913	
Whittenberger, and Robert R. !	Martine	au		10. Work Unit No.	
				778-34-22	
9. Performing Organization Name and A				11. Contract or Grant	No.
National Aeronautics and Space	Admii	nistration			
Lewis Research Center Cleveland, Ohio 44135-3191				13. Type of Report an	d Period Covered
				Technical Mem	
2. Sponsoring Agency Name and Addres	5				
U.S. Department of Energy Office of Vehicle and Engine R Washington, D.C. 20546	&D			14. Sponsoring Agenc	y Code
Supplementary Notes					
6. Abstract A powder metallurgy composite	(DM2	12) composed of metal-	bonded chromi	ium carbide and soli	d lubricants is
known to be self-lubricating to a compressive strength, tensile st sliding contact bearings and sea Other properties presented are appear to have adequate strength is fully dense, is much stronger material is less costly to make,	maxim rength, als are proom to be of than the	num application temperature thermal expansion and the treported for sintered and temperature density, hard considered as sliding content of the sintered version which the temperature considered version which the temperature is the sintered version temperature.	re of 900 °C. hermal conduction isostatically process, and elast act bearing materials about 1	In this paper, the high tivity data needed to ressed (HIPed) version modulus. In generaterials, but the HIPed percent pore volumes.	temperature of design PM212 dons of PM212. Tral, both versions of the version, which me. The sintered
17. Key Words (Suggested by Author(s))		18.	Distribution State		
Sintered high-temperature beari	ngs			- Unlimited egories 27, 23	
Self-lubricating composites Mechanical strength of PM bea	ring m	aterial	•	ory UC – 336	
Thermophysical properties of P			8	•	
Security Classif. (of this report)		20. Security Classif. (of this	page)	21. No. of pages	22. Price*
Unclassified		Unclassif		28	A03