# Martin

[45] Oct. 16, 1973

[54]	IN SITU FLUORINATION OF GRAPHITE IN IRON ALLOY		3,567,521 3,607,747	3/1971 9/1971	Toy et al	
[75]	Inventor:	Jerry Thomas Martin, Plainview, Minn.	Primary Examiner—Murray Katz Assistant Examiner—Dennis C. Konopacki Attorney—Robert W. Lahtinen et al.			
[73]	Assignee:	International Business Machines Corporation, Armonk, N.Y.				
[22]	Filed:	Mar. 31, 1972				
[21]	Appl. No.	: 240,268	[57]		ABSTRACT	
[52] [51]	U.S. Cl	A self lubricating bearing surface is formed on high carbon iron alloy parts by fluorinating the exposed carbon particles at the surface of the part to form carbon monofluoride. The part is fluorinated in a reac-				
[58]	3 JAN 19 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			tion chamber by exposure to fluorine gas not exceeding a partial pressure of 76 torr in an inert gas and a temperature not exceeding 700° centigrade to substan-		
[56]	References Cited tially completely convert all exposed carbon particle to carbon monofluoride.					
3,091,	549 5/19	63 Kanter 117/75		10 CI	aims, No Drawings	

# IN SITU FLUORINATION OF GRAPHITE IN IRON **ALLOY**

## BACKGROUND OF THE INVENTION

This invention pertains to bearings and more particu- 5 larly to creating self-lubricating bearing surfaces at the surface of high carbon alloy iron materials by fluorinating the carbon particles exposed at the surface to form carbon monofluoride.

Many plating and other surfacing techniques are uti- 10 lized to enhance the wear and lubricating qualities of bearing surfaces. In the method of the present invention a self-lubricating bearing surface is created at the surface of high carbon alloy materials such as cast iron carbon particles to form carbon monofluoride.

The present invention provides a self-lubricating bearing on a cast iron surface by converting the exposed particles of free carbon to carbon monofluoride. A part formed of high carbon iron alloy is machined to 20 a desired dimension with an allowance for a slight growth of the carbon particles along the surface during the fluorination process. This compensation is necessary to avoid any post fluorination surface treatment face is a machined cast iron surface with exposed particles of carbon monofluoride that project a few ten thousandths of an inch from the surrounding iron matrix and form the self-lubricating bearing surface.

### DESCRIPTION

The surface of a high carbon iron alloy to be treated is prepared by machining the surface with accommodation for material growth where appropriate. For example, it is has been found necessary when practicing the 35 present invention to oversize a % bore by 0.0005 of an inch to accommodate the material growth during the conversion of carbon to carbon monofluoride.

The prepared part is placed in a nickel or nickel lined chamber for the fluorination process. On the first occasion that fluoride gas is introduced into the chamber, the fluoride promptly forms a film of nickel fluoride along the surface of the chamber which is adherent, invisible and which precludes further reaction between the nickel surface and the fluorine gas.

The chamber is thereupon purged of oxygen and moisture by the introduction of an inert gas such as helium or nitrogen. The chamber is also slowly heated simultaneously with the purge to a temperature usually not exceeding 200° centigrade to assure that any moisture is vaporized and removed from the chamber. When the purge is complete, fluorine gas is introduced as a partial pressure in an inert gas such as helium or nitrogen. To control the reaction, causing carbon to be converted to carbon monofluoride while not reacting with the iron, the concentration of fluorine in the inert gas (at atmospheric pressures) is not allowed to exceed a 10 per cent partial pressure and the usual practice is to use a partial pressure in the range of 3 to 4 percent or a partial pressure of about 25 torr. During fluorination, the temperature is elevated to a value not exceeding 700° centigrade. The temperature limitation likewise is imposed to assure that the fluorine will react selectively with the carbon particles and to the exclusion of the iron.

The fluorination process is continued until the exposed graphite or carbon particles resident at the surface are converted to carbon monofluoride. When the process has been completed, the part is removed from the chamber and rinsed in a mild alkaline solution to neutralize any hydrofluoric acid that might be resident on the surface and is thereafter dried by exposure to a forced flow of air. To prevent degradation of the surface during subsequent shipment or prior to assembly, the part is dipped in a water replacing oil to remove any residual moisture.

### EXAMPLE 1

A high carbon alloy sample is placed in a 1,000 cc nickel lined reaction chamber. The chamber is thereupon purged with helium at the rate of 50 cc's per minhaving free carbon particles exposed, by reacting such 15 ute for a period of 1 hour to remove moisture and oxygen while slowly heating the chamber to 200° centigrade. Fluorine is thereafter introduced into the chamber at a partial pressure of 25 torr in helium at a rate of 50 cc of the gas mixture per minute and the temperature is simultaneously gradually raised to 640° C. Then the introduction of the gas mixture is continued for a period of 1 hour, while maintaining the reaction chamber at a temperature of 640° centigrade. The sample is then removed and rinsed in a mildly alkaline water sothat would involve material removal. The resulting sur- 25 lution to neutralize any HF present and thereafter dried in blowing air. The sample is finally dipped in a water replacing oil in preparation for storage and shipment.

# **EXAMPLE 2**

A high carbon iron alloy sample with exposed particles at the surface is placed in a 1,000 cc nickel lined reaction chamber. The chamber is thereafter purged with nitrogen at the rate of 50 cc's per minute for a period of 1 hour to remove moisture and oxygen while slowly heating the chamber to 200° centigrade. Fluorine is then introduced into the chamber at a partial pressure of 25 torr in nitrogen at a rate of 50 cc of the gas mixture per minute while simultaneously, gradually raising the chamber temperature to 640° C. Thereafter the introduction of the gas mixture is continued for a period of 1 hour while maintaining the reaction chamber at 640° centrigrade. The sample is then removed and rinsed in a mildly alkaline water solution to neutralize any HF present and thereafter dried in blowing air. The sample is thereupon treated with a water replacing oil in preparation for storage and shipment.

# **EXAMPLE 3**

A high carbon iron alloy sample is placed in a 1,000 cc nickel lined reaction chamber. The chamber is then purged by the introduction of helium at the rate of 50 cc's per minute for a period of 1 hour to remove moisture and oxygen while slowly heating the chamber to 200° centigrade. Fluorine is thereupon introduced into the chamber at a partial pressure of 25 torr in helium at the rate of 50 cc per minute while simultaneously, gradually raising the reaction chamber temperature to 400° C. Thereafter the introduction of the gas mixture is continued for a period of 4 hours while maintaining the reaction chamber at a temperature of 400° centigrade. The sample is then removed and rinsed in a mildly alkaline solution to neutralize any HF present and thereafter dried in blowing air. The sample is fi-65 nally dipped in a water replacing oil in preparation for

In addition to materials such as cast iron, selflubricating bearing surfaces may be formed on other parts composed of iron and presenting free carbon particles at the surface in accordance with the above described technique. By way of example, powdered metal parts which possess free carbon particles could be similarly treated.

While the invention has been particularly shown and described with reference to a preferred embodiment thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of 10 the invention.

What is claimed is:

1. The method of forming a self-lubricating bearing surface on a high carbon iron alloy part comprising placing said part in a reaction chamber which is resistant to reaction with fluorine gas; purging said reaction chamber with an inert gas to remove substantially all moisture and oxygen; and introducing fluorine gas at a partial pressure not exceeding 76 torr in an inert gas while maintaining said chamber at a temperature not 20 exceeding 700° centigrade until the exposed carbon particles at the surface of said part are substantially completely converted to carbon monofluoride.

2. The method of claim 1 further comprising slowly heating said chamber to a temperature in excess of 25 100° centigrade during said purging step to induce vaporization of moisture.

3. The method of claim 2 wherein the inert gas utilized during said purging and fluorinating steps is helium.

4. The method of claim 2 wherein the inert gas utilized during said purging and fluorinating steps is nitro-

gen.

5. The method of claim 2 further comprising the step of rinsing said part in a mild alkaline solution after said carbon particles exposed at the part surface have been substantially completely converted to carbon monofluoride.

6. The method of claim 5 further comprising the step of drying said part following the rinsing thereof and applying a water replacing oil to the surface thereof.

7. The method of claim 6 wherein said reaction chamber has a nickel surface which develops a reaction resistant nickel fluoride film thereon in the presence of fluorine gas.

8. The method of forming a self-lubricating bearing surface on a high carbon iron alloy part comprising placing said part in a reaction chamber that is resistant to fluorine gas; purging said reaction chamber with an inert gas while slowly raising the temperature of said chamber to an excess of 100° centigrade to remove substantially all moisture and oxygen therefrom; and introducing fluorine gas at a partial pressure not exceeding 10 percent of the total pressure in an inert gas into said reaction chamber while not exceeding a temperature 700° centigrade until the carbon particles exposed at the surface of said part are substantially completely converted to carbon monofluoride.

9. The method of claim 8 wherein said inert gas is helium.

10. The method of claim 8 wherein said inert gas is nitrogen.

35

40

45

50

55

60