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[54] EROSION RESISTANT WARES COMPOSED PREDOMINANTLY OF CHROMIUM BEARING STEEL

[75] Inventor: **Monroe J. Hordon**, Newton, Mass.

[73] Assignee: **National Research Corporation**, Cambridge, Mass.

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[58] **Field of Search** .... 29/195 A

[56] **References Cited**

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Primary Examiner—Hyland Bizot
Attorney, Agent, or Firm—Kenneth W. Brown; Barry R. Blaker; Lawrence A. Chaletsky

[57] **ABSTRACT**

Chromium bearing steel wares are rendered resistant to aerodynamic erosion by multiple coating thereof. The outermost coating comprises boron carbide.

6 Claims, No Drawings

EROSION RESISTANT WARES COMPOSED PREDOMINANTLY OF CHROMIUM BEARING STEEL

FIELD OF THE INVENTION AND PRIOR ART

In present aircraft design and manufacture it is often necessary or desirable to construct certain parts of the engine or airframe from chromium bearing ferro-alloys including, specifically, stainless steels. Accordingly, various leading edge structures for airfoil surfaces such as wings, rotors, vertical and horizontal stabilizers; thrust reversers; nacelle inlets; spoilers; exhaust deflectors; fairings; etc. are often composed of stainless steels. So, too, are various turbine engine parts such as compressor blades and inlet guide vanes. Further, in various chemical processing equipment such as conduits, cyclones, bag filters and the like it is often found necessary to employ stainless steels for their characteristic of chemical inertness with respect to many chemicals and corrosive atmospheres.

A problem which is often encountered in the use of wares such as described above resides in the resistance thereof to aerodynamic erosion, i.e., the deleterious abrasive phenomenon arising when fluid entrained particulate solids are impacted at high velocities against the surfaces of such wares. It has been proposed, of course, to coat such wares with an extremely hard substance such as boron carbide. Unfortunately, such boron carbide coatings, when applied to chromium bearing ferroalloys in thicknesses greater than about 10 microns, are generally found to be severely deficient in adhesion to the substrate and are often quickly spalled and stripped from the substrate under aerodynamic erosion conditions. In accordance with the present invention, however, this adhesion problem has been substantially ameliorated and highly erosion resistant boron carbide coatings are provided on chromium bearing steel wares.

OBJECTS OF THE INVENTION

It is a principal object of the invention to provide novel erosion resistant chromium bearing wares.

It is another object of the invention to provide novel erosion resistant coatings for chromium bearing wares.

It is still another object of the invention to provide a novel method for rendering chromium bearing wares erosion resistant.

Other objects and advantages of the invention will in part be obvious and will in part appear hereinafter.

GENERAL DESCRIPTION OF THE INVENTION

In accordance with the present invention, chromium bearing steel wares are rendered erosion resistant by physical vacuum vapor deposition of sequential coatings thereon, said coatings comprising, seriatim, (1) nickel or iron, (2) titanium and (3boron carbide.

DETAILED DESCRIPTION OF THE INVENTION

The coatings of the invention are applied by the technique of physical vacuum vapor deposition (PVD) which technique broadly comprises the condensation of vapors of the respective coating materials onto the stainless steel substrate under vacuum. Such PVD processes are further characterized by the fact that the coating materials are vaporized by heating thereof such as by electron beam or induction heating techniques. Thus, PVD processes are readily distinguishable from chemical vapor deposition coating processes wherein an actual chemical conversion of a precursor material (such as BCl_3) to the ultimate coating (B_4C) takes place in the vapor phase at or near thp substrate surface. Further details of PVD coating processes may be had by reference to the following U.S. patent literature: No. 3,579,415, Allen et al., May 18, 1971; No. 3,460,976, Allen, Aug. 12, 1969; No. 3,414,655, Allen, Dec. 12, 1968; No. 3,551,247, Feakes, Dec. 29, 1970. Finally, it is much preferred that the chromium bearing steel ware undergoing the coating method of the invention be heated to above ambient temperature during the PVD coating thereof. Of course, said heating should not be so great as to deleteriously affect the strength of the ware. For the majority of chromium bearing steels, heating to a temperature of between 250 and 700°C during the PVD coating steps will generally be found satisfactory.

Chromium bearing steels suitable for coating by the process of the invention are those ferro-alloys comprising between about 5 and about 20 weight percent chromium. The invention is particularly applicable to wares composed of stainless steels comprising between 11 and 16 weight percent chromium. Specific examples of suitable stainless steel materials are: 410, 304, AM-355, V-57, W-545, CRM-6D, PH 15-7 Mo, Greek Ascoloy, Labelloy C, H-13, 314, AF 71, etc. Complete material balances of the above and other suitable stainless steels may be had by reference to the 1971 *SAE Handbook*, Society of Automotive Engineers, Inc., New York, N.Y., pages 240–243.

The initial nickel or iron coating may be construed as a flash or "prime" coating and may be relatively extremely thin, in other words, less than 1 micron and preferably from 0.05 to 0.20 micron in thickness. While the initial nickel or iron coating may be extremely thin, said coating is nevertheless of extreme importance in providing the improved adhesion characteristics of the overall coatings of the invention. While there is no intent to be bound by this explanation, it is thought that the nickel or iron coating interacts intimately with the tenacious complex oxides and spinels of the substrate chromium bearing steel surface, thereby to form a firmly adherent bond thereto while concomitantly presenting a fresh, clean and compatible surface to the titanium coating subsequently applied thereto.

The next layer of the triplex PVD coatings of the invention is formed of titanium metal and may have a thickness of from about 0.1 to about 25 microns. Generally speaking, the preferred thickness of the intermediate titanium coating will reside in the range of from about 10 to about 15 microns. The role played by the intermediate titanium metal coating is believed to be that of reduction of deleterious residual mechanical stresses induced by thermal expansion or dilation differences between a chromium based ferro-alloy substrate and a boron carbide coating applied thereto. Accordingly, the titanium coatings forming part of the overall coatings of the invention are believed to act as thermo-mechanical buffers between the chromium bearing steel substrate and the boron carbide exteriormost coating layer in the sense that titanium metal has a thermal coefficient of expansion intermediate that of the steel substrate and the boron carbide outer coating.

Also, it is believed that the titanium coating forms strong interfacial bonds with the underlying nickel or iron coating and with the boron carbide coating. In particular, it is known that boron carbide can react to some extent with titanium metal so as to form titanium boride and/or complex titanium-boron carbides. Said reactions are thought to play a beneficial role in providing the markedly improved adherent coatings of the invention.

The outermost layer of the coatings of the invention is composed of boron carbide having a total carbon content of between 15 and 30 atomic percent and preferably between 18 and 25 atomic percent. The thickness of the boron carbide coating can be between about 10 and about 50 microns and is preferably greater than about 20 microns.

There follows an illustrative, non-limiting example.

Example

Stainless steel plate specimens were coated with the triplex Ni/Ti/B_4C coatings of the invention employing a vacuum coating chamber equipped with two electron beam serviced copper crucibles, one of the crucibles containing a 99.7 percent pure boron carbide charge having a carbon concentration of about 23 atomic percent and the other crucible containing a charge of pure wrought titanium metal. Provision for the initial flash coating of nickel is made by positioning a 6 inch nickel plated tri-filament twisted tungsten wire about 4 inches over the boron carbide containing crucible. The wire is placed in series with an electric power source. The specimen undergoing the coating operation is cleaned by vapor blasting, placed in a heater equipped specimen holder suspended about 10 inches above the source crucibles and positioned so as to maintain the exposed surface of the specimen substantially parallel to the open ends of the crucibles. The vacuum chamber is closed to the atmosphere, pumped to a pressure of about 1×10^{-5} torr and the specimen heated to a temperature of between 400 and 500°C. Coating of the specimen is then undertaken employing the following regimen:

a. a flash coating of nickel is applied by flowing a current of between 0.6 to 1.2 KW power through the nickel coated tungsten wire for a period of about 4 minutes;

b. titanium deposition substantially immediately follows (a) by impinging a 2.4 KW electron beam onto the surface of the titanium charge contained in one of the crucibles for a period of 5 minutes; and

c. substantially immediately after (b) the electron beam serving the titanium source is shut down and the electron beam serving the boron carbide source initiated at a power input of 8 KW and for a period of 10 minutes.

Following the above coating procedure the specimen is cooled to about room temperature, in vacuo, the vacuum chamber vented to the atmosphere and the specimen removed from the vacuum chamber.

Control specimens were also produced comprising duplex coatings consisting of the titanium and boron carbide coatings only, i.e., without the initial flash coating of nickel. The coating procedures employed for the control coated specimens were essentially the same as outlined above for the production of the coatings of the instant invention with the sole exception that step (a) of the above procedure was not accomplished. Accordingly, the nickel plated tungsten wire was not activated.

Several specimens comprising the triplex coatings of the invention and the duplex Ti/B_4C control coatings were prepared from sample plates composed of 410, 304 and AM-355 steels. Type 304 steel is an austenitic nickel/chromium/iron/alloy steel having a chromium content of about 17–19 weight percent. Type 410 steel is a martensitic chromium steel having a chromium content of about 12.25 weight percent. AM-355 is a semiaustenitic precipitation hardening steel having a chromium content of about 15.5 weight percent. The thicknesses of the coatings applied to the triplex coated specimens were determined to be: nickel coating — about 0.1 micron; titanium intermediate coating — about 6.5 microns; boron carbide coatings — about 40 microns. In the case of the control duplex coated samples the titanium coating had a thickness of about 6.5 microns and the boron carbide coating a thickness of about 30 microns. Attempts to produce boron carbide coatings on the duplex coated specimens of equivalent thickness to those of the triplex coated specimens failed due to immediate lack of adhesion of the duplex coatings on the stainless steel specimens upon cooling thereof in the vacuum chamber. Accordingly, in the case of the control group, it was necessary to reduce the boron carbide coating thickness to the aforementioned 30 micron thickness in order to obtain specimens having reasonably adherent and defect-free coatings. The manner in which the thinner control group boron carbide coatings were produced on the duplex coated specimens was by reducing the time of coating from the original 10 minutes to about 7 minutes.

The resulting coated specimens were subjected to Scotch tape, boiling water immersion and erosion resistance tests. In the so-called "Scotch tape" test, a ½ by 2 inch strip of a commercially available celluloid mending tape is applied to the coated specimen, rolled manually so as to remove all air bubbles and provide a continuous interfacing between the coating and the tape adhesive and allowed to stand for about 1 minute. The tape is then stripped rapidly from the coating and any non-adherent portions of the coating are thereby stripped from the stainless steel substrate. Any amount of the metal carbide coating adhering to the tape is taken as a failure of the test.

The boiling water immersion test is undertaken simply by placing the coated specimens into a boiling demineralized water bath for a period of about 1 hour. Deficient coatings are at least partially removed by this treatment and their absence is readily noted upon removal of the specimens from the bath. Again, substantially any coating removal during the immersion period is construed as a failure of the test.

The accelerated erosion testing of the specimens is carried out by weighing the coated virgin specimens and then subjecting the coated surfaces to controlled sand blasting. In accomplishing this the coated surface of the specimen is held perpendicular to the axis of a boron carbide sand blasting nozzle at a stand-off distance of about 6 inches. The specimen is then blasted with silicon carbide grit having a particle size population ranging from about 25 to 125 microns for 10–100 seconds. The specimen is then cleaned lightly to remove adherent grit therefrom and re-weighed. The thickness of coating removed from the specimens due to erosion is calculated from the weight loss, taking into account the exposed surface area of the specimen under test. In the Table following, said erosion data is expressed in terms of the Erosion Improvement Factor which factor represents the ratios of erosion time required to remove 25.4 microns of the boron carbide coating compared to the time required to remove 25.4 microns of a bare stainless steel standard.

TABLE

Stainless Steel Substrate	Coating Type	Scotch Tape Test	Boiling Water Immersion Test	Erosion Improvement Factor
304	duplex Ti/B_4C	Fail	Fail	6.0
	triplex Ni/Ti/B_4C	Pass	Pass	6.7
410	duplex Ti/B_4C	Pass	Fail	3.5
	triplex Ni/Ti/B_4C	Pass	Pass	4.2
AM-355	duplex Ti/B_4C	Pass	Fail	2.8
	triplex Ni/Ti/B_4C	Pass	Pass	3.4

What is claimed is:

1. An erosion resistant ware composed predominantly of chromium bearing steel having a chromium content of between 5 and 20 weight percent and having coated over at least that portion of the surface thereof adapted for exposure to an aerodynamic erosion environment a coating comprising, seriatim:

a. a first metal layer bonded to said surface, said layer having a thickness of less than 1 micron and being composed of a metal chosen from the group consisting of nickel and iron;

b. an intermediate titanium metal layer bonded to said first layer and having a thickness of between 0.1 and 25 microns; and

c. an exteriormost layer of boron carbide bonded to said intermediate layer, said exteriormost layer having a total carbon concentration of between 15 and 30 atomic percent and a thickness of between 10 and 50 microns.

2. The coated ware of claim **1** wherein the coating layer of (a) is nickel and has a thickness of between 0.05 and 0.20 micron.

3. The coated ware of claim **1** wherein the intermediate titanium layer of (b) has a thickness of between 10 and 15 microns.

4. The coated ware of claim **1** wherein the boron carbide layer of (c) has a thickness of greater than 20 microns.

5. The coated ware of claim **1** wherein the boron carbide layer of (c) has a total carbon concentration of between 18 and 25 atomic percent.

6. The coated ware of claim **1** having the form of a turbine compressor blade.

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