

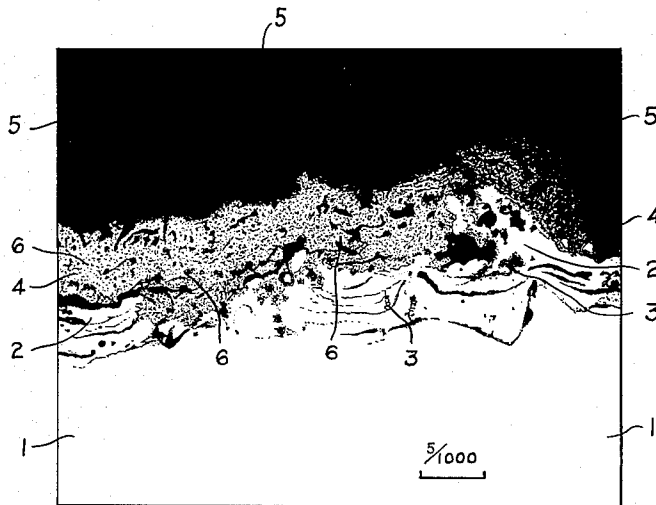
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OXIDE COATED ARTICLES WITH METAL UNDERCOAT

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OXIDE COATED ARTICLES WITH METAL UNDERCOAT

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Continuation of application Ser. No. 570,602, Mar. 9, 1956. This application May 17, 1963, Ser. No. 281,366 9 Claims. (Cl. 29—195)

The invention relates to oxide coated articles and provides an improvement therein and in the art of making them.

This application is a continuation of my application Serial No. 570,602 filed March 9, 1956, for Oxide Coated Articles with Metal Undercoat.

One object of the invention is to provide flame resistant articles. Another object of the invention is to provide metal articles coated with refractory material which is oxidation resistant and providing further protection against oxidation for the underlying metal. Another object of the invention is to make certain articles resistant to dampness and moisture. Another object is to provide superior refractory coatings on articles, which coatings are strongly adherent under diverse conditions of use. Another object is to provide composite articles which are flame resistant and which can be easily manufactured. Another object is to provide erosion resistant articles which are also resistant to oxidation.

Another object of the invention is to provide a coating on steel parts for use in aircraft engines and gas turbines giving them longer life. Another object is to provide a coating for combustion chambers and nozzles of rocket motors and the like to give them longer life. Another object is to provide superior coverings for rockets and aircraft. Another object is to provide a coating of refractory oxide on a metal part without the use of the step of extreme roughening of the surface being coated, by severe sandblasting or gritblasting to obtain maximum adhesion. Another object is to provide a coating on metal articles with improved thermal shock resistance.

Another object of the invention is to provide a superior class of coatings for such materials as plastics, carbon, and graphite. Another object is to provide a refractory coating which is also substantially impermeable for various materials such as those mentioned. Another object of the invention is to provide bearing surfaces resistant to oxidation at high temperatures. Another object is to provide bearings and bearing surfaces that are resistant to corrosive condition. Another object is to provide an adherent construction for multiple and for sand-wich coatings. Another object is to obtain a refractory coating on a metal without deleterious contamination of the original metal surface by particles of the blasting medium. Another object is to substitute a mild surface cleaning operation for a severe blasting operation and yet obtain superior adhesion without metal distortion. Another object is to roughen the surface being coated by employing a sprayed metal coating. Another object is to interpose an oxidation resistant metal layer between a slightly permeable refractory oxide coating and an oxidizable base metal. Another object is to provide a heat resisting oxidation resisting metal layer between a base material and a refractory oxide flame-sprayed coating.

Other objects will be in part obvious or in part pointed out hereinafter.

The accompanying figure is a photomicrograph of a section of a zirconia coating on a nickel coating on a piece of stainless steel constituting an article according to the invention.

For the formation of articles according to the invention, I procure a metal spray gun of the general type

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disclosed in patent to Erika Morf No. 1,100,602 of June 16, 1914. Such metal spray guns are now well known and are readily available on the market so need not further be described. The metal in the form of a rod or wire is fused, atomized and sprayed by the gun and projected onto a surface in the form of discrete molten particles which freeze in situ.

I further provide another spray gun, or under certain circumstances it might be the same one. This may have the characteristics disclosed in my Letters Patent No. 2,707,691 of May 3, 1955. This gun is for the purpose of coating material with refractory oxide. In general a spray gun which is suitable for fusing, atomizing and spraying refractory oxide can be used for fusing, atomizing and spraying metals so long as the metal is provided in the form of wire or a rod of the proper diameter for the gun and if the gun has adjustments for the feed and the flame so as to adapt it to the spraying of metal. In other cases, I may use a gun for flame spraying the refractory oxide that is adapted for the spraying of the oxide constituent in powder form. Also, a powder gun may be used for the metal spraying.

I provide an article or some material which is to be coated in accordance with the invention. This may be any of those materials indicated in the objects in solid phase provided it is reasonably rigid. This I call the base member. In many embodiments of the invention the base member is a piece of metal, either a shaped metal part or a piece of sheet metal. In other embodiments the base member may be a plastic, meaning a piece of organic material which can be molded, extruded or otherwise shaped, such as a piece of phenol-formaldehyde resin, of methacrylate polymer, of styrene or many other organic materials used in the plastic industry. Hard rubber can be used for the base member and also rigid articles made of rubber substitute material such as butadiene styrene, butadiene acrylic nitrile and chlorinated butadiene.

Example I

I caused six pieces of stainless steel to be coated to produce articles according to my invention. The stainless steel was No. 321 stainless steel (American Iron and Steel Institute). The pieces of stainless steel were squares two inches by two inches and a sixteenth of an inch thick. These constituted the base members. They were sandblasted with Pangborn G-25 steel grit, at 40 pounds per square inch, until a uniformly roughened surface was obtained. They were then spray coated with nickel using a Metco metallizing gun with one-eighth inch wire feed. Following this, alumina was sprayed on with a gun of the type described in U.S. Patent No. 2,707,691. The air pressure was 80 to 90 pounds per square inch. In each case the flame was produced by burning acetylene with oxygen and in each case a blast of air was used for atomizing and spraying. The control of the valves for feeding gases to the spray guns is well understood by workers in this art and need not be described in detail as is also the control of the feeding mechanism, and furthermore, all of this is described in the aforesaid patents and in the case of metal spraying in many other patents.

The first coating of nickel upon the stainless steel was .004 to .008 inch thick. The superimposed coating of alumina, which is a coating of refractory oxide stable in air at room temperature and has a melting point of over 1000° C., was .010 to .020 inch thick. Actually this alumina coating was substantially completely crystalline and its melting point as determined by recent evaluations is 2015° C. ± 15° C.

The coatings and the bases constituted integral pieces. The adhesive strength between the coating of metal and

the coating of refractory oxide (alumina) was substantially equal to the cohesive strength of the refractory oxide coating. The individual particles of the coating of metal and the individual particles of the coating of refractory oxide were self-bonded together so that each coating constituted a rigid integral structure independently of the base member. The metal coating was made up from discrete molten particles of metal frozen in situ on the base member. The alumina coating was made up from discrete molten particles of oxide frozen in situ on the metal coating. Nickel has a melting point of 1455° C. Both the nickel and the alumina, otherwise known as aluminum oxide, were substantially pure.

The adhesion between base metal and refractory oxide coating may often be mechanical in nature. Under these conditions the degree of surface roughness of the metal becomes an important factor toward good adhesion. Mechanical anchorage is aided by reentrant angles which are often present.

Under some circumstances chemical adhesion may be involved. Observations have been made on a specimen with a nickel coating on stainless steel, which had been given a final coating of zirconia. It was subjected to cyclic heating and cooling and withstood the treatment very well. Dark areas that were thought to be nickel oxide were observed in microscopic examination of a cross section, and this nickel oxide may have contributed to the superior performance by a chemical or physical mechanism.

Example II

I provided more pieces of stainless steel in squares two inches by two inches and a sixteenth of an inch thick as base members. Some of these were coated with alumina in accordance with my patent. Some were coated with zirconia in accordance with my patent. Some were coated with nickel prior to coating with alumina; some were coated with nickel prior to coating with zirconia, some were coated with nickel-chromium alloy prior to coating

with alumina and some were coated with nickel-chromium alloy prior to coating with zirconia. The nickel-chromium alloy that was used was Nichrome, which is 60%–80% nickel, 11%–20% chromium and the balance iron. Just where in this range the particular metal used was, I am not sure; but all through this range the difference would be practically immaterial. I believe the wire used for spraying the metal coating was 60% Ni, 24% Fe and 16% Cr.

Following is a description of the samples, their preparation for coating, the coating operation, the thickness of coatings, and the test procedures.

Specimens.—Forty-two specimens of size 2" x 2" x $\frac{1}{16}$ " were sheared out of a sheet of 321 stainless steel.

Preparation.—All specimens were blasted with Pangborn G-25 steel grit at 40 to 50 pounds per square inch pressure. Grit impinged on the plates at an angle of about 90°. Blasting was just sufficient to produce a uniformly rough surface. This caused specimens to be

slightly convex due to compressive stress having developed in the surface because of the impact.

Coating operation.—Coating was carried out with the equipment described in Example I. Specimens were separated into seven lots of six specimens each. Three lots were coated with refractory metal oxide directly on the blasted stainless steel surface. The coating for two of these lots was alumina (thus giving two identical lots, of which one can be considered a "check test") and for the other was zirconia. The fourth and fifth lots were given a metal spray coating of nickel and the sixth and seventh lots were given a metal spray coating of Nichrome. Then the fourth and sixth lots were coated with alumina and the fifth and seventh lots were coated with zirconia.

Coating thickness.—Oxide coatings were

$$0.015'' \pm 0.005''$$

thick. Metal undercoatings were $0.006'' \pm 0.002''$ thick.

Test procedure.—All specimens were then put through a test to determine the relative ability of the coatings to withstand repeated heating and cooling. A laboratory electric furnace was heated to 1040° C. The six test specimens constituting a single lot were placed side by side, coated side down, on a flat piece of stainless steel screen of about $\frac{1}{4}$ " mesh. The screen with the specimens on it was then placed on the bottom of the furnace, and the door was closed.

After 60 seconds the screen with the red hot specimens on it at a temperature of about 1000° C., were withdrawn from the furnace and supported over a vertical stream of fan-driven room temperature air moving at about 300 feet per minute. This brought the samples down to room temperature in three or four minutes. They were then examined to determine the condition of the coating, and the cycle was repeated.

Failure criteria.—Lifting or flaking off of a small amount of coating constituted failure. The number of cycles to failure was recorded.

The results were as given in the following table.

TABLE I
[Results of cyclic heating and cooling test]

Lot No.	Coating	Max. Cycles	Min. Cycles	Avg. Cycles	Type Failure
1-----	Alumina-----	6	1	3½	Coating lifted from base metal at corners.
2-----	Alumina, Check Test-----	4	1	2	Same as above.
3-----	Zirconia-----	11	6	7½	Edges failed by slight lifting of coating.
4-----	Nickel under Alumina-----	10	3	8	Alumina lifted slightly from nickel undercoat at edges.
5-----	Nickel under Zirconia-----	Over 40	40	40+	Slight crumbling at edges, no lifting.
6-----	Nichrome under Alumina-----	Over 40	9	34+	Edge crumbling with slight lifting of alumina coating from Nichrome undercoat at corners.
7-----	Nichrome under Zirconia-----	Over 40	40	40+	Slight crumbling at some edges.

Another method of formation of the metal undercoating for articles according to the invention utilizes electric arc sputtering of the coating metal onto the base surface, in this case a metal or other conductor of electricity. The coating metal, in the form of a rod, functions as one electrode; the base material as the other electrode. A generator, such as is used in arc welding, is used to develop sufficient voltage difference between the base material and the coating metal rod so that an arc is drawn between the two when they are brought sufficiently close together. The arc causes bits of the rod to be melted and fused onto the base material. The rod of metal to be used as coating is held in a pistol type holder on which is mounted an air vibrator that causes the holder and rod to vibrate, depositing the metal coating in discrete molten particles which freeze in situ. Then the oxide coating is applied over the metal coating as previously described. Coating with metal by sputtering is known per se and has been described in patents and technical literature.

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The specimen for the photomicrograph was one of those made as hereinbefore described, with zirconia over nickel over stainless steel. It was one from lot No. 5 of Table I. After the cyclic heating and cooling test, the specimen was sectioned with a diamond cut-off wheel. It was then mounted in Bakelite resin (phenol-formaldehyde) and polished by metallurgical techniques using fine diamond powder.

The light solid area 1 is the stainless steel which is rough due to the blasting operation. The light laminated area 2 is the nickel coating. Dark areas 3 in the light laminated area 2 are believed to be nickel oxide formed by oxidation of the nickel. The medium dark laminated area 4 is the zirconia coating. The medium dark laminated-like structure of the zirconia coating formed with a gun by the impact and bonding of the heat-plasticized particles of the refractory oxide as they deform on hitting the solid surface and bond together, is thought to give the refractory oxide coating a looseness of structure that improves its adherence characteristics to the metal. However, other types of bonding may occur with other types of refractory oxide coatings such as for example, those that are softer, and the nature of adherence is not limited to the above described mechanism. The dark practically black area 5 thereabove is the mounting resin. At least some of the dark irregular areas in the zirconia coating are believed to be artifacts consisting of areas where surface particles were torn out in the polishing operation that was used to prepare the surface for microscopic examination.

The photomicrograph was taken with a magnification of 100 and the scale marking $\frac{1}{4000}$ " thereon is plainly visible. The length of the line below the fraction represents .005 inch. Pores 6 can readily be seen in the area 4 of zirconia. The interlocking of the coatings and the base is readily apparent in this photomicrograph indicating great strength of the coatings.

Example III

Four stainless steel plates, supplied for test panels by a leading manufacturer of jet engines, were of size about 6" x 2½" x ¼". These were sandblasted with G25 crushed steel shot and then spray-metallized with 5 mils of nickel followed by refractory oxide spray coating of 25 mils thickness by the process of U.S. Patent No. 2,707,691. Two plates were thus coated with alumina and two with stabilized zirconia.

The heating tests were made under simulated engine conditions in the laboratory of the jet engine manufacturer, who reported that the steel plates, metallized with nickel, and flame sprayed with alumina and zirconia coatings, showed marked improvement over all panels previously submitted. The manufacturer also reported that the alumina panel became loose near the ends, but failure was in the coating itself, and not at the interface. The manufacturer further reported that the zirconia was even more adherent; showing no indications of failure in one test, and only slight shear failure (within the coating) near one corner of the panel in another test.

Various examples of my invention have been produced experimentally which will have many practical uses in accordance with the objects and otherwise. Among them are the following:

1. Alumina over copper on steel;
2. Alumina over iron on steel, especially stainless steel;
3. Alumina over molybdenum on steel;
4. Alumina over cobalt on steel;
5. Alumina over Monel metal on steel;
6. Alumina over Inconel on steel.

In place of the alumina in all of the above examples could be substituted zircon, zirconia, chromium oxide, wollastonite, spinel, etc.

Example IV

Other test samples which have been made experimentally include employment of a base member of cemented

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tungsten carbide in one case, and stainless steel in another. A rough layer of nickel was deposited employing the sputtering process previously described. After this, an alumina coating 10 to 20 mils thick was sprayed over the nickel. There was excellent adhesion of the alumina coating. Other metals and other hard carbides such as are used for tool bits, etc., could have been used. However, for the sputtering process, the base member must be electrically conducting.

Example V

Multiple coatings can be achieved. For example, black iron specimens were used in one series of experiments. These were roughened by grit-blasting at 40 pounds per square inch pressure and a nickel coating about 6 mils thick was sprayed on using ⅛" nickel wire. Then a moderately soft alumina coating was sprayed on using a powder gun (Model A gun of Wall Colmonoy Co., Detroit) with alpha alumina monohydrate as feed. Following this, a coating of hard alumina was sprayed on using a ⅛" alumina rod as described in Example I. The article produced had a hard alumina erosion resistant surface coating with a laminar microstructure which adapted it to withstand heating and cooling stresses without flaking off, with yieldable alumina back-up layer and nickel anchoring layer. Other oxides and combinations of oxides, as well as other metals, can be used.

Other combinations of multiple coatings and sandwich coatings may be employed depending on the use conditions. For example, the metal undercoating may be sprayed on with a metallizing gun and then heated on the surface to consolidate the particles and render the coating less permeable or substantially impermeable. Torchng the surface is one way to accomplish this. If the heated surface is not sufficiently rough to serve as good anchorage for the refractory oxide layer, then it may be roughened by sandblasting or by other methods such as further metal spray deposition.

Example VI

Two specimens of black iron were grit blasted at 40 pounds per square inch with steel grit to produce a roughened surface and then spray-coated using the Model A Wall Colmonoy gun feeding tin powder in one case and lead powder in another. They were then spray-coated with alumina by the process of Example I. A good adherent hard alumina coating was obtained in both cases. Care had to be taken to avoid deleterious melting of the tin undercoating, during the alumina spraying, due to its low melting point of 232° C.

The gun of my patent, under suitable conditions, is capable of spraying refractory oxides over stable metals including alloys with melting points at least as high as 200° C. However, refractory metals with melting points over 1000° C. are particularly adapted for high-temperature refractory purpose use in conjunction with refractory oxides of melting points over 1000° C. Oxides and metals that are refractory to chemical and erosive action can be useful for many purposes at less elevated temperature, and even at room temperature or below.

A preferred metal undercoating is in the range of 4 to 15 mils thickness, but thinner and thicker coatings such as from 1 to 30 mils or more are usable. Likewise, a usually preferred refractory oxide coating thickness is in the range from 5 to 50 mils but thinner and thicker coatings such as from 2 to 200 mils or more may be desirable depending on the use conditions. There is nothing critical about any of these ranges and the most practical thicknesses are chosen as the best commercial balance in each case between cost and performance under the use conditions desired, such as degree of heat insulation, corrosion resistance, heat-shock resistance, erosion resistance, etc., of the product.

Rough surfaces on the metal coating are desirable as a foundation for the refractory oxide coating. Reentrant angles in the surface hills and valleys are help-

ful. The exact desired degree of roughness will depend on several factors such as the use conditions, the configuration of surfaces coated, the sandblasting medium and blasting techniques if blasting is employed, the metallizing techniques if metallizing is employed as the roughening agent to help to anchor the final refractory oxide coating in place. Cost and use requirements have to be balanced commercially. Concave surface configuration, especially inside cylindrical surfaces, heated in use on the inside such as combustion chambers and rocket nozzles are particularly satisfactory for giving good results. Sandblasting with hard tough inorganic abrasives such as fused alumina or silicon carbide avoid surface embedding with particles of metal grit that may occur when crushed steel shot is used.

Likewise the surface of the base member should be sufficiently clean and rough to give good adhesion with the metal undercoating. My work, however, indicates in general that the surface roughness condition of the base member is of less importance as an anchoring factor for the metal coating, than is the surface condition of the metal coating as an anchoring factor for the refractory oxide coating. Thus a lightly cleaned base member surface is often adequate for anchoring the metal undercoat to the base member, and the rough surface obtained directly on the metal layer coating such

All of the metals which are stable in air and have a melting point of over 200° C. can be used but for many practicable applications of the invention an oxidation resistant metal will be preferred. For the purposes of this invention an oxidation resistant metal is defined as a metal or an alloy which is at least as resistant to oxidation as is stainless steel at a temperature of 1000° C. in air. For purposes of this invention the term alloy is intended to include commercially pure metals as well as higher alloyed compositions.

The elementary metals which as such are oxidation resistant and which are components of many oxidation resistant alloys are, excluding such noble metals as platinum and gold which are most oxidation resistant but too expensive for most applications, nickel, cobalt and chromium.

In general, heat resistant oxidation resistant alloys can be classified into groups based respectively on nickel, cobalt and iron, in which the classification name corresponds to the ingredient that is present in higher numerical amount than any other ingredient of the alloy. Although chromium is an important ingredient in many of these alloys, chromium base alloys are not usually important today.

Typical heat resistant alloys are given in the following table.

TABLE II
[Typical heat resisting alloys]

	Percentage by Weight								
	Ni	Cr	Co	Fe	W	Mo	Ti	Al	Nb
Nickel Base:									
Nichrome I.....	65	11		24					
Nichrome V.....	80	20							
Inconel.....	80	15		5					
M-252.....	55	19	10	2		10	2½	1	
Cobalt Base:									
Haynes Stellite No. 31.....	10	25	55	1	8				
Haynes Stellite No. 30.....	17	24	51	1		6			
J-1570.....	28	20	38	2	7		4		
S-816.....	20	20	43	4	4	4			4
Iron Base:									
Timken 16-25-6.....	25	16		51		6			
18-8 Stainless steel.....	8	18		74					
Kanthal A.....		23	2	69				6	
Smith No. 10.....		37½		55				7½	

as with a metallizing gun or with a sputtering process, by depositing heat plasticized metallic particles, is adequate for anchorage to the refractory oxide coating. Under these conditions, one feature of the step of applying a metal layer coating to a base member becomes a process of roughening the surface of application for the refractory oxide coating to promote adhesion to the base member. If the metal undercoating layer is deposited by processes that do not leave it with a sufficiently rough surface, then it can be roughened in other ways such as by sandblasting. Electroplating leaves a smooth surface.

Articles made in accordance with the invention are useful for all of the purposes described in the objects. Combustion chambers of reaction motors and the nozzles of rocket motors are more resistant to flame and will last longer when coated in accordance with this invention using oxidation resistant metal undercoating, than when coated merely in accordance with my prior patent which itself greatly advanced the art. The outstanding improvement in length of life under thermal shock made by the combination coatings of the invention is hard to explain.

Any refractory oxide having a melting point of over 1000° C. and which will form a coating as described can be used in accordance with this invention. A long list of complex oxides which can be used is given at the top of column 5 in my aforesaid Patent No. 2,707,691 and in column 4 of this patent is a general discussion of the oxides mentioning many of the better ones.

The step of coating the base of rigid material with metal involves, in the preferred embodiment of the invention, fusing the metal, atomizing it and projecting it upon the surface of the rigid material. But in another embodiment the particles of molten metal are produced by separating the molten metal to form said particles without the step of atomizing. In another embodiment of the invention the metal coating can be produced by electroplating. Similarly, in the preferred embodiment of the invention, the step of coating the metal coating with refractory metal oxide involves fusing the metal oxide to form molten metal oxide and atomizing it and projecting it upon the surface of the metal coating. In the above, "fusing" is synonymous with melting.

When I say that the melting point of the metal is at least as high as 200° C., I mean that 200° C. is the minimum and this is substantially the same as stating that the melting point of the metal is above 200° C. Similarly the melting point of the oxide must be at least 1000° C. meaning at least as high as 1000° C. and this is substantially the same as stating that the melting point of the metal oxide should be over 1000° C. Especially in the upper range a difference of one degree is of no consequence in temperature limits like these.

The atomizing of the metal in accordance with the preferred embodiment of this invention is achieved by the use of a blast of gas as described in many prior patents, for example the patent to Morf referred to and other patents, such as the patent to Schoop No. 1,128,058 referred to in my prior patent. As described in my prior

patent referred to herein the molten metal oxide is atomized in like manner, namely by the use of a blast of gas. Usually in both cases the blast of gas is largely air but the products of combustion of the gases such as acetylene and oxygen is part of said blast.

It will thus be seen that there has been provided by this invention oxide coated metal coated articles in accordance with which the various objects hereinabove set forth together with many thoroughly practical advantages are successfully achieved. As many possible embodiments may be made of the above invention and as many changes might be made in the embodiments above set forth, it is to be understood that all matter hereinbefore set forth or shown in the photomicrograph is to be interpreted as illustrative and not in a limiting sense.

I claim:

1. As a new article of manufacture, a metal base member having a rigid integral oxidation resistant composite coating integral with said metal base member, said composite coating comprising a first coating bonded to said base member and consisting essentially of particles of a metal being selected from the group consisting of nickel, chromium, cobalt, iron, molybdenum, copper, lead, tin and alloys thereof and being self-bonded throughout the mass of said first coating so that said first coating itself constitutes a rigid integral structure and a second coating bonded to said first coating and consisting essentially of particles of refractory metal oxide material having a melting point over 1000° C. and being self-bonded throughout the mass of said second coating so that said second coating itself constitutes a rigid integral structure, the adhesive strength between the second coating and the first coating being substantially equal to the cohesive strength of said second coating.

2. An article according to claim 1 in which the metal

base member is stainless steel, the first coating is flame sprayed nickel and the second coating is alumina.

3. An article according to claim 1 in which the metal base member is stainless steel, the first coating is flame sprayed nickel and the second coating is zirconia.

4. An article according to claim 1 in which the metal base member is steel, the first coating is copper and the second coating is alumina.

5. An article according to claim 1 in which the metal base member is steel, the first coating is iron and the second coating is alumina.

6. An article according to claim 1 in which the metal base member is steel, the first coating is molybdenum and the second coating is alumina.

7. An article according to claim 1 in which the metal base member is black iron, the first coating is tin and the second coating is alumina.

8. An article according to claim 1 in which the metal base member is black iron, the first coating is lead and the second coating is alumina.

9. An article according to claim 1 in which the metal base member is cemented tungsten carbide, the first coating is nickel and the second coating is alumina.

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