United States Patent

[11] 3,628,994

[72] Inventors **Sol S. Blecherman**
Newington;
Nicholas E. Ulion, Vernon, both of Conn.

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[73] Assignee **United Aircraft Corporation**
East Hartford, Conn.

[54] **METHOD OF COATING A PLURALITY OF SUBSTRATES BY VAPOR DEPOSITION**
4 Claims, 2 Drawing Figs.

[52] **U.S. Cl.** **117/107.1,** 117/106, 117/107
[51] **Int. Cl.** **C23c 13/00,** C23c 11/00
[50] **Field of Search** 117/106, 107, 107.1, 107.2

[56] **References Cited**
UNITED STATES PATENTS

2,351,536	6/1944	Osterberg et al.	117/106 X
2,408,529	10/1946	Osterberg et al.	117/106 X
2,532,971	12/1950	Van Leer et al.	117/106

Primary Examiner—Alfred L. Leavitt
Assistant Examiner—Kenneth P. Glynn
Attorney—James A. Kane

ABSTRACT: In the processes and method for forming protective coatings on metals, particularly the nickel-base and cobalt-base superalloys, by deposition or vacuum, the positioning of a plurality of substrates of complex geometry in the chamber in a predetermined region of vapor isodensity.

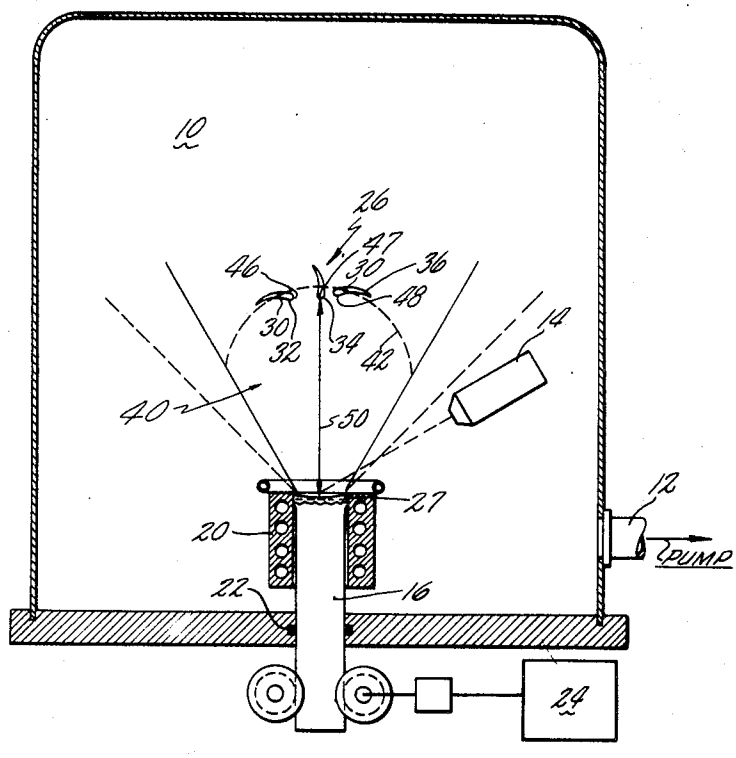


FIG. 1

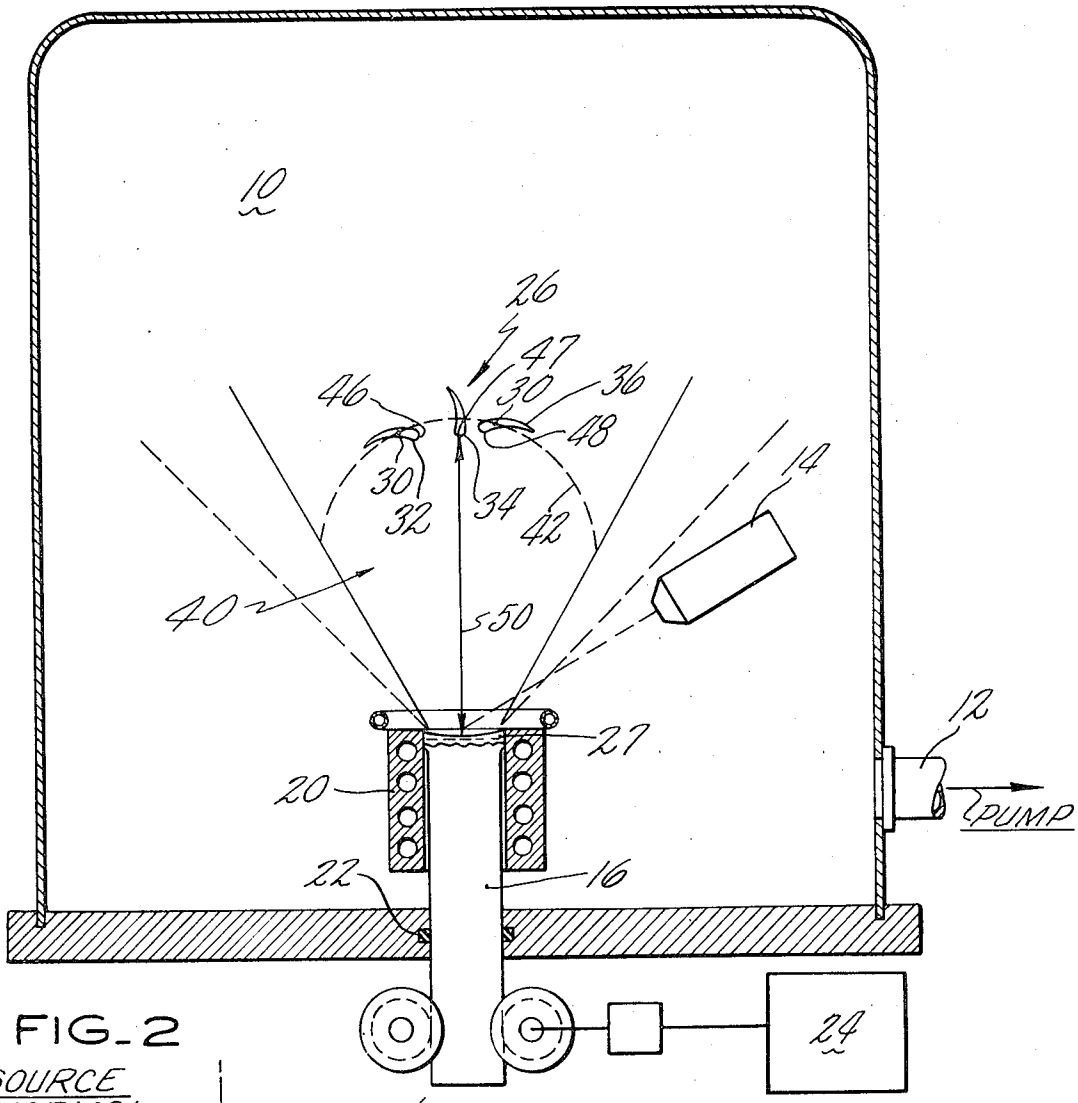
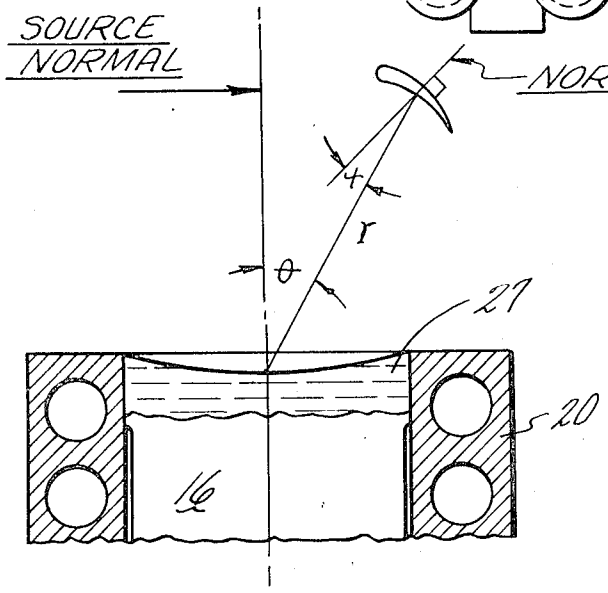


FIG. 2



INVENTORS
SOL S. BLECHERMAN
NICHOLAS E. ULION
BY James A. Kane
AGENT

METHOD OF COATING A PLURALITY OF SUBSTRATES BY VAPOR DEPOSITION

BACKGROUND OF THE INVENTION

The present invention relates to metal-coating processes and more particularly to vacuum deposition of a coating on a substrate.

It is well known that the conventional nickel-base and cobalt-base superalloys do not of themselves exhibit sufficient oxidation-erosion resistance to provide component operating lives of reasonable duration in the dynamic oxidizing environments such as those associated with the operation of gas turbine engines. Accordingly, it has been the usual practice to provide these alloys with a protective coating in such applications.

Many of the more advanced coatings developed for the next generation of jet engines depend in the first instance on the deposition of a high-melting-point coating alloy with a concurrent or subsequent reaction with the substrate to attain the desired end composition, microstructure or adherence. These new alloys generally demand the application of special coating techniques to provide the right species in the right amounts at the surface to be protected.

Several coating compositions of current interest are described in detail in copending applications of the present assignee. Among these compositions is that hereinafter referred to as the FeCrAlY coating at a nominal composition of, by weight, 30 percent chromium, 15 percent aluminum, 0.5 percent yttrium, balance iron, as discussed in the copending application of Frank P. Talboom, Jr., et al. entitled Nickel or Cobalt Base With a Coating Containing Iron Chromium and Aluminum U.S. Ser. No. 731,650, filed May 23, 1968, now U.S. Pat. No. 2,542,530. Another such composition is the CoCrAlY composition at about, by weight, 20 percent chromium, 15 percent aluminum, 0.7 percent yttrium, balance cobalt.

The basic problems associated with the deposition of these coating alloys relates to their high melting points and the difficulty of providing the right amount of all of the alloy species in the coating as applied. Satisfactory results have been attained through the use of vacuum vapor deposition techniques, such as that suggested in the patent to Steigerwald, U.S. Pat. No. 2,746,420. These processes, which have in the past been primarily directed toward the application of relatively low-temperature materials of relatively simple composition, are in the present instance characterized by extreme sensitivity to variations in the process parameters and, accordingly, reproducibility as well as processing expense is a problem.

The vacuum vapor deposition of electron-beam-melted metals has essentially been limited to line-of-sight coating from the source to a rotating or linearly moving substrate. The principal problem encountered when coating a substrate which is rotating is the formation of intergranular precipitates which have the undesirable characteristics of reducing the melting temperature of the coating and additionally reducing the high temperature oxidation resistance characteristics of the coated substrate. One method of removing these intergranular precipitates and hence avoiding the undesirable characteristics which they cause is the method described in the copending application of Richard C. Elam, et al. entitled Method for Coating the Superalloys, U.S. Ser. No. 731,649 filed May 23, 1968, now U.S. Pat. No. 3,528,861, and assigned to the same assignee as the present application.

Heretofore, the description has been limited to the coating of a single rotating substrate. Obviously, when a plurality of substrates, particularly in substrate in the form of a gas-contacting element such as a blade or vane, are to be coated, the problems become intensified. For example, it becomes necessary to apply a uniform coating thickness on all sides of substrates. Another problem encountered with complex shapes or airfoil geometry is that of shielding or shadowing, or more specifically, the shielding of an area of a substrate from the vapor cloud so as to prevent deposition of coating material on the shielded area.

Clearly from the production and cost standpoint, the coating of a plurality of asymmetrical or complex shapes is very desirable. Just as clear is the multitude of problems which have to be surmounted to achieve this desire. The foregoing paragraph is illustrative of some of these problems.

SUMMARY OF THE INVENTION

The present invention relates to a method of applying a coating to an alloy, including the nickel-base and cobalt-base superalloys, wherein the applied coating improves the temperature and corrosion-resistant properties of the substrate. More specifically, it relates to a method wherein a coating is applied to a plurality of substrates that are nonsymmetrical about their longitudinal axis such as turbine blades or vanes, the coating being applied thereto uniformly over all areas and surfaces of each of the substrates.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a schematic cross-sectional illustration of a vacuum vapor deposition apparatus adapted to utilize the process of the present invention.

FIG. **2** is a schematic presentation describing the terms of the formula contained herein.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In the most preferred embodiment illustrated herein a vacuum chamber **10** is shown. Vacuum chamber **10** includes an outlet or evacuation port **12**, port **12** being connected to a conventional-type vacuum pump. The vacuum pump, not shown herein, is of a type suitable for providing the rapid and continuous evacuation of chamber **10**.

Positioned within chamber **10** is an electron beam gun **14**, the purpose of which is to vaporize an ingot of source material **16** and hence establish a vapor cloud of coating material. A conventional magnetic deflection pole piece, not shown, is positioned adjacent electron beam gun **14** to suitably direct the electron beam generated thereby. Ingot **16** is movable within chamber **10** and is slidably received at its upper end by an annular water-cooled crucible **20**. In operation, ingot **16** is normally continuously fed upward into crucible **20** through a heat-resistant vacuum seal **22** in chamber **10**. The rate of the upward feed of ingot **16** is a controlled rate, this control being provided by drive means **24**. In this way, a constant pool height **27** at the top of ingot **16** is maintained and therefore the electron beam will impinge only on the desired pool area. Since the coating process is fundamentally line-of-sight, substrates **26** are usually mounted to affect rotation about their individual axes **30** typically utilizing a pass through (not shown) vacuum chamber **10** to an external drive system. Substrates **26** are herein illustrated as gas-containing elements such as turbine blades or vanes that are nonsymmetrical about their longitudinal axis **32**, **34** and **36**.

According to the method of the present invention, it has been determined that for minimum nonuniformity of coating between the individual gas-contacting elements, or more specifically a substantially constant deposition of coating material on each of the gas-contacting elements, it is necessary that substrates **26** by positioned or mounted within a predetermined region of vapor isodensity **40**, this region of vapor isodensity **40** is a preferred region to permit the depositing of a uniform coating on each of the substrates **26**. This vapor isodensity region **40** includes a curved portion **42** and it is along this curve that the substrates to be coated are positioned. To determine or define this curved portion of constant vapor concentration along which the substrates are positioned, the following formula is to be utilized:

$$H=\frac{\alpha}{\pi\rho}\frac{\cos\theta\cos\psi}{r^2}$$

where:

H= coating rate, cm./min.

α = rate of evaporation gms./min.
ρ = density
ψ = angle between substrate normal and direction cosine
θ = angle between direction of coating and normal to the source
r = distance from pool

For a more complete understanding of these terms, reference is hereby made to FIG. **2** wherein a single rotating substrate is shown positioned above a source of coating material.

The necessity of positioning or distributing substrates **26** along curve **42** arises principally from the fact that the several substrates were at a constant height above the pool, the substrate closet to a vertical axis **50** passing through the center of the molten pool would receive a greater amount of coating material than a substrate positioned on either side of vertical axis **50**. This should be clear from the fact that the coating rate H is at its maximum when θ and ρ=0. By positioning the substrates along curved portion **42**, which defined by the foregoing formula is essentially a cosine curve, a uniform thickness is deposited on each substrate.

Not only is it necessary to be able to deposit a uniform thickness on the substrates but it is also necessary to deposit a controlled depth of coating thickness on various areas of the substrate and additionally to avoid any localized shadowed regions on the substrates. It is to be noted that uniform thickness as used in the context herein refers to surface or area covering. To achieve the desired objective of depositing a controlled depth of thickness on a particular area of each substrate, e.g., the leading edge **46**, **47** and **48** of gas-contacting elements **32**, **34**, and **36** respectively, it is necessary to position the substrates on either side of vertical plane **50** so that the substrates on either side thereof exhibit mirror image symmetry. In the embodiment herein illustrated three gas-contacting elements are shown. Gas-contacting element **34** is positioned or located substantially on vertical plane **50** while gas-containing elements **32** and **36** are located on either side thereof and gas-contacting element **32** is positioned on curve **42** so as to exhibit a mirror image of gas-contacting element **36**. Additionally, in this embodiment wherein three gas-contacting elements are be coated, it is necessary that the gas-contacting elements be arranged and oriented so that the leading edge of gas-contacting elements on either side of vertical plane **50** is nearest plane **50** when the concave side of the gas-contacting element airfoil faces the pool or source.

As hereinbefore described, each of the gas-contacting elements rotates about its own axis, herein illustrated by reference character **30**. It is pointed out that each substrate **26** is located on the vapor isodensity region and positioned on the curve **42** so that the axis of rotation of each substrate substantially coincides with this predetermined curve. More specifically, the distance and angle of incidence with which the vapor impinges on the substrate is constantly changing as the substrate rotates about its axis, this latter axis being the rotational axis and being at a fixed distance and angle from the source. This fixed distance and angle is defined by the predetermined curve **42** since the axis of rotation of each substrate is positioned along this curve.

The present invention is equally usable where substrates of different complex shapes are to be coated. However, in order to avoid a problem of shadowing or shielding of areas on the substrates, it may be necessary to adjust the center of rotation of the substrates relative to one another and the source. In other words, it may be necessary to displace the axis of rotation of one substrate from the centerline or vertical plane **50** of the pool.

A number of tests were conducted with various coating materials and various substrate alloys. In one series of tests, helium gas was introduced at a line pressure of 17 p.s.i.a. through a 4-inch diameter ring manifold oriented concentric with the 2-inch diameter molten pool. Fifty-three evenly spaced, 0.036-inch diameter holes through the wall of a stainless steel tube, oriented at an angle of approximately 45° with respect to the vertical, were provided for orificing purposes.

When weight measurements were made, corresponding specimens coated with and without the admission of inert gas, increases were noted in those tests utilizing inert gas admission. Additionally, spurious deposits of the coating material in low angle locations on the vacuum were significantly reduced.

The results of a number of these tests are summarized in the following table:

TABLE I.—HELIUM COLLIMATION OF CoCrAlY VAPOR CLOUD

	Specimen	Distance above pool (inches)	Angle of incidence with respect to pool horizontal, degrees	Article weight gain (grams)	Coating time (min.)	Chamber pressure (torr)
Test 1	1	9½	70	15.9	30	3.5×10^{-5}
	2	10½	90	18.8		
	3	9½	70	17.1		
Total				51.8		
Test 2	1	9½	70	18.4	30	2.5×10^{-4} He
	2	10½	90	19.6		
	3	9½	70	19.6		
Total				57.6		
Test 3	1	9½	70	17.9	28.2	4.8×10^{-4} He
	2	10½	90	20.3		
	3	9½	70	19.2		
Total				57.4		
Test 4	(1)	9½	52	0.80	28.2	3.6×10^{-5}
	1	9½	70	16.4		
	2	10½	90	17.6		
	3	9½	70	16.3		
Total				49.3		
	(1)	9½	52	1.45		

[1] Vertical ref.

Referring to table I, it will be noted that tests 2 and 3 were run utilizing the inert gas cascading and thus should be compared with tests 1 and 4 which were run without vapor cloud collimation. In each case, three specimens were coated simultaneously, specimen 2 being the specimen located directly over the centerline of the 2-inch diameter pool, specimens 1 and 3 being located closer to the pool but offset from the vertical centerline thereof at an angle of 70° with respect to the pool horizontal.

A vertical reference specimen located at an angle of 52°

with respect to the pool horizontal was utilized to ascertain the effect of vapor cloud collimation on the incidence of low angle deposition.

The typical coating procedure has utilized a power setting of the electron beam gun at 21 kilowatts for the CoCrAlY material and at 15.5 kilowatts for the FeCrAlY coating. Control of coating thickness to ±0.0005 inch at a designed thickness of 0.005 inch has been consistently achieved.

We claim:

1. The method of depositing a coating by vacuum deposition on any one of or all sides of a plurality of substrates nonsymmetrical about their longitudinal axis comprising:

positioning each of the substrates within a vacuum chamber with their longitudinal axis substantially parallel;

establishing a moving cloud of coating material vapor between the source of coating and the substrate to be coated;

positioning each of the substrates in a predetermined region of vapor isodensity, the region including at least a curved portion defining a zone of constant vapor concentration,

placing the substrates such that the substrates on either side of a vertical axis passing through the source exhibit mirror image symmetry;

rotating all of the substrates about their longitudinal axis, each axis being a fixed distance and angle from the source; and

depositing during said rotation a coating of said material on all sides of the substrates.

2. The method according to claim 1 including:

spacing the axis of the several substrates apart along the vapor isodensity curve to prevent shielding of any area of a substrate from the source.

3. The method of depositing a coating by vacuum deposition from a source of the coating on all sides of a plurality of substrates nonsymmetrical about their longitudinal axis comprising:

positioning each of the substrates within a vacuum chamber with their longitudinal axis parallel;

establishing a moving cloud of coating material vapor within the chamber between the source of the coating and the substrate to be coated;

positioning each of the substrates in a predetermined region of vapor isodensity, with opposed substrates on opposite sides of a vertical axis through the cloud having a mirror image symmetry and uniformly spaced from the vertical axis, the region including at least a curved portion defining a zone of constant vapor concentration,

the region of isodensity being defined and determined in accordance with a formula as follows:

$$H=\frac{\alpha}{\pi\rho}\,\frac{\cos\theta\cos\psi}{r^2}$$

where

H = coating rate cm./min.

α = rate of evaporation g./min.

ρ = density

ψ = angle between substrate normal and direction cosine

θ = angle between direction of coating and normal to source

r = distance from, and

rotating each of the substrates about its longitudinal axis, each axis being a fixed distance and angle from the source thereby

continuously varying the distance and angle of incidence of all the substrate surfaces relative to the source of the coating material.

4. The method according to claim 3 including:

spacing the axis of the several substrates apart along the vapor isodensity curve to prevent shielding of any area of a substrate from the source.

* * * * *

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,628,994 Dated December 21, 1971

Inventor(s) Sol S. Blecherman and Nicholas E. Ulion

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Claim 3, Column 6, line 25 - after "distance from" insert

--pool--

Signed and sealed this 6th day of June 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents