N78-12080
NASA TM-75181

NASA TECHNICAL MEMORANDUM

# HIGH TEMPERATURE CORROSION-RESISTANT PROTECTIVE COATINGS IN STATIONARY GAS TURBINES

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Translation of "Hochtemperatur-Korrosionsschutzschichten in stationären Gasturbinen," in Verbundwerkstoffe; Tagung, 3rd (Composites, Meeting, 3rd), Kontanz, West Germany March 28-29, 1974, Technical Reports. Oberursel, West Germany, Deutsche Gesellschaft für Metallkunde, 1974, pp. 168-195 and 197.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON D.C., 20546 NOVEMBER 1977

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## 1. Introduction /168

The working life of the blades in stationary gas tubines in most cases is not limited by mechanical stress but by high temperature corrosion attack. This gave rise to the development of measures to protect against corrosion, among which the use of protective layers is commonly included today. Along with this efforts have been made in the area of alloy technology to develop likewise corrosion and creep resistant materials. In accordance with data on the connection between high temperature corrosion resistance and chrome content, the chrome content in nickel-based alloys has again been raised in some cases to over 20%. The effects of these measures on corrosion resistance are described, for example, in [1] and [2]. References [3] and [4] discuss the properties of the alloys.

Economic considerations most recently have allowed the gas input temperatures to rise sharply. By means of internal cooling of the blades the temperature of the material is kept so low that the creep properties of currently available materials result in satisfactory operational life spans for the blades. However the remaining temperature increases and the locally small exterior /169 wall thicknesses of the blades due to the interior cooling present increased demands on corrosion resistance. Thus the use of protective coatings has once again taken on importance in spite of more corrosion-resistant alloys.

In the past, several review articles have been published on the question of protective coatings in gas turbines [5-10]. Only

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* Numbers in the margin indicate pagination in the foreign text.

[5] and [8] though are primarily concerned with protective coatings for use in stationary gas turbines.

## 2. Selection Criteria

The selection of a protective coating must orient itself in two directions:

1. towards the specific use along with the mechanical, thermal and chemical stresses resulting from the conditions of operation and the required life span;
2. towards the basic material with which the protective coating, under the conditions determined by the use of the turbine, forms a combined system via chemical and physical interactions.

Fig. 1 shows this mutual relationship in the form of a triangle suggested by Grisaffe [12].

The most important property of a coating is of course its resistance to corrosion. This results from its specific ability to form adherent corrosion-resistant oxide coatings due to its chemical composition and, if need by, its morphological structure. Furthermore, it depends on the chemical, thermal and also mechanical stresses determined by the use of the gas turbine.

The chemical stresses which cause corrosion result from the type and amount of condensable components contained in the flue gas. These form in the turbine out of impurities in the fuel (primarily sulfur compounds, but also alkali chloride and vanadium) and in the combustion air. /170

The thermal stresses can affect the life of a coating to a large degree. Gas and metal temperatures determine the nature and rate of the corrosion reactions. This brings up special factors in connection with cooled blades. In unfavorable cases the corrosion temperatures produced by the corrosion rates can be up to 50°C above the measured material temperatures [11].

Temperature cycles can cause the protective oxide films to peel off. The constant new formation of chips in the coating causes an earlier loss of protective elements, which then ultimately leads to the formation of less protective oxide and faster decomposition of the surface. This mechanism, for example, is regarded as the reason for the premature failure of aluminide coatings at temperatures above about 950°C [7,9].

In the future special attention must also be paid to mechanical stress with respect to corrosion processes. Thus, for example, under the effect the creep rupture strength of superalloys can drop at an increasing rate with respect to the reduction in cross-section due to corrosion [12]. The causes for this are still not clear. Processes comparable to stress corrosion cracking are perhaps possible. It is largely unknown what effect coatings exert under these stresses.

An essential factor in the selection of a coating is the projected life span of the component. The design life span of /171
stationary turbine blades currently ranges from 50,000 to 100,000 hours. Inspection are made at intervals of about 8,000 to 20,000 hours. The object, of course, is to select a material or a combination of materials which outlasts not only the time between inspection checks without breaking due to corrosion, but also reaches the achievable life span measured in terms of strength.

In comparison with this the requirements in a airplane gas turbine are indeed considerably higher for reasons of safety, however the overhaul intervals are considerably shorter, and accordingly also the expected lifespan of the blades measured in terms of their strength. The protective coating can even be used up earlier. It is even quite common for blades subject to operational stress to be recoated.

Just the consideration of required lifespans makes it clear that even in the case of purely oxidative high-temperature stress a mere aluminide coating in a stationary gas turbine has hardly any chance, since its life span is limited to about 5,000 hours. [13].

## 3. Production Methods

Table 1 shows typical coating methods along with their most important variables and the commonly deposited elements or applied coatings. Combinations are also often used. The most important methods used todya are also shown schematically in Fig. 2 [9].

The current literature distinguished between diffusion coatings and deposit coatings. One speaks of diffusion coatings when layer-forming elements are diffused into the surface. The transition between the diffusion layer and the base material is for the most part continuous. The concentration of the alloy elements added by /172 diffusion decreases towards the interior. As a rule, changes in the dimensions of the component are small in conjunction with diffusion coatings.

In contrast to this, deposit coatings are always linked with a change in the size of the component. In most cases the composition changes sharply at the interface between the layer and base material. Diffusion zones are limited to small areas. However, while the chemical composition of diffusion coatings and thus also the protective effect against corrosion of these layers strongly depends on the composition of the base material, the composition of deposit coatings can be kept largely independent of the base material.

Today the most commonly used method for coating gas turbine blades is the pack-cementation or packing method (Fig. 2a), which is a type of chemical gas phase deposit which produces typical diffusion layers. The parts are packed in a powder mixture of the layer-forming elements, a small amount of activator (as a rule a halogen salt, e.g. $NH_4Cl$) and an inert filler (in most cases $Al_2O_3$ which is supposed to prevent the powder mixture from fusing. The process takes place isothermically at temperatures between 800°C and 1,100°C. The necessary temperature depends on the coat to be deposited. The reaction time lasts several hours. The process can be carried out in an atmosphere of hydrogen or also in an inert atmosphere.

With this process coatings of aluminide, chrome and silicide can be produced [6,7,8]. Even several elements can be inserted simultaneously or in series into the surface of the material. Thus, for example, in the case of chromaluminization both aluminum and chrome can be deposited [14]. In the tancralization process the following elements are added sequentially: first tantalum and then chrome and aluminum simultaneously [14,15]. In this /173 last process the tantalum layer assumes the role of a diffusion barrier for the aluminum. In another process a layer of platinum is first deposited as a fusion barrier prior to the diffusion of aluminum or chrome [6,15]. In the case of an aluminide layer, however, the platinum does not lie as a barrier between the base material and the layer, but is inserted into the aluminide which can result in an increase in platinum content in the surface by up to 30%. In the case of the chrome layer, as will be shown below, the platinum is concentrated in a more interior region of the coating and in the surface of the coating.

The pure CVD processes which make direct use of gaseous metal halides or organmetallic compounds have so far not won approval for coating gas turbine blades in comparison with the packing process. They have proved to be successful for producing silicide coatings on nickel- or cobalt-based alloys [16,17,23,24].

As variants of the above packing process we may also mention the vacumn-packing process and the vacumn-drossing-packing process [18]. With the latter the packing mixture is applied to the surface of the material in the form of dross. The uniformity of the dross layer is unimportant for the uniform formation of the coating, since here too the coating is produced by a gas transport mechanism.

Another diffusion process is metallization [19]. The process takes place in an electrolytic bath of melted fluorides at a temperature between 600°C and 1,200°C. The metal to be diffused is connected as an anode and the part to be coated as a cathode. With this process, in addition to the usual elements such as aluminum, chrome or silicium, others such as beryllium, yttrium /174 and boron can also be deposited. Two elements can also be diffused at the same time or one after the other.

Among the processes for producing deposit coatings we must first of all mention physical gas phase deposition -- deposit by evaporation. Fig. 2 shows a schematic diagram of such an evaporation process using an electron beam. The main advantage of this process lies in the possibility of depositing alloys of almost any composition. In connection with the development of advanced protective coating alloys of cobalt, chrome, aluminum and yttrium or iron, chrome, aluminum and yttrium this process has achieved greater technical importance in recent times [20].

Similar possibilities are offered by thermal sprays (flame and plasma sprays), as shown in Fig. 2c. In addition to pure metals such as aluminum and corrosion-resistant alloys of the kind named above, glasses or enamels can also be sprayed onto the surface of the material. As a rule, the sprayed coatings are porous and by means of subsequent heat treatment must be sealed by sintering or melted. This causes a limited amount of diffusion between the layer and the base material in the case of metallic coatings and at the same time produces satisfactory adhesion.

Dross coating processes can produce similar results. The coating material is applied by immersion or spraying in the form of a drossed powder onto the surface of the part to be coated. Also in this process glasses and enamels can be applied in addition to pure metals and alloys. Sealing of the coating and an adherent bond with the base material are achieved in the spray method by a subsequent sintering or melting process. In a /175 few cases, especially with silicide coatings, sintering under isostatic pressure has proved successful. This method has also been used in connection with thermal spraying processes [21].

The isostatic hot-press is also used to apply films of protective coating to component surfaces [22]. This is shown schematically in Fig. 2d.

Less versatile than the methods named so far are the methods of galvonic and currentless deposition from aqueous solutions. The simultaneous deposition of several elements is possible only to a very limited extent. For the most part one is confined to layer-by-layer depostion of elements followed by diffusion treatment. Nevertheless, galvonic chrome coatings in connection with intermediate and deposit coatings of other metals have gained importance because of their excellent resistance to corrosion in stationary gas turbines [5].

## 4. Coating Structure and Corrosion Behavior

Of the various protective layers currently available on the market so far only those based on chrome have won acceptance in stationary gas turbines. Aluminide coatings and even chromaluminide coatings have repeatedly proved to be insufficiently resistant [8].

Fig. 3 shows typical chrome diffusion coatings produced by the packing process on a maleable alloy (left) and a cast alloy (right) on a nickel base. The curves below are concentration curves for the most important elements. One can distinguish between a diffusion zone and a built-up zone above the original surface of the material. The diffusion zone is characterized by a continuous drop in concentration of the base material elements and at the same time a for the most part steady rise in the layer elements, in this case chrome and iron. Moreover, it can be seen /176 that the elements of the base material -- nickel, cobalt and molybdenum -- diffuse outwardly relatively far into the build-up zone. Concentrations of iron in the region of the original surface of the material to be coated are frequently found. Less significant here in the example of Nimonic 105 are the concentrations of chrome on the original surface of the material and the concentrations of nickel, cobalt and molybdenum found at the base of the built-up zone. The reasons for these concentrations are not clearly known.

The important elements in the built-up zone consisting primarily of two phases are chrome and iron. The matrix lower in chrome content can reach chrome contents of about 75% on the surface. Often chrome contents of only around 60% are found with a corresponding increase in iron and nickel concentration. Embedded in the matrix are α-chrome precipitate with 80-86% chrome and about 6-10% iron (the remainder being formed by elements of the base material). Also found embedded in this zone are oxide particles stemming from the embedding powder. Many times finely distributed carbide phases are precipitated in the built-up layer. In the region of the original surface of the material is a zone with $Al_2O_3$ and TiN precipitates which must be regarded as selective oxidation or nitridation products of the γ-phase. Needle-shaped particles of $TiO_2$ have also already been found in this zone [8]. In many cases a very thick border of such precipitates forms which seems to promote the tendency of the built-up layer to peel.

The structure of a chromed coating with a platinum diffusion barrier layer is very similar. Fig. 4 shows such a layer made of Nimonic 80 A. On top of a thin external platinum-enriched zone comes a chrome-rich, two-phase zone with α-chrome precipitates. To this is attached a platinum-enriched zone about 30-50 μm thick. The platinum concentration can be up to 45% next to the /177 overlying two-phase zone [6,15]. In the vicinity of the original surface of the material to be coated precipitates of $Al_2O_3$ and TiN are also found.

In contrast to aluminide coatings, little has yet been reported on the corrosion behavior and corrosion mechanism of chrome protective layers [5,8 and 23]. In the past years in our own laboratories numerous studies have been carried out in this direction. The chmeical and metallurgical processes in the coatings are presently still being studied.

The following pictures may give a brief impression of the behavior of such protective coatings. Fig. 5 shows in succession an unprotected blade made of Fe-Ni-Co-Cr alloy S 590; a blade of the same material protected an electrolytic chrome coating; and a blade made of Nimonic 80 A provided with a Cr-Pt coating. All of the blades came from a field test under difficult corrosion conditions in blast furnace gas. The working time was 5,300 hours at a gas input temperature of 720°C. Apart from a thick deposit in some places the chrome-protected blades showed practically no changes in comparison with heavily corroded and practically deposit-free unprotected blades.

Fig. 6 shows that the Cr-Pt coating is already clearly corroded in various spots. In places on the leading edge it has brokem through to the base material. In other places the external platinum-rich zone is worn off an oxidation has penetrated deeply into the two-phase zone rich in chrome. An interesting fact is that in the region of the leading edge the chrome-rich -phase seems to be becoming corroded, while on the back of the blade away from the leading edge the matrix with a lower chrome content shows more serious corrosion.

Similar observations were also made on other types of chrome coatings. However the fact that even diffucult corrosion /178 conditions, as for example can appear in blast furnace gas turbines, can be well controlled by suitable chrome coatings is shown by Fig. 7. The corrosion and operation conditions correspond more or less to those in Fig. 5. While the unprotected blade had to be exchanged as soon as after 5,200 hours, the blade made of the same base material but provided with an electrolytic chrome coating is still in excellent condition after 26,000 hours.

Fig. 8 shows an example of a silicide coating produced by the packing process. The base material is Nimonic 80 A. Attached to a multiple phase silicide zone is a silicium-rich transition zone, and next to the base material is a diffusion zone with decreasing silicium concentration. Erdös and co-workers [8] found in silicide coatings on Nimonic 105 precipitations of (Co, Ni)Si in a matrix consisting primarily of $Ni_2Si$. A similar structure is shown by silicide coatings produced not by the packing process but by the CVD process [23,24]. On alloy IN 738 the phase MSi, $M_2Si$ and $M_5Si_3$ were certainly found, and, with some reservations, the $Ni_3Si_2$ phase and the Laves phase were found.

Results from corrosion experiments on silicide coatings have also already been reported [8,16,17,23]. The protective oxide film which forms on the surface seems not to be $SiO_2$ but the the nickel olivine $Ni_2SiO_4$. Other oxide phases found were spinel and NiO. Along with binary and ternary silicides the corrosion-stressed coating also contains the G-phase $(Cr,M)_6Ni_{16}Si_7$ and probably also the T-phase $Cr_3Ni_5Si_2$ [8]. On IN 738 chrome oxide was also found under the nickel olivine layer. On the basis of these findings the protective effect is outlined in Table 2.

A field test comparison on Nimonic 80 A in a blast-furnace-gas-fired turbine (material temperature 710°C, running time 30,000 hours) showed better corrosion resistance for the silicide /179 coating than for the diffused chrome coating (Fig. 9) [24]. Excellent resistance against $V_2O_5$ corrosion is reported in [23]. At the same time, however, the brittleness and liability to cracking of these coatings is pointed out. Cracks in the coating can cause the formation of an intermediate aluminum oxide zone on alloys with a high aluminum content [8], thus increasing the risk of peeling for such coatings.

Experiments with high-temperature enamel as a protective coating against corrosion on gas turbine blades were reported for the first time by Möller [5]. In the meantime further data has been collected. Fig. 10 shows a $SiO_2$-rich enamel with $Cr_2O_3$ and $TiO_2$ on Nimonic 80 A in the delivery condition (left) and after 35,000 hours in operation in a blast-furnace-gas turbine. After this period of time the enamel had survived almost completely intact on the entire surface of the blade and had exerted an excellent protective effect. Its behavior was better than that of the diffused chrome coatings tested in the same field experiment. To be sure, other enamels showed considerably poorer behavior. Most of these enamels had peeled off during operation.

The knowledge that aluminide coatings under the effect of corrosive and cyclic thermal stress are consumed more quickly because of constant peeling off of the protective oxide film [7] has resulted in the development of new types of coatings. These are deposit coatings made of alloys of FeCrAl or CoCrAl with small additions of yttrium or rare earths. Fig. 11 shows the typical structure by way of example of a layer consisting of cobalt with 25% chrome, 14% aluminum and 0.2% yttrium. The precipitates involve CoAl. As a result of interdiffusion with the base material a NiAl layer formed in the interface which shows up in the picture as a dark gray border [20].

The small amounts of yttrium (also cerium, Scandium and lanthanum have already been used) improve the adhesion of the protective oxide film and thus prevent it from peeling off during temperature cycling. Fig. 12 shows an example of this with a FeCrAl alloy with scandium and yttrium added [25]. The mechanism which improves adhesion is due to the formation of vacancy complexes or the creation of internal oxide interfaces on which excess vacancies can condense. If these alloy components are lacking then the vacancies condense in the oxide - protective coating interface and there form pores which cause the coating to peel off. This theory of "vacancy sinks" can also be applied to such cases where in place of the above-named elements finely dispersed oxides are inserted such as $ThO_2$, $Y_2O_3$, $CeO_2$ and also $Al_2O_3$. Further information on these processes is collected in [26]. /180

Up to now little information has been available on the corrosion behavior of such coatings in stationary gas turbines. It is to be expected that the improved oxide adhesion will also bring advances in this area. In addition, these adhesion-promoting elements can form very stable sulfoxides which can bind the sulfur which gets into the coating and thus remove it from the corrosion reaction [27].

## 5. Prospects

Modern development in the area of stationary gas turbines is characterized, among other things, by higher material temperatures for the blades and by the use of cooled blades.

The higher temperatures can cause a speeding up of the corrosion processes. Possible changes in the corrosion mechanisms themselves may, on the other hand, have a positive effect [2]. In this connection one must also take into consideration the /181 vapor pressures of the protective oxides which are formed. Thus, for example, it has been found that above about 90°C $Cr_2O_3$ in the form of $CrO_3$ evaporates off in significant quantities and thus sharply restricts the life span of chrome protective layers [28].

The use of interior-cooled blades with cooling holes running a few millimeters beneath the surface presents additional demands on the corrosion resistance of the material used and the resistance and life span of a protective coating. Local penetration through the material of the blade to the bore for the coolant caused by corrosion can result in extensive damage.

Modern research and development in the field of protective layers, just like research and development on alloys, has begun to take these problems into account. On the one hand, thicker deposit coatings are being produced with a composition independent of the base material with which the outward diffusion of the protective elements, e.g. chrome, can be partially controlled by a thin covering layer which is itself not corrosion-resistant [13]. On the other hand, coatings such as of CoCrAlY, or even the platinum-aluminide coating, are to be considered progressive in this respect. Steps which have already been taken indicate that in the future more and more complexly constructed coatings will find use. Along with this the successive application of various coating processes may take on increased importance.

Much of the information treated here is based on earlier studies of my colleagues Möller, Faber, Dr. Felix, Erdös and their co-workers. The microsection surfaces were prepared by Ellner and Eisenbeis. The studies with the microprobe were performed by Dr. Demny. My thanks to all of them here.

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## Table 1 - Coating Processes /185

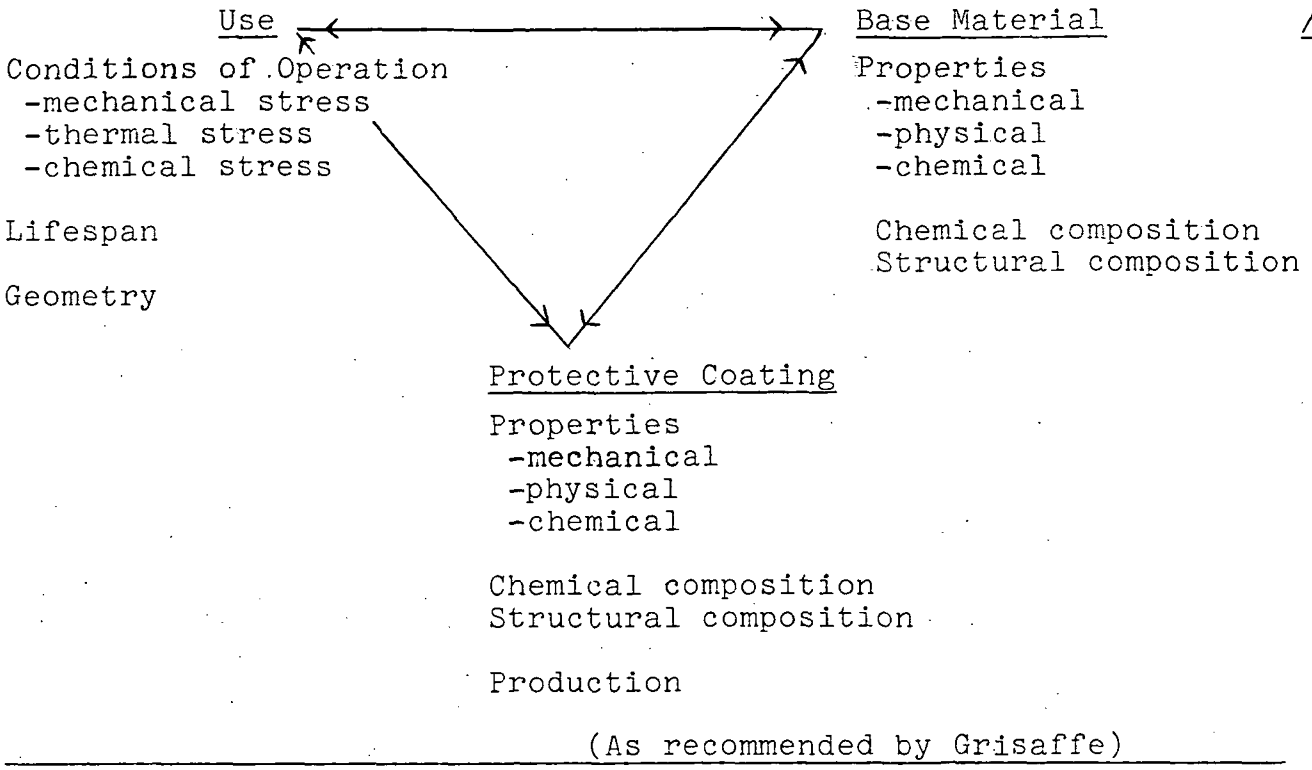
| Process | Variants | Deposited Elements |
|---|---|---|
| Chemical vapor position (CVD) | Pack-cementation | Cr, Al, Si(Fe,Ni) |
| Physical vapor deposition (PVD) | Vacumn evaporation | Cr,Al,Si; complex alloys such as Co-Cr-Al-Y or Fe-Cr-Al-Y |
| Electrolytic and currentless deposition from aqueous solutions or salt melts | Electrolytic chrome plating | Cr |
|  | Metallization | Cr,Al,Si |
| Thermal sprays | Flame sprays<br>Plasma sprays | Complex alloys such as Ni-Cr or Co-Cr-Al-Y and others |
| Hot dip coating |  | Al |
| Electrophoresis |  | Al,Cr,Si |
| Dross coating | Enamelizing | Complex silicate glasses |
| Isostatic hot-press |  | Complex alloy |

## Table 2 - Breakdown of Functional Components in a Silicide Coating on Nickel Alloys

| Structure | Si-content | Ni-content | Cr-content | Most important requirements |
|---|---|---|---|---|
|  | in % by weight |  |  |  |
| Nickel silicate | 10-12 | 40-50 | 0-5 | Corrosion protection |
| Chromium oxide | 0-5 | 10-20 | 30-60 | Diffusion barrier for corrosion products |
| Silicide layer | 25-30 | 40-50 | 4-10 | Corrosion resistance |
| Chromium concentration | 20-25 | 25-40 | 20-40 | Corrosion barrier for Si |
| Base material | 0-2 | 50-65 | 10-20 | Creep rupture strength |

(After Felix and Erdős)

/186



<u>Use</u>

Conditions of Operation
- -mechanical stress
- -thermal stress
- -chemical stress

Lifespan

Geometry

<u>Base Material</u>

Properties
- -mechanical
- -physical
- -chemical

Chemical composition
Structural composition

<u>Protective Coating</u>

Properties
- -mechanical
- -physical
- -chemical

Chemical composition
Structural composition

Production

(As recommended by Grisaffe)

Fig. 1. Selection criteria for base material and protective coating.

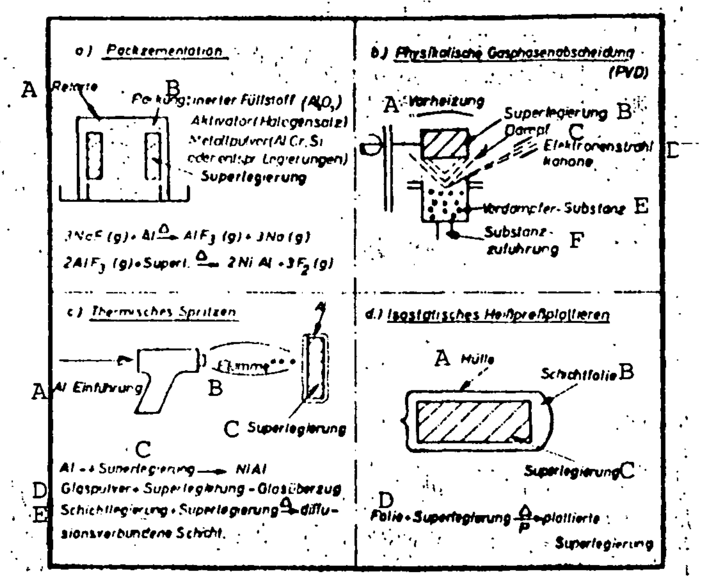


Fig. 2. Different coating processes (after Grisaffe)

Key: a) Pack-cementation

A) Retort
B) Packing: inert filler ($Al_2O_3$)
Activator (halogen salt)
Metallic powder (Al,Cr,Si or corresponding alloys)
Superalloy

b) Physical Vapor Deposition (PVD)

A) Preheating
B) Superalloy
C) Vapor
D) Electron beam gun
E) Evaporated substance
F) Substance input

c) Thermal Spraying

A) Al input
B) Flame
C) Superalloy
D) glass powder plus superalloy plus glass coating
E) Coating alloy plus superalloy plus diffusion-united coating

d) Isostatic Hot-press Plating

A) Sheath
B) Coating film
C) Superalloy
D) Film plus superalloy → plated superalloy

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[Fig. 3-11 not available]

/197

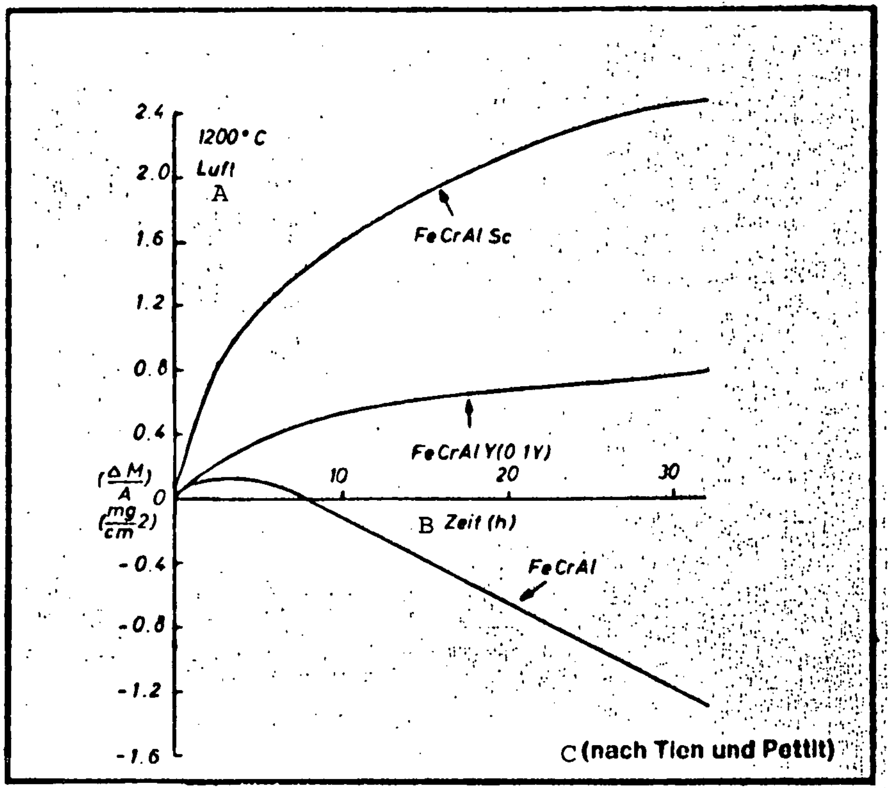

Fig. 12. Effect of Sc and Y on oxide adhesion

Key: A) Air
B) Time in hours
C) (After Tien and Pettit)