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A PROCESS FOR THE PRODUCTION OF A SCALE-PROOF AND CORROSION-RESISTANT COATING ON GRAPHITE AND CARBON BODIES

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Translation of "Verfahren zur Herstellung einer Zunder- und korrosionsfester Abdeckschicht auf Graphit- und Kohlekörpern" German Patent Office (West Germany), Auslegeschrift 1009093. Application date 20 January 1956, 2pp.



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16. Abstract A process is described for the production of a corrosion-resistant coating on graphite and carbon bodies, characterized by the fact that the carbon or graphite body is coated or impregnated with titanium silicide under the addition of a metal-containing wetting agent in a nitrogen-free atmosphere, so that a tight coating layer is formed..			
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A Process for the Production of a Scale-proof and Corrosion-resistant Coating on Graphite and Carbon Bodies

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The invention concerns a process for the production of a scale-proof and corrosion-resistant coating of carbon and graphite bodies, so that these synthetic carbon objects are protected against oxidation, i.e., burning.

Carbon and graphite have become increasingly recognized as fireproof materials, e.g., the cladding material for blast furnaces and other purposes. In respect of temperature, there are practically no limits to their application, only providing that air and oxygen are kept out above 400°C, so that a fire is prevented. The other characteristics are outstanding. Let us mention the good thermal and electrical conduction, the exceptionally low capacity for thermal expansion, and the resulting resistance to temperature change. A stability to chemical influences is specified. Sealed with synthetic resin, graphite is thus employed as a corrosion-resistant material in the chemical industry. But also advantageous is the high sublimation point of the carbon, which lies well above the technologically feasible temperatures.

It is known that graphite bodies can be coated with oxidation-proof and thick layers, so that they are capable of use in air for rather high temperatures. Thus, e.g., ceramic protective coatings of oxides, silicates, and carbides, primarily silicon carbide as well as molybdenum silicide, are deposited either by spraying and subsequent searing or by direct chemical surface reaction. SiC-layers are produced by the effects of Si- or SiO₂- vapor at temperatures of [illegible] to 2000°C. Molybdenum silicide is stirred with synthetic resin to form a paste,

which is applied and converted into a layer containing Mo-Si-C by searing at temperatures of 2000°C. Although the scale-proof properties, especially of the last two combinations, are [missing word], rather narrow bounds are placed on the technical applications of the thus-protected synthetic carbon objects, as any damage to the extremely [thin?] layers, especially any interruption or [illegible], results in the [illegible] of the carbon scale-proof surface film remains as a [illegible] skin.

By searing the molybdenum silicide on the graphite, a ternary compound, Mo-Si-C, is formed, as already mentioned. This is known to be slightly scale-proof. In an identical manner, other [illegible] are formed by reaction with the carbon at the [illegible] site, ternary compounds, or direct [illegible]. Under the effects of higher temperature, these reactions steadily proceed by the increasing diffusion of the carbon. Ultimately, the resistance of the protective layer depends not only on the behavior of the silicide, but also on that of the compounds of the silicocarbide and the carbide. Experimental tests have confirmed that, despite the good wetting capacity of the carbon by the molybdenum or molybdenum-containing compounds, the molybdenum silicide forms no thick and scale-proof protective layer on the carbon bodies, as the ternary compound Mo-Si-C is apparently formed.

By means of the invention, a protective layer is obtained on carbon and graphite bodies, which is superior with respect to scale-resistance and thickness, to the conventional coatings. This is achieved, according to the patent, by the fact that the carbon or graphite body is coated or impregnated with titanium silicide under the addition of a metal-containing wetting agent in a nitrogen-free atmosphere, so that a coating is formed, which is anchored in the pores of the carbon body. If, specifically, titanium disilicide is deposited in the place of the molybdenum silicide, it is established by inflammation testing that this behaves much better and is more scale-proof than the silicide of the other metal. This is attributed to the fact that no titanium silicocarbide is formed, and the titanium carbide is much more scale-proof than the carbide of the other high-melting metals. For an identical

reason, titanium carbide is in fact used, e.g., in cutting knives for high cutting speeds instead of the highly unstable tungsten carbide, and even in metal-ceramic bodies in a mixture with oxides or with binder metals the good temperature stability of the titanium carbide is of special importance.

Under the addition of the wetting agent, the titanium silicide is immediately applied to the graphite at 1600°C and, after a prolonged exposure, penetrates into the pores of the graphite or carbon body, so that the latter becomes gas- and water-tight. By the exceptionally strong adhesion of the titanium silicide to the carbon, it is furthermore possible to apply thicker films, e.g., up to 1 mm in thickness, which form a special protection.

In addition to the use of such a material, based on synthetic carbon, protected by titanium silicide, as a fireproof and chemically-resistant structural material, this can also find applications for combustion chambers, jet engines, and rocket components, on account of its high mechanical strength and abrasion resistance.

Titanium disilicide can be produced in an inert or reducing atmosphere at roughly 1200°C from the stoichiometric mixture of its components; however, care must be taken that the process takes place in an atmosphere with as little as possible nitrogen, otherwise titanium nitride is formed. Titanium silicide in the molten condition cannot, however, be applied to graphite, as it does not wet it. Nevertheless, a wetting can be achieved by introducing small amounts of molybdenum powder or molybdenum silicide powder, as well as other materials such as zirconium, vanadium, or compounds of these.

Thus, the process may be implemented in two ways:

1. by dipping in a melt of titanium disilicide:
2. by application of solid powdered titanium disilicide and the melting down of this nonconsistent coating in protective gas. However both processes must take place in N_2 -free atmospheres.

In the first case, the molded piece of synthetic carbon, e.g., a plate or such article, is placed in the pre-heated condition in a melt of titanium disilicide at 1600°C. Within several minutes, after immediate wetting this penetrates into the surface irregularities and pores, so that after removal of the graphite from the melt it is furnished with a tight, very-adhesive, and well-secured superficial layer. The wetting action is not confined to the surface, as has been mentioned, but rather penetrates to the interior, so that there is an impregnation of the outer layers, as clearly shown by polished sections. In a short time, the titanium silicide penetrates to 1 mm or more. Obviously, such a coating provides a very good protection against scale, especially since the titanium silicide, even in this form, is a very hard and wear resistant compact material, and resists mechanical damage by its sturdy structure; furthermore, this layer of titanium disilicide is entirely free of pores and thus impermeable to gas and liquids.

The second possibility for application of such layers of titanium disilicide can be carried out in the most diverse versions. For example, good success has resulted from the application of a paste, consisting of 80% titanium disilicide, prepared in a familiar manner with an alcoholic solution of silicic acid and ester, or with a water slurry of colloidal alumina. After drying, such layers are again seared by rapid heating in an inert atmosphere at 1500 to 1600°C. Under argon as a protective gas, it is also possible to spray the titanium disilicide on incandescent molded pieces. In one such case, it was found to be advisable to previously incorporate the wetting compounds of molybdenum, vanadium, or other substances in the graphite.

For special applications, the surface layer of titanium disilicide can again be coated with a metal layer. This can be done either by metal spraying or dipping, or even in the galvanic manner, since both the carrier of synthetic carbon and the applied layer of titanium disilicide are good conductors. A silver coating has proved itself to be a specially resistant metal on layers of titanium disilicide, e.g., as a current feed for graphite heating rods in contact with $TiSi_2$, or as a fusing material for the fastening of graphite rotor blades, coated

with titanium disilicide, in the rotor shroud of gas turbines.

Patent Claims:

1. A process for the production of a corrosion-resistant coating on graphite and carbon bodies, characterized by the fact that the carbon or graphite body is coated or impregnated with titanium silicide under the addition of a metal-containing wetting agent in a nitrogen-free atmosphere, so that a tight coating layer is formed, secured in the pores of the carbon body.

2. A process as per claim 1, characterized by the fact that the coating of the carbon body with titanium silicide is carried out in the molten state of titanium silicide, implemented by dipping the carbon body in a melt of titanium disilicide under a protective gas.

3. A process as per claim 1, characterized by the fact that the coating in the form of a paste of powdered titanium silicide with a binder is applied to the synthetic carbon body and, after drying, seared to the carbon body by roasting at a temperature of roughly 1600°C in an inert N₂-free atmosphere.

4. A process as per claim 1, characterized by the fact that the titanium silicide is applied to the heated carbon body and fused, depending on the type of metal spray process.

5. A process as per claims 1 through 4, characterized by the fact that the carbon body, coated with titanium silicide, is coated with an additional metal layer.

6. A process as per claims 1 through 5, characterized by the fact that silver is used for the metal coating on the titanium silicide.

7. A process as per claims 1 through 6, characterized by the fact that the carbon body is impregnated, prior to application of the titanium silicide, with a molybdenum compound, which potentiates the

wetting of the carbon body with titanium silicide.

8. A process as per claims 1 through 6, characterized by the fact that powdered molybdenum or molybdenum silicide in the amount of roughly 5 to 15 Wt.-% is added to the titanium silicide as wetting agent.