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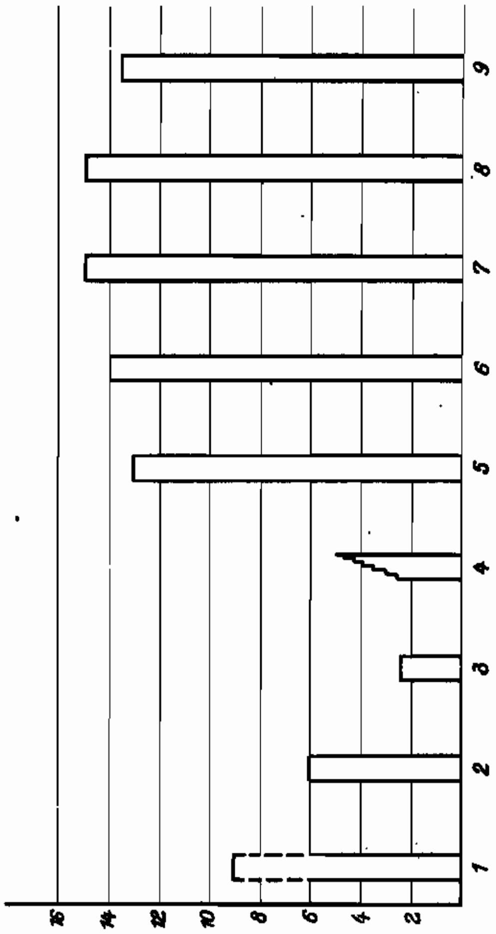

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ALIEN PROPERTY CUSTODIAN

PROCESS FOR THE PRODUCTION OF OXIDIC PROTECTIVE COATINGS ON MAGNESIUM OR MAGNESIUM ALLOYS

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Different processes are already known for the production of oxidic protective layers on magnesium or magnesium alloys by anodic oxidation in alkaline baths.

Strong alkaline baths were first proposed containing about 60 gr. KOH per litre, in which the magnesium object was submitted at room temperature to the action of alternating current, or as anode to the action of continuous current.

Moreover baths are known which, besides similar or smaller amounts of alkalies, contain carbonates, cyanides and if necessary also other additions. In such baths the oxidation of the magnesium must be carried out at temperatures between 20° C and 25° C. Finally baths have also been proposed which require it is true a high temperature of from 80 to 90° C but have a considerably lower alkalinity than the baths before mentioned. According to this proposal the electrolyte must consist of an aqueous solution of phosphates such as sodium phosphate, or acid ammonium phosphate. In this case the pH number must be over 6, and be further raised by addition of ammonia or ammonium salts. Already after an addition of 50 cc. of conc. ammonia, the pH number however, does not increase further and remains constant at about 11. The addition of ammonia or ammonium salts is accompanied by considerable inconveniences by heating the baths to 80–90° C at which temperature ammonia is evolved in large amounts. Thereby a continuous and uneconomic addition of ammonia becomes necessary, and the workmen are very must inconvenienced by the gas evolved. The increase in layer thickness very soon ceases, on account of the rectifier action of ammoniacal phosphate solutions, and also with higher bath voltages there may be maintained only a very small residual current. Moreover salt deposits make the protective layers unusable on long hanging on.

It has now been surprisingly found that considerably better results may be obtained if the oxidation of the magnesium and its alloys is carried out in heated, strongly alkaline baths, of which the pH values lie above 12. The protective layers obtained with large excess of alkali are much harder and denser than those hitherto known. Above all however, in these baths at high temperatures the falling off of the current density occurs considerably more slowly. There thus does not occur so soon an automatically interrupted increase in thickness. The result thereof is that considerably stronger protective layers can be obtained. The new knowledge stands in contrast to the processes for the anodic oxidation of aluminium where strong alkaline baths cannot be used and any temperature increase leads to a diminution in the hardness and an increase in the porosity. According to the invention favourable results are obtained for example in baths heated to 50 to 80° C, and, which, if necessary besides other alkali compounds, contain such an amount of fixed alkali and/or alkaline earth hydroxides that the pH value lies above 12.

The drawing is a graphic illustration of the strength of layer of different protective layers which have been prepared partly according to known or proposed processes (Nos. 1–4) partly according to methods of carrying out the present process (Nos. 5–9).

The thicknesses of layers are given in the perpendicular ordinates in thousandths of a millimetre, whilst the horizontal ordinate shows the successive number of the experiments. With all the experiments shown in the drawing an amount of current of 30 ampere-minutes per sq. dm. of surface was employed. In the following are given the experimental conditions and the results in detail:

Experiment 1.—Electrolyte 50 g. ammonium biphosphate per litre. Ammonia addition up to pH=10.0. Temperature 85° C. Current density falls rapidly to 1 amp/sq. dm. Voltage 10 volts. After 5 minutes almost no layer. After 20 minutes, thickness of layer maximum 0.009 mm. in section. Not uniformly thick colour dark grey. After 30 minutes salt like deposits. Layer peels off.

Experiment 2.—Electrolyte 60 g. KOH per litre. Temperature 20° C. Current density 1 amp/sq. dm. Voltage 5 volts. After 30 minutes, thickness of layer 0.006 mm. Colour medium grey.

Experiment 3.—Electrolyte 50 g. NaOH. 50 g Na_2CO_3, 20 g. KCN 1 gm. Na_2WO_4 per litre. Temperature 22° C. Current density 3 amp/sq. dm. Voltage 16 volts. After 10 minutes 0.0023 mm. layer thickness, after 30 min. 0.0065 mm. layer thickness.

Experiment 4.—Electrolyte 50 g. NaOH. 50 g. $Na_2B_4O_7$. 20 g KCN per litre. Temperature 20° C. Current density 1 amp/sq. dm. voltage 10 volts. After 30 minutes 0.005 mm layer thickness with a strongly pitted surface.

Experiment 5a.—Electrolyte 40 g. NaOH per litre. Temperature 70° C. Current density 1 amp/sq. dm. Voltage 4 volts. After 30 minutes 0.013 to 0.014 mm layer thickness.

Experiment 5b.—Electrolyte 60 g KOH per litre. Temperature 60° C. Current density 2 amps/sq. dm. voltage 4 volts. After 15 minutes 0.012 mm layer thickness.

Experiment 6.—Electrolyte 100 g. NaOH per litre. Temperature 70° C. Current density 1 amp/sq. dm. Voltage 4 volts. After 30 minutes 0.014 mm. layer thickness.

Experiment 7.—Electrolyte 150 g. NaOH per litre. Temperature 70° C. Current density 1 amp/sq. dm. Voltage 3.4 volts. After 30 minutes 0.015 mm. layer thickness.

Experiment 8.—Electrolyte 200 g NaOH per litre. Temperature 70° C. Current density 1 amp/sq. dm. Voltage 3.4 volts. After 30 minutes 0.015 mm. layer thickness.

Experiment 9.—Electrolyte 150 g. NaOH, 75 g. $Na_2B_4O_7$ g. per litre. Temperature 70° C. Current density 1 amp/sq. dm. Voltage 3.7 volts. After 30 minutes 0.0135 mm. layer thickness.

The superiority of the anodic treatment carried out according to the invention shows itself however not only in the layer thickness but also in the nature, namely in the hardness, density adhesiveness and general appearance of the layers.

While the hitherto obtained layers were deposited more or less poor-looking grey or black, the layers deposited according to the invention show bright grey to whitish colours of enamel-like appearance.

In this way is also possible to bring about the subsequent coloration in bright colour tones.

A comparison of experiments **5a** and **5b** shows that equimolar solutions yield about the same results. The increase of the concentration of alkali hydroxide over 50 gm. per litre gives no appreciable increase in the layer thickness but however gives a further brightening of the colour as well as an increase in density and hardness.

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