Producing of ZrO_2 Coating by Method Zol-Gel

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Abstract – Producing of ZrO_2 coating by zol-gel method is presented in the paper. For solutions preparation the salts and ethyl spirit have been used. As a result of their interaction the alkoxy compounds specific for group IV elements of Periodic system are formed. In our case, the alkoxy compounds are produced from zirconium nitrate. The reaction is accelerated by nitric acid addition. The reaction of alcoholyat formation takes place with local heating. The alcoholyat is decomposed in an exsiccator. Flasks with reagents are placed into snow to prevent a reagent splashing. On application of alcoholyat to a copper substrate and its subsequent alcoholyat decomposition the coating is found to exfoliate. Therefore, a nickel sublayer from U_{ots} electrolyte is applied to the copper substrate. Further, a zirconium nanooxide is applied to the nickel sublayer by electrochemical technique to form a composite electrochemical coating. Then zirconium alcoholyat is applied by casting. The alcoholyat is decomposed in an exsiccator. The resulting zirconium alcoholyat coating is subjected to thermocyclic tests. A maximum heating temperature is 980 °C.

The proposed zol-gel coating technique from alcoholyats shows good promise due to its simplicity and relatively low cost.

1. Introduction

The technique of applying coating from coating solutions alcoholyats is simple and reliable relatively low cost.

Zol-gel technology of drawing of coverings coating simple and cheap.

The simple and reliable industrial equipment. Technological process is perspective and attractive because of simplicity.

For preparation of film-forming solutions use salts and ethyl spirit. As a result of their interaction are formed alkoxy combination. Alkoxy combination is decomposed. Oxides are form on the surface of metal. The purpose of work is to achieve good adhesion oxide with a surface of metal.

2. Experiment

Alkoxy compounds were produce from zircon nitrate. For acceleration of reaction added nitric acid.

The alcoholyat of formation reaction occurs with heating. Flasks with reagents are placed in snow.

In Table, the list of solutions which were used for reception alcoholyat is presented.

Table. Seven solutions with different alcoholyat concentration were produced

No.	Component	Quantity
1	zirconium nitrate acid ethyl spirit water	1 1.5 6 1
2	zirconium nitrate acid ethyl spirit water	1 1.5 5 1
3	zirconium nitrate acid ethyl spirit acid water	1 1.5 4 1
4	zirconium nitrate acid ethyl spirit water	1 1.5 3 1
5	zirconium nitrate acid ethyl spirit water	1 1.5 2.6 1
6	zircon nitrate acid ethyl spirit water	1 1.5 2 1
7	zirconium nitrate acid ethyl spirit water	1 1.5 1.3 1

Then alcoholyat zirconium put by watering. Last decomposed in exsiccator. At drawing alcoholyat it is direct on copper samples after decomposition alcoholyat a covering removal.

Destruction of a ceramic layer is the following factors:

– a flat interface between a metal layer and a ceramic layer is formed;

– the linear expansion coefficient are different;

– chemical oxidation and corrosion reactions take place in the contact zone.

To prevent coating exfoliation it was necessity to produce a thermogradient sublayer, that increase the coating service life. This enables the adhesion of

the ceramic layer to the metal one to be improved. A thermogradient transition zone is formed.

Use thermogradient transitive layers promotes increase in durability of coverings. It allows replacing in many cases flat border of section between metal and ceramic layers.

Good adhesion of a covering to a substrate is reached. A composite material is produced by adding a mixture of components with low fusion temperature to the nickel coating.

A composite material may be formed in the course of natural processes or produced artificially as two or multiphase compact material with flat interfaces between individual constituent components.

Thermogradient sublayer was applied to a copper substrate.

The thermogradient coating technique under review consists of two stages:

– a heat and corrosion resistant nickel sublayer is produced by an electrochemical coating is applied;

– a composite electrochemical coating is applied.

In thermogradient coatings. electrochemical nickel sublayer was produced from Uots electrolit.

Another sublayer was a composite electrochemical coating where nickel is the matrix.

The following electrolit were used nickel coating.

Thus the matrix was. as a rule. a continuous solid material with discrete distribution of the second – phase particles. In our case, those were zirconium and magnesium nanooxide.

Sedimentation of nickel conducted from electrolit [1]:

$NiSO_4$, g/l	300.0;
$NiCl_2$, g/l	60.0;
H_3BO_3, g/l	40.0;
t, °C	25–30;
I, A/dm^2	0.8–1.0.

Thus one of phases – the matrix – is usually continuous and is in a firm condition. In a matrix, particles of the second phase are distributed discretely. In our case, it is zirconium nanooxide and magnesium nanooxide. A following sublayer was sedimentation of a composite electrochemical covering. Nickel is the matrix. Zirconium nanooxide with the additive of 3% magnesium oxide magnesium have applied as add. Sedimentation of a composite nickel covering conducted from electrolit:

$NiSO_4$ g/l	300.0;
$NiCl_2$, g/l	60.0;
$NiCl_2$, g/l	40.0;
nanooxide, g/l	1.0;
t, °C	25–30;
i, A/dm^2	0.8–1.0.

3. Results and discussion

Reception of the first sublayer have lead electrochemical sedimentation of nickel.

Figure 1 dependence of increase in an additional weight. i.e., thickness of a nickel covering on time of sedimentation is presented. At drawing nickel by electrochemical way with increase in time of a covering thickness of a coating is increases.

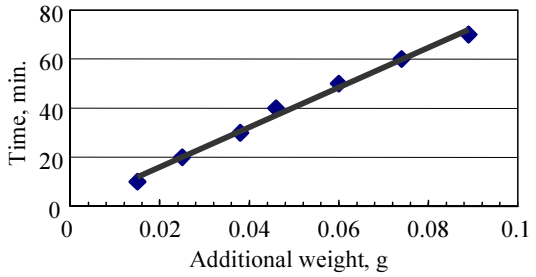

Fig. 1. Shows temporal variations of the gain in weight of nickel

Thermogradient coverings have applied to reception as the second sublayer a composite electrochemical covering with inclusion as the second phase zirconium nanooxide with the additive of magnesium nanooxide.

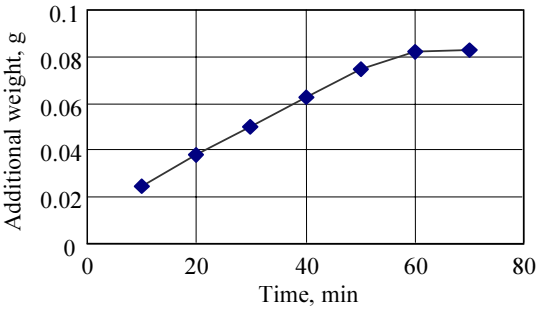

Fig. 2. Shows temporal variations of the gain in weight of nickel coating on addition of 1 g/l zirconium nanooxide with 3% of magnesium nanooxide. Apparently from the schedule is presented at drawing a composite nickel

In alkoxy compounds molecular atoms of constituent a element are bonded with an organic radical via oxygen. By the following layer it is put alcoholyat zirconium. The zirconium alcoholyat put by watering. Flasks with reagents placed during a snow with the purpose of avoidance throw out a solution. Seven solutions with different alcoholyat concentration were produced Table.

Last decomposed in dry to a case zirconium oxide – a high-temperature component. High-temperature (cubic) form ZrO_2 a rack at $T > 2100$ K. At cooling it passes in the tetragonal form. The volume noticeably increases for 4–5%. It leads to occurrence of cracks in products from zirconium oxide. At addition MgO to zirconium oxide polymorphic transition is excluded down to cooling up to a room temperature. Therefore manufacturing of coverings and details is carried out from their mix. [2]

The coating put on a sublayer. has passed thermocyclic tests. The maximal temperature of heating has made 980 °C. After the first 30 s the coating became gray-brown.

In the following 30 s 3% of a covering zirconium oxide are broken. Still through 30 s 5% of a coating zirconium oxide are broken.

After 1 min of heat treatment 8% of a coating zirconium oxide are broken. In next minute heat treatments have appeared cracks in a nickel coating. In 2 min thermocyclic copper began to look through on 8% of the area of the sample. In next minute – on 12% of the area of the sample. In next minute – on 25% then in a minute – on 40% and through following – on 60% of the area there was a destruction of a coating.

4. Conclusion

Thermocyclic tests show that the adhesion of the coating deposited to nickel sublayers with nanooxides is of 40% higher than that for coating deposited directly to copper substrate.

References

[1] Russian State Standart 9.305 Metallic and inorganic coatings. Operations of technological processes of reception of coverings.

[2] R.S. Sajfullin, *Physicochemistry of inorganic polymeric and composite materials*, Moscow, Khimiya, 1990, 240 pp.