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METHOD OF PRODUCING FINELY DIVIDED METALLIC LAYERS

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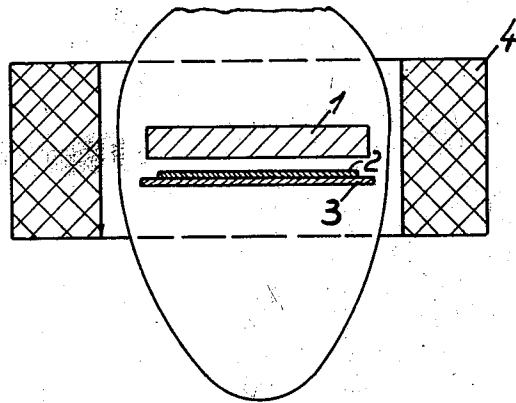


Fig. 1

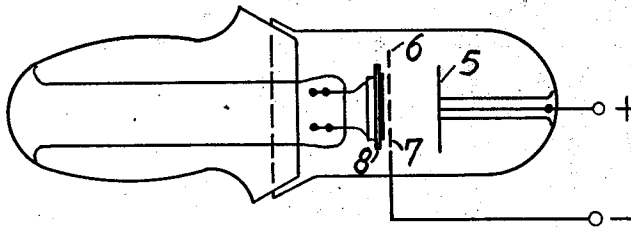


Fig. 2

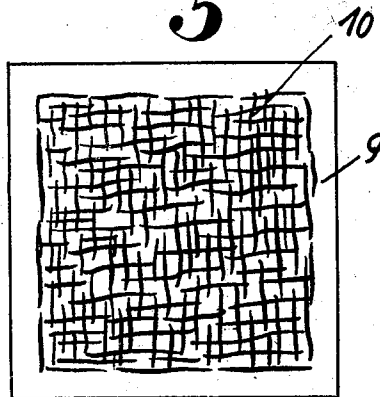


Fig. 3

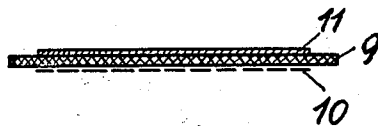


Fig. 4

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METHOD OF PRODUCING FINELY DIVIDED
METALLIC LAYERS

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9 Claims. (Cl. 91—70.1)

The present invention relates to a method of producing metallic layers on insulating materials, such, for example, as mica or the like, whereby the metallic layer consists of the finest particles, which are insulated against each other, and represents in a manner of speaking a mosaic screen composed of single particles of metal. A screen of this nature may be employed more particularly in certain television image recording methods, in which the images to be transmitted are recorded with the assistance of an Ikonoscope camera. After corresponding treatment the mosaic screen represents a surface collection of elementary photo-cells, which produce electrical charges corresponding with the particular exposure. These charges may then further be employed for controlling the transmitter in suitable fashion.

The production of the layer consisting of finest particles of metal is accompanied by considerable difficulties, both as regards selection of the material for the intermediate insulating layer as well as the method of insulation.

It is possible to employ mica for the intermediate insulating layer. The use of this material when employing a heating process for dividing a metallic layer into single elementary parts is confronted by difficulties owing to the inconstancy of the temperature and the nature of the surface. When employing a method for producing the distribution by means of engraving machines or in the case of chemical treatment it is impossible in numerous cases, owing to the small powers of adhesion, to produce a metallic layer in equal distribution.

The methods set forth in the present invention are devoid of these disadvantages.

According to the invention, there are employed as intermediate dielectric layers sheets of mica or other suitable bases, for example, ceramic material, certain kinds of glass, etc., the surface of this base preferably being made smooth by polishing. On this base there is produced in suitable fashion a metallic coating, for example a silver, copper or the like coating. A metallic coating of this nature may be produced, for example, chemically by cathode atomization or by thermal application in vapour form. The metallized plates are then heated to a high temperature in an atmosphere of indifferent gas or in a vacuum. If the metallic coating is of a certain thickness (no longer transparent), the temperature is required to be very high, for example in the case of silver to about 1000°, in order to obtain a brief fusing of the layer of silver, whereby the surface is then converted from the previously unitary

structureless metallic surface into a very even mosaic consisting of small drops of metal of negligible diameter and insulated against each other. According to the invention, this heating is performed extremely rapidly, preferably by means of eddy current heating in high-frequency furnaces. In this connection the metallic coating is acted upon not directly, but indirectly by radiation from a heating plate. In the case of very thin metallic layers it is possible to manage with considerably lower temperatures, which in view of the low temperature constancy of mica is of advantage when employing a material of this kind. When using other materials, such as ceramic substances, glass or the like, the surface of the base is preferably made smooth by polishing, in order to accelerate the insular formation during the heating process.

According to the invention, it is also possible to deposit the metallic layer on a heated base, so that small globules are likewise formed right from the commencement. When using mica, however, the method of subsequently heating the deposited metal as described in the above is to be preferred.

An additional method for insular formation resides according to the invention in the fact that a thin layer of silver deposited, for example, on mica is alternately oxidized and reduced. This method is performed in the most convenient form by glow discharge in an atmosphere of alternately introduced oxygen and hydrogen at suitable pressure (approximately 1 mm. mercury). The alternate oxidation and reduction might also be replaced to a certain extent by a suitable temperature treatment. In this connection the silvered plate is heated in oxygen at first up to the temperature for formation of silver oxide, and then additionally heated in vacuum until the silver oxide has become dissociated. The temperature is thereupon again lowered, etc. In the case of the glow discharge method the silver layer to be granulated is in the most convenient form placed opposite to a plate connected up as anode, with the intermediate connection of a grid-like electrode connected up as cathode. In this respect the layer of silver does not possess any electrically conductive connection with the cathode. The layer of silver might also be connected up as cathode. In this, however, there has been found to exist the disadvantage that the oxidizing and reducing process takes place more strongly in the vicinity of the connection between the lead and the layer than at other points. As final stage of the process, after production of the discrete particles of silver, there is conveniently performed

a light oxidation, as in this manner it is possible to obtain a layer which is advantageously suited for photo sensitization.

The drawing shows diagrammatically how to carry out the method according to the invention. In the drawing Fig. 1 shows the indirect heating by means of a heating plate 1, which heats by radiation the layer of silver 2 mounted on the supporting material 3, for example mica. Since this heating is required to commence instantaneously, the heating is preferably performed by eddy currents, which is represented diagrammatically by the high-frequency coil 4.

Fig. 2 shows the arrangement for carrying out the glow discharge process. In the same there is provided an electrode 5, which is connected up as anode and is situated opposite to a grid-like cathode 6. The metallic layer 7 to be granulated is arranged within reach of the glow discharge which develops in the case of a suitable filling of gas. 7 is the metallic layer on the base 8. In Figs. 3 and 4 there is illustrated by way of example the finished product. In these 9 is the intermediate di-electric layer, which acts as base for the coatings 10 and 11. The coating 10 represents the thin layer of metal divided up into finest elementary particles, whilst 11 is intended as capacitative counter-coating for the photocell arrangement.

The following special embodiments of the present invention have been proved useful:

A well-cleaned mica plate with all grease removed is coated by the thermal vaporization of silver with a silver layer of approximately $\frac{1}{10}$ – $\frac{1}{100}\mu$ in thickness. A thickness of the layer approximately midway of the stated range, i. e., approximately $\frac{1}{100}$ to $\frac{1}{1000}\mu$ produced the best results. The application of the layer by vaporization is performed in a suitable vessel from which the air has been removed, the distance between the mica plate to be treated and the evaporation furnace being of such extent that a very even coating is obtained. Insulation edges of a desired kind may be allowed to remain by selective screening. After the application of the silver layer by vaporization has been completed a certain amount of air or oxygen is allowed to enter the vessel, and the part of the vessel in which the plate is situated, is heated to a temperature below 500° C. approximately 400–450° C. This latter operation extends over a period of approximately 15 to 30 minutes. After cooling it will be noticed that the coating on the plate has been divided into single well-separated crystals of about 1μ in size.

It has been found that to obtain this effect it is necessary

1. To adhere to the above stated range as regards the thickness of the layer, and
2. To employ a suitable gas atmosphere.

If the initial layer is made thinner, a "granulation" of any kind is not to be determined with the usual optical means available. If the layer is too thick, there is certainly a granulation up to a certain point, but the particles are situated so close together that the requisite insulation may be not obtained. If on the other hand the layer is still thicker (completely opaque), a distribution according to the described process may be not accomplished at all. In this case temperatures would require to be employed which come very near to the fusing point of the silver (800 to 1,000° C.), and considerable difficulties are then encountered in preventing the mica from becoming entirely useless. The size of the formed particles is also a wholly different one in the last

mentioned case. Further, it has been found that even with suitable layers a separation does not take place when the process is performed in an extremely high vacuum (higher than 10^{-5} mm. Hg). The presence of a certain gas atmosphere (presumably oxygen) is accordingly necessary for the separation at the low temperature stated.

Mica plates of this kind, which are furnished with a silver screen in the manner stated, may then be employed in the known fashion, by silvering the rear side and by oxidation and sensitization of the front screen surface, as light-sensitive mosaic electrodes in television transmission tubes, more particularly tubes known under the name of "Ikonoscope".

I claim:

1. A process of producing a finely subdivided metallic layer on an insulating base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade.

2. A process of producing a finely subdivided metallic layer on an insulating base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade during 15 to 30 minutes.

3. A process of producing a finely subdivided metallic layer on an insulating base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of 400 to 450° centigrade.

4. A process of producing a finely subdivided metallic layer on an insulating base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade by causing the radiation of a plate heated by eddy currents to act on said coated base.

5. A process of producing a finely subdivided metallic layer on a mica base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade.

6. A process of producing a finely subdivided metallic layer on a glass base comprising the steps of coating said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade.

7. A process of producing a finely subdivided metallic layer on a base of a ceramic material comprising the steps of coating said base with silver, of interrupting this coating action when

a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade.

5 8. A process of producing a finely subdivided metallic layer on a glass base comprising the steps of polishing the surface of said base, of coating the polished surface of said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen

at a temperature of at least about 400° centigrade and below 500° centigrade.

9. A process of producing a finely subdivided metallic layer on a base of ceramic material comprising the steps of polishing the surface of said base, of coating the polished surface of said base with silver, of interrupting this coating action when a layer of 0.01 to 0.1 μ thickness has been formed, and of subjecting the coated base to a thermal treatment in a gas atmosphere containing oxygen at a temperature of at least about 400° centigrade and below 500° centigrade.

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