

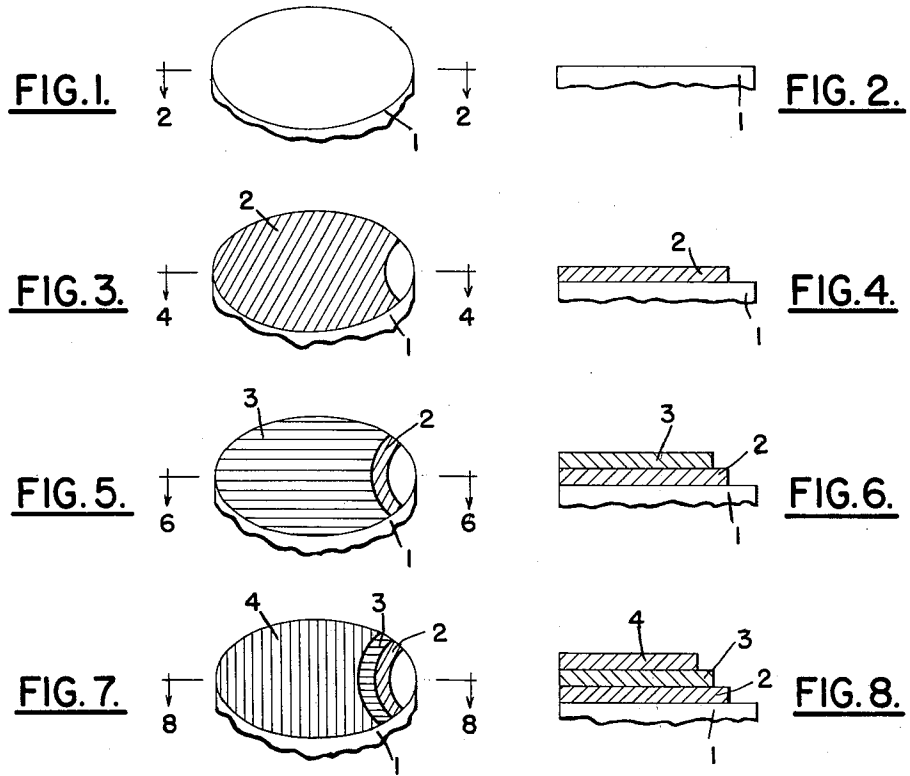
May 15, 1962

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3,034,916

METHOD OF OXIDIZING TITANIUM METAL

Filed April 6, 1959



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3,034,916

METHOD OF OXIDIZING TITANIUM METAL

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Filed Apr. 6, 1959, Ser. No. 804,557

3 Claims. (Cl. 117—33.3)

(Granted under Title 35, U.S. Code (1952), sec. 266)

The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment to me of any royalty thereon.

This invention relates to optical devices and their production wherein a layer having a low index of refraction such as a layer of magnesium fluoride or the like is coated with a layer having a high index of refraction such as titanium dioxide. It provides an improved method whereby the multilayer titanium dioxide coating is deposited more rapidly and less expensively than has been possible heretofore.

In oxidizing these multiple layers of titanium dioxide onto a magnesium fluoride surface it has been heretofore considered necessary but difficult (1) that the titanium metal be deposited in the same jar of the vacuum system as the underlying film having a low index of refraction without breaking the vacuum, and (2) that the magnesium fluoride obtained in one vacuum must not be placed in another vacuum for titanium deposit because gases and vapors absorbed in the magnesium layer, when exposed to air, will remain there to cause serious damage later when the titanium is furnace oxidized. This old method is expensive and time consuming. In accordance with the present invention it is replaced by an improved method which involves furnace oxidizing the titanium metal in air to titanium dioxide, this being accomplished in steps between which the coated object is allowed to cool to a predetermined temperature.

Oxidation of a film of titanium metal over an underlying layer having a low index of refraction such as magnesium fluoride in multilayers causes extensive irreparable damage to the low index layer after two hours at 820° F. Four hours at this temperature is usually required for complete oxidation. Gases and vapors absorbed by the low index film almost as soon as it is deposited and trapped when the overlying titanium layer is furnace oxidized at high temperature are believed to produce the damage to the magnesium fluoride layer.

FIGS. 1 and 2 are perspective and edge views respectively of an optical element with its lower portion broken away.

FIGS. 3 and 4 are similar views of the same disc with a layer of titanium dioxide thereon but without the gold and copper oxide intervening layers shown in FIG. 2 of Morgan 2,750,832, dated June 19, 1956.

FIGS. 5 and 6 show a layer of magnesium fluoride thereon in the usual manner.

FIGS. 7 and 8 are similar views with a layer of titanium dioxide deposited thereon over the fluoride layer.

In multilayer uses of these layers as beam splitters in an optical device 1, i.e. in which some of the layers transmit and absorb light differently from other layers, the suggestion for a magnesium fluoride layer 3 between layers 2 and 4 of titanium dioxide is not new. This invention may be viewed as a method whereby the titanium may be oxidized more rapidly yet without injury to the magnesium fluoride in absorbed gas.

From these considerations it is evident that the problem

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to be solved is that of furnace oxidizing titanium metal in air in such a way as to coat a surface having a low index of refraction with titanium dioxide without injury to the low index surface.

5 The present invention solves this problem by oxidizing in two steps. The first oxidation is continued for approximately thirty-five minutes at about 820° F. The coated article is then cooled to a temperature of 400° F. after which the temperature is increased to 820° F. and is maintained at this value until the oxidation is complete. The usual time to reach the lower temperature is about forty-five minutes and the usual time to return to the higher temperature is about fifteen minutes. It has been found that oxidation controlled in this manner permits continuing at the high oxidizing temperature for as much as five times the required time without damage to magnesium fluoride layer.

10 It is of course possible to oxidize the titanium metal onto a layer of material having a low index of refraction at temperatures lower than 820° F. without injury to the low index surface. It is found, however, that oxidation at these lower temperatures greatly extends the time required to complete the oxidation. As the result of extensive tests it has been found that the herein described oxidizing method reduces the required time to a minimum for the best product. It is to be understood that the temperature of 820° F. may be varied by five degrees in either direction without considerably extending the time required to complete the oxidation. This range of temperatures is hereinafter designated as "a temperature of the order of 820° F."

15 It is not definitely known why cooling at an intermediate stage in the deposition of the titanium dioxide eliminates the deleterious effect of the gas and vapor in the magnesium fluoride layer. It is possible that at this stage of the operation, the deposit is porous enough to allow the gas and vapor to be driven out by the heat and that the cooling time is such as to permit ejection of the gas and vapor to be completed. However this may be, it is evident that the herein disclosed method is less expensive and involves much less time than the method previously used.

I claim:

1. In a method of oxidizing a titanium metal layer in air onto a magnesium fluoride surface having a low index of refraction for an optical device, the steps which include preliminarily oxidizing said metal layer at a temperature of the order of 820° F. for a time of the order of 35 minutes, cooling said surface to a temperature of about 400° F. and completing said oxidation at a temperature of the order of 820° F.

2. A method according to claim 1 in which the cooling to about 400° F. is at a rate taking about 45 minutes, the return to 820° F. is at a more rapid rate than the cooling and is at a rate taking about 15 minutes, and the reheating at 820° F. is continued for about 4 hours until oxidation of the titanium is completed.

3. A product of the process of claim 2 in which the magnesium fluoride is substantially freed from gaseous impurities.

References Cited in the file of this patent

UNITED STATES PATENTS

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2,732,313	Cusano et al.	Jan. 24, 1956
2,750,832	Morgan	June 19, 1956
2,789,062	Cusano et al.	Apr. 16, 1957