

## Titanium Oxides For Optical Coating

### Applications

Titanium dioxide ( $\text{TiO}_2$ ) has been used in optical coatings for the visible region for many years.  $\text{TiO}_2$  has two particularly desirable qualities. It provides the highest index film material for the visible region, and it is hard and stable in combination with other oxides. Coating examples using  $\text{TiO}_2$  as the high-index material include beam splitters, cold mirrors and heat-reflecting mirrors, and AR on glass and polymer substrates. Absorption below 450 nm limits the applications to the visible and near-ir regions. When paired with  $\text{SiO}_2$  films, multilayer combinations totaling in excess of 50 layers have been deposited that show excellent durability and low mechanical stress because the tensile stress of titania layers is balanced by the compressive stress of the silica layers.

### Evaporation Notes

Titanium and oxygen form a number of stable phases, the most practical being  $\text{TiO}$ ,  $\text{TiO}_2$ ,  $\text{Ti}_2\text{O}_3$  and  $\text{Ti}_3\text{O}_5$ . Any of these compositions can be evaporated and subsequently oxidized to the final stable phase,  $\text{TiO}_2$ . The lower evaporation temperatures of the latter two compounds is an advantage because particle ejection and strong outgassing is avoided. The resultant titania layers are composed of columnar microcrystallites with appreciable void volume and undesirable optical absorption unless films are grown under high-energy conditions such as high substrate temperature or Ion Assist (IAD) or by sputtering. IAD has been found to produce the highest refractive indices, yet the

structure has neither the pure rutile or anatase long-range crystal phase.

When heated above their melting points, titanium oxides decompose into suboxides and liberate oxygen. It is therefore necessary to prepare a dense homogeneous slug of melted material before beginning evaporation. The material should be conditioned as follows. Condense the starting material into a homogeneous mass by melting at low power, using a swept beam. Oxygen evolves during this process, leaving a black, oxygen-deficient melt. Repeated fillings of the crucible will be required unless a pre-melted cone is used as the starting charge. After the outgassing stops, the melt can be raised to evaporation temperature. The evaporant will re-oxidize reactively in the supplied partial pressure oxygen to form films of colorless  $\text{TiO}_2$ .



All of the titanium oxides can be evaporated by resistance heating or electron beam. If Tungsten crucibles are used, some reaction between container and material results. For electron beam evaporation, Tantalum or water-cooled copper crucibles are recommended.

### Advantages of Using $\text{Ti}_3\text{O}_5$ Starting Material<sup>(1,2)</sup>

Whenever any of the various oxide states of Titanium (see above) is evaporated, the vapor consists of various Ti-O combina-

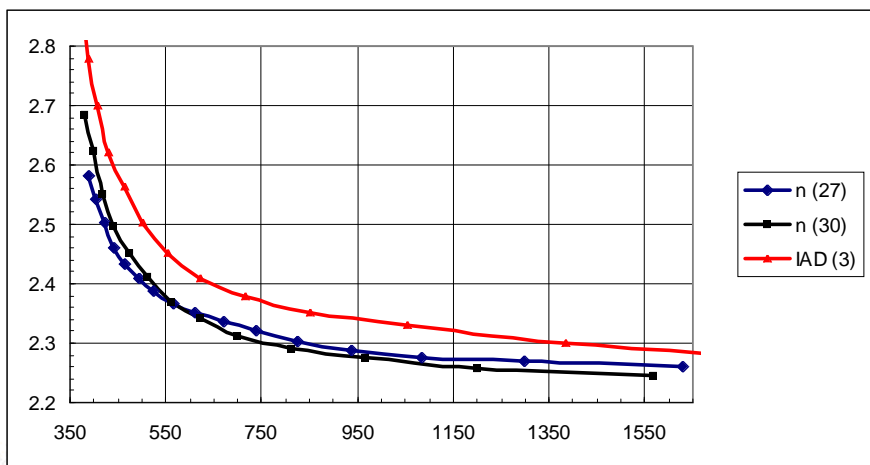


Figure 1. The use of IAD increases the refractive index ~2%.

tions, which change in oxygen content as the evaporation proceeds. The refractive index of the deposited film thus is not constant because most Ti-Ox materials evaporate incongruently.

Starting with Ti<sub>3</sub>O<sub>5</sub> composition ensures that the only titanium species in the vapor is TiO, and the oxygen content in the vapor remains constant. Also the Ti<sub>3</sub>O<sub>5</sub> composition has the advantage that it melts and thus a constant rate and pressure can be established for the duration of the deposition process.

### Optical Properties

As seen in figures 1 and 2, the curves show the influences of IAD and post-deposition air baking on the refractive indices. The air bake consisted of holding the film layer at 400°C for 4 hours.

The application of IAD increases the extinction coef. from <0.002 to ~0.005 in the visible region. The practical useable wavelength limit (when k ~0.01) for the IAD film is ~400 nm.

### Evaporation Parameters

|                                     |   |
|-------------------------------------|---|
| Source                              | Electron Beam   |
| Crucible                            | Ta, water-cooled copper   |
| Substrate temperature               | 250°C min.  |
| Rate of condensation                | -3-5 Å/sec.   |
| Partial pressure of supplied oxygen | -1.2 x 10 <sup>-4</sup> Torr  |
| Refractive Index of film            | 2.4 at 450nm<br>2.35 at 500nm<br>2.26 at 700nm<br>2.20 at 1000-1200nm |

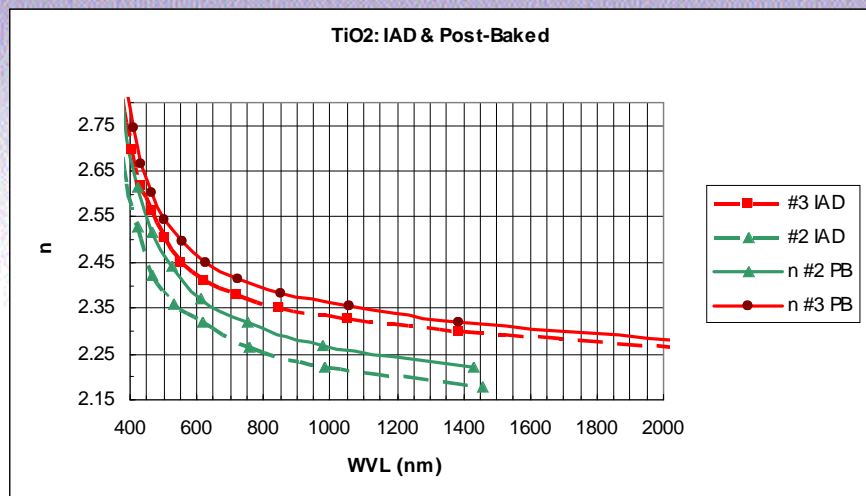


Figure 2. Post-deposition air baking increases the index further.

### Forms and Sizes Available

CERAC offers materials for evaporation as well as sputtering targets. To view pricing on our standard catalog items, please visit our on-line catalog at [www.cerac.com](http://www.cerac.com) and look-up by item number, chemical name or CAS number. If you require a custom manufactured item, please contact our sales department at +1-414-289-9800 or [cerac-sales@beminc.com](mailto:cerac-sales@beminc.com) with your specific require-

| Formula                        | Item Number      | Description  |
|--------------------------------|------------------|--|
| TiO                            | T-2038           | Gold tablet 8-9mm dia. x 6-7mm thick                         |
| TiO <sub>2</sub>               | T-1183           | White tablet, fully oxidized, 8-9mm dia. x 4-5 mm thick cone |
|                                | T-1192           | 3-6mm irregular sintered pieces, fully oxidized              |
|                                | T-5003<br>T-5503 | Pre-melted ingot<br>Pre-melted, sized pcs                    |
| Ti <sub>2</sub> O <sub>3</sub> | T-1257           | Purple tablet 8-9mm dia x 6-7mm thick                        |
| Ti <sub>3</sub> O <sub>5</sub> | T-2039           | Black tablet 8-9mm dia x 7-9mm thick                         |
| Ti <sub>3</sub> O <sub>5</sub> | T-2100           | 1-4 mm pieces  |

ments. You can also fill out our quotation request form.

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### References

- "Refractive Indices of TiO<sub>2</sub> Films Produced by Reactive Evaporation of Various Titanium-Oxygen Phases" H.K. Pulker, G. Paesold and E. Ritter, Applied Optics, Vol 15, No. 12 (1976), p. 2986-2990.
- "Method of Depositing Titanium Dioxide Layers by Evaporation of a Molten Titanium-Oxygen Phase," H.K. Pulker, U.S. Patent 3,927,228.

CERAC, inc.  
Subsidiary of Williams Advanced Materials  
P.O. Box 1178  
407 N. 13th Street  
Milwaukee, WI 53201-1178 USA  
Phone: +1-414-289-9800 Fax: +1-414-289-9805  
[ceracinfo@beminc.com](mailto:ceracinfo@beminc.com) [www.cerac.com](http://www.cerac.com)

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