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Photostability of Coatings Based on TiO_2 (Rutile) Doped with Potassium Peroxoborate

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The effect of the doping of titanium dioxide powders with potassium peroxoborate on the photostability of reflecting coatings based on these powders is investigated. An increase in the stability of the diffuse reflection spectra and of the total absorptance on exposure to radiation that imitates the solar spectrum is established for oxidant concentrations up to 30 mass % with saturation of the efficiency at a concentration of 15 mass %. It is demonstrated that, on irradiation, the doping leads to a decrease in the absorption bands of color centers caused by defects of the anionic sublattice.

Nomenclature

a_s = integral solar absorptance factor
t = exposure time
Δa_s = change in integral solar absorptance
$\Delta\rho$ = change in diffuse reflectance
$\Delta\rho_\lambda$ = change in diffuse spectral reflectance ($\Delta\rho$ spectrum)
ε = thermal emittance
λ = wavelength
ρ_λ = diffuse reflection spectra
τ = time constant of the processes of forming the absorption centers

Introduction

TITANIUM dioxide powders are used as pigments of reflecting thermal control coatings (TCC) of space vehicles due to low values of the total solar absorptance a_s and high values of the emissivity ε. As a rule, the emissivity of reflecting TCC changes insignificantly,[1] and the TCC lifetime is limited by a gradual increase in the absorptance a_s to large values due to coloring on exposure to solar electromagnetic radiation (EMR), electrons, protons, and other factors of the space environment.[2] Therefore, the development of production technologies of TCC for space vehicles based on titanium dioxide that possess photo- and radiation stability is an actual problem.

The degradation of the TiO_2 pigment due to its nonstoichiometry with respect to oxygen causes a low stability of the lattice, and the presence of anionic vacancies that form F and F^+ color centers with free electrons generated on exposure to radiation reduces the reflectance and increases the total absorptance a_s. This disadvantage can be compensated by the doping of the pigment with chemical elements capable of supplying additional oxygen atoms to restore the normal stoichiometric composition not only of initial but also of irradiated powders when oxygen of the lattice escapes from the pigment in the process of photolysis or radiolysis. Such chemical elements, oxidants, easily donate oxygen atoms. Investigations of this problem were also conducted at the Centre National d'Etudes Spatiales (CNES), Toulouse, France.[3] Specialists from CNES studied the influence of doping by different oxidants on increasing integral coefficient of absorption a_s of TiO_2, ZnO, and Zn_2TiO_4 pigments in action under electron, proton, and ultraviolet irradiation. The present study analyzes the effect of the doping by potassium peroxoborate of the TiO_2 pigment on the diffuse reflection spectra ρ_λ, absorptance, and their changes on exposure to EMR.

Experiment

A potassium peroxoborate (PPB) modifier with concentrations of 5, 10, 15, and 30 mass % was mixed in a distillate with the TiO_2 pigment of specified purity grade to obtain a homogeneous mass. Then water was vaporized, and the mixture so obtained was ground and heated in a muffle furnace. The doped TiO_2 powder was then mixed with a KO-859 silicone varnish binder in the ratio 0.6 : 0.4. Then a thin layer (with a thickness of ≤ 1 mm) of this mixture was deposited on an AD-1 M alloy substrate and dried at room temperature for 24 h. Samples of the coatings were put on a stage of the Spektr-M equipment,[4] pumped out to 10^{-5} Pa, and their diffuse reflection spectra were registered point by point with steps of 10–50 nm in the wavelength range 320–2100 nm. The error in determining ρ was 0.2–0.3% for the wavelength range 0.32–1.1 μm and 2% for longer wavelengths. The total absorptance a_s was calculated from the well-known formula[2]

$$a_s = 1 - R_s = 1 - \frac{\int_{\lambda_1}^{\lambda_2} I_\lambda \rho_\lambda \, \mathrm{d}\lambda}{\int_{\lambda_1}^{\lambda_2} I_\lambda \, \mathrm{d}\lambda} = 1 - \frac{\sum_{i=1}^{n} \rho_\lambda}{n} \tag{1}$$

where R_s is the total reflectance of solar radiation, ρ_λ is the spectral reflectance, I_λ is the solar radiation intensity in the wavelength range λ_1–λ_2 (320–2100 nm), and n is the number of equienergy ranges of the solar spectrum.

As a source of EMR imitating the solar spectrum, a DKSShRB-5000 arc xenon lamp was used. To choose an optimal oxidant concentration, the examined samples were exposed to radiation with the intensity by a factor of 2.1 greater than the solar radiation intensity [1 equivalent of solar radiation (ESR) $= 0.139$ W/cm$^2 \cdot$ s] in the UV range (2.1 ESR) at temperatures in the range 305–308 K for 5 h. The kinetics of changes in the spectra ρ_λ and absorptance a_s of the coatings with 0, 5, and 30 mass % PPB was investigated for 67.5 h. The spectra ρ_λ were recorded every 10–11 h from the beginning of the irradiation.

Results and Discussion

Figure 1 shows the spectra ρ_λ of nonirradiated coatings where it can be seen that all coatings have high reflectance in the visible and near-infrared (IR) ranges of the spectrum. The edge of the fundamental absorption band is at 405 nm, which is in satisfactory agreement with the rutile band gap, 3.02 eV (Refs. 5 and 6) In the wavelength range 500–1500 nm, no qualitative differences are observed in the spectra of different coatings; the average difference of the absolute values of the reflectance is 5%. For $\lambda > 1500$ nm, the reflectance of all samples is reduced to 70%.

Table 1 gives the total absorptance calculated from these spectra. From Table 1, it follows that the total absorptance is virtually

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Table 1 Dependence of total absorptance a_s and its changes vs PPB concentration in pigment on exposure to EMR

Composition of the coating, mass %	a_s	Δa_s
TiO_2 + KO 859	0.236	0.137
(TiO_2 + 5% PPB) + KO 859	0.281	0.091
(TiO_2 + 10% PPB) + KO 859	0.271	0.087
(TiO_2 + 15% PPB) + KO 859	0.223	0.078
(TiO_2 + 30% PPB) + KO 859	0.258	0.08

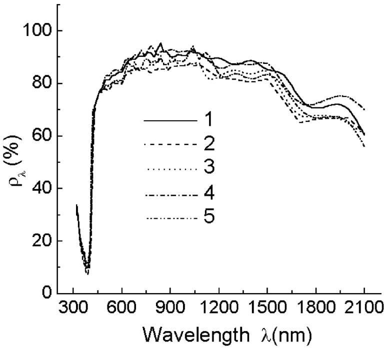

Fig. 1 Diffuse reflection spectra of coatings based on TiO_2 pigment doped with PPB at concentrations of 0 (curve 1), 5 (curve 2), 10 (curve 3), 15 (curve 4), and 30 (curve 5) mass %.

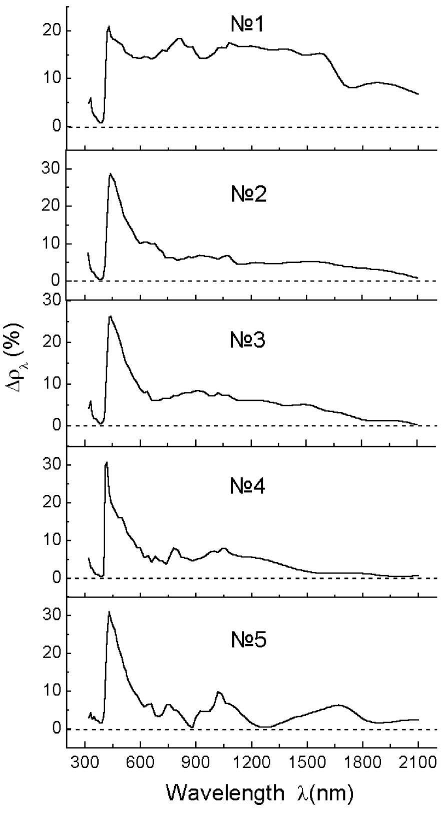

Fig. 2 Induced absorption spectra $\Delta\rho_\lambda$ of coatings based on TiO_2 pigment doped with PPB at concentrations of 0 (number 1), 5 (number 2), 10 (number 3), 15 (number 4), and 30 (number 5) mass % on exposure to EMR for 5 h.

Table 2 Dependence of induced absorption of coatings $\Delta\rho_\lambda$ on PPB concentration in TiO_2 pigment on exposure to EMR for 5 h

	λ, nm		
C, %	430	740	1040
0	20.9	15.8	16.5
5	27.1	6.2	6.4
10	26.0	6.6	7.5
15	23.1	3.8	8.0
30	31.1	6.4	9.4

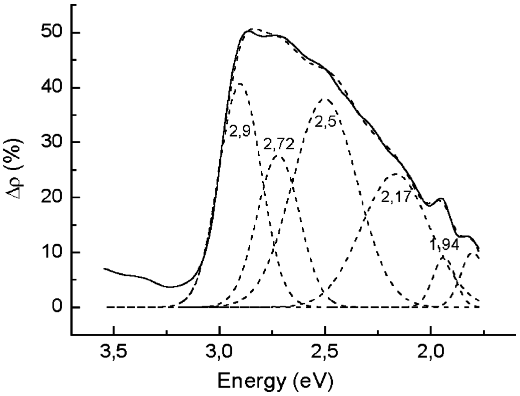

Fig. 3 Decomposition of 430-nm induced absorption band of coatings based on TiO_2 pigment doped with 30 mass % of PPB into elementary components of Gaussian shape on 5-h exposure to EMR.

independent of the PPB concentration in the pigment: The minimum values of the total absorptance are characteristic of coatings with the pigment doped by 15% PPB and with the undoped pigment, whereas the maximum values are characteristic of coatings with PPB concentrations of 5 and 10%.

Figure 2 shows the different diffuse reflection spectra of coatings $\Delta\rho_\lambda$ obtained by subtraction of the spectra recorded after exposure to EMR from the spectra recorded before irradiation. Because the transmission of coatings does not change on exposure to radiation, the difference spectra are the absorption spectra induced by irradiation. Changes are qualitatively identical for all spectra $\Delta\rho_\lambda$: A clearly pronounced absorption band centered at 430 nm is observed in the visible range together with weak longer wavelength absorption maxima at 650, 780, and 1050 nm.

A distinguishing feature of these spectra is that the absorption of coatings with the doped pigment in the long wavelength range from the 430 to the 2100 nm band is less than that of the coating with the undoped pigment. Moreover, the higher the PPB concentration in the pigment, the lower is the absorption in this wavelength range. For vivid presentation, Table 2 gives values of $\Delta\rho_\lambda$ at wavelengths of 740 and 1040 nm from which it follows that $\Delta\rho_\lambda$ decreases by a factor of 2.5 (from 15.8 to 6.4%) for $\lambda = 740$ nm and by a factor of 1.75 (from 16.5 to 9.4%) for $\lambda = 1040$ nm when the PPB concentration increases from 0 to 30 mass %. Note that the dependence of the intensity of the 430-nm band on the PPB concentration is opposite that of the intensity of bands in the near-IR range, namely, $\Delta\rho_\lambda$ increases from 20.9 to 31.1%, that is, by a factor of 1.5 when the PPB concentration in the pigment increases from 0 to 30 mass %.

In the experimental induced absorption spectra of irradiated coatings, a clearly expressed band centered at 430 nm is recorded. The decomposition of this band into elementary components yields four bands with maxima $E_{\max}$ at 428, 456, 496, and 570 nm and half-widths $H_{1/2}$ of 0.2, 0.2, and 0.3 and 0.32 eV (Fig. 3). Their area $S = H_{1/2} \cdot \Delta\rho$, is 8.1, 5.6, 11.4, and 7.8 eV · %, respectively. When it is assumed that the oscillator forces for color centers are identical, the defect concentrations that determine the absorption in these bands will differ by no more than double, that is, they will be of the same order of magnitude. An analysis of the literature data on the nature of the TiO_2 (rutile) absorption bands demonstrates that the absorption bands in the spectra from the fundamental absorption band edge to 2 μm can be caused by the following defects of crystalline structure. The first band near the fundamental absorption band edge ($\lambda_{\max} \leq 420$ nm) is analogous to that of alkali–halide crystals (AHC) (Ref. 7), that is, it can be caused by anionic vacancies. The band at 3.0 eV (413 nm) was detected in the difference reflection spectra of ideal and actual rutile at 77 K (Ref. 8). The next absorption bands can be caused by Ti^{2+} ions and titanium vacancies. In Ref. 8, the analogous band with a maximum at 2.9 eV (427 nm) was detected after decomposition of the difference reflection spectrum.

The band caused by Ti^{3+} ions also falls within the experimental band centered at 430 nm. It was repeatedly recorded in the absorption spectra of TiO_2 and minerals incorporating titanium ions.[9,10] The band maximum was in the wavelength range 500–580 nm, depending on the state of titanium dioxide or compounds incorporating titanium ions.[5,6,9–15]

There are no clearly pronounced bands in the near-IR range. The band with a maximum at 800 nm is attributed to anionic vacancies, incorporated titanium, or simultaneous presence of these and other defects. The position of the maximum of this band is determined by the degree of TiO_2 deoxidation. The absorption in this wavelength range is determined by F centers (800–1200 nm), polarons of small radius (1400–1600 nm), and F^+ centers (1700–1900 nm) (Refs. 9, 14, and 15).

No absorption bands of titanium vacancies in different charge states and Ti^+ ions are known in the scientific literature. However, by analogy with AH crystals and ZnO and ZrO_2 pigments, we can assume that the bands of titanium vacancies (V centers) and interstitial titanium ions must have energies higher than those of F- and F^+-center bands (F, R, M, and other bands of electron color centers). Therefore, the 430-nm band arising after irradiation and its elementary components can be attributed to the bands caused by defects of the cationic sublattice.

From the induced absorption spectra, it can be seen that the maximum values of $\Delta\rho_\lambda$ are characteristic of the entire spectrum of coatings based on the undoped pigment, whereas the minimum values are characteristic of the coating based on TiO_2 doped with 30 mass % of PPB. The doping notably affects the concentration of defects in the cationic and anionic sublattices: It reduces the concentration of F and F^+ centers on irradiation compared to the concentrations in the coating with the undoped pigment and increases the concentration of color centers caused by defects of the cationic sublattice.

This demonstrates that PPB decomposes on exposure to radiation and liberates oxygen, which substitutes for oxygen atoms leaving the pigment. The simultaneous increase in $\Delta\rho_\lambda$ in the maximum of the 430-nm absorption band is most likely due to an increase in the concentration of the earlier examined defects of the cationic sublattice in the process of photolysis of titanium dioxide.

The total absorptance a_s on exposure to radiation reproduces the dependence of changes in the main part of the diffuse reflection spectra (for $\lambda > 430$ nm) on the PPB concentration in the pigment (Table 2): The higher the oxidant concentration, the less pronounced are changes in the reflection spectra and absorptance a_s.

The induced absorption spectra of coatings with the pigment treated by the PPB oxidant in which the intensity of the absorption band near the edge of the fundamental absorption band (in our case, near the edge of the band centered at 430 nm) is much greater than the absorption intensity in the near-IR range that are characteristic of coatings with the oxidized pigment. The same spectra $\Delta\rho_\lambda$ are characteristic of anatase powders prepared using the fluoride technologies,[16–18] in which the sample surface is oxidized and the sample volume is saturated with the oxidant. The same spectra were recorded for American coatings based on anatase and rutile[19] in which the pigment was most likely treated in an oxidizing atmosphere (in oxygen or fluorine). The Russian pigments are not oxidized; therefore, the relationship between the band intensities in the visible and near-IR ranges of the induced absorption spectra differs for these pigments[20,21] and coatings based on these pigments,[22] namely, they are either equal to each other or (which is more often) the intensity of the band in the near-IR range is higher.

The kinetic dependences of the spectra $\Delta\rho_\lambda$ and absorptance a_s on the exposure time were investigated for three coatings: with the undoped pigment and with the pigment doped by 5 and 30 mass % of PPB. Figure 4 shows the spectra $\Delta\rho_\lambda$ for the indicated exposure times, and Fig. 5 shows the kinetics of changes in the absorptance a_s for these three types of coatings. From Figs. 4 and 5, it can be seen that the most pronounced changes are observed during the first 15–20 h of irradiation; then the rate of their increase notably decreases. The greatest changes are observed for the coating based on the undoped pigment. No qualitative changes are observed in the spectra $\Delta\rho_\lambda$ compared to the changes observed for shorter exposure time (5 h; Fig. 2).

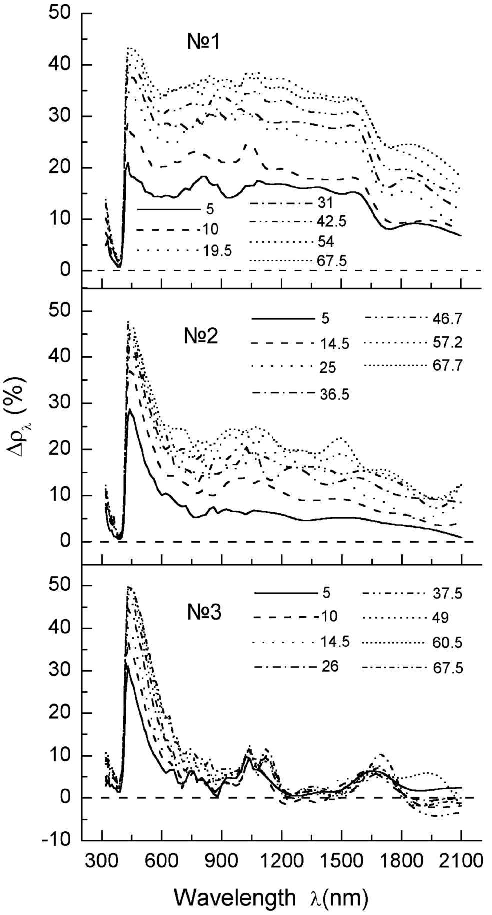

Fig. 4 Dependence of induced absorption spectra $\Delta\rho_\lambda$ of coatings based on TiO_2 pigment doped with PPB at concentrations of 0 (number 1), 5 (number 2), and 30 (number 3) mass % on EMR exposure time (hours) indicated by the numbers adjacent to the curves.

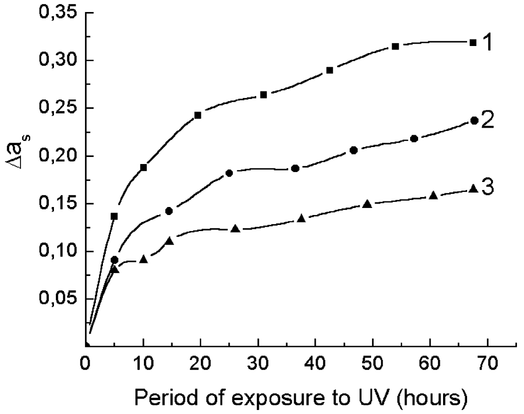

Fig. 5 Changes in total absorptance a_s vs EMR exposure time for coatings doped with PPB at concentrations of 0 (curve 1), 5 (curve 2), and 30 (curve 3) mass %.

Figure 6 shows the kinetics of changes in the reflection coefficient at 430, 740, and 1040 nm for coatings with the undoped pigment and the pigment doped with 30 mass % of PPB. From Fig. 6, it can be seen that the induced absorptance in the spectra of coatings with the doped pigment either is not increased at all or the rate of their increase is very low. At the same time, first, the induced absorptance in the maxima of these bands is much higher for coatings

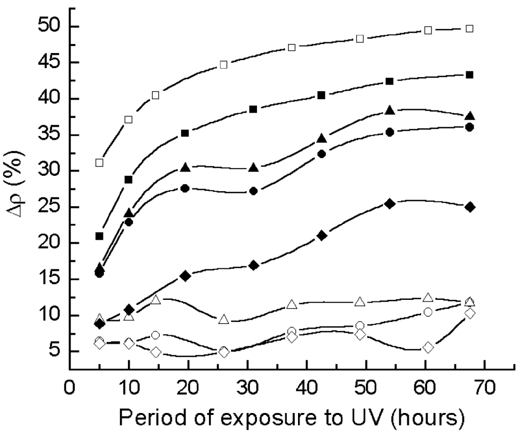

Fig. 6 Kinetic curve of changes in the induced absorptance $\Delta\rho$ in the maxima of the bands centered at ■ and □, 430; ● and ○, 740; ▲ and △, 1040; and ◆ and ◇, 1700 nm vs EMR exposure time for coatings based on undoped pigment (solid symbols) and pigment doped with 30 mass% of PPB (open symbols).

with the undoped pigment and, second, two stages are clearly pronounced in the kinetic curves. The first stage ends approximately after irradiation for 36 h.

Based on the results of investigations into the kinetics of coloring of ceramic and enamel coatings of other types and pigments,[23] we conclude that the two-stage degradation of the optical properties is the rule rather than the exception. Moreover, the first stage is determined by coloring at the expense of surface photo- or radiation-induced defects; it ends fast, but can introduce significant or even the main contribution to changes in the total absorptance. The second stage is determined by the photolysis or radiolysis of the pigment and binder lattice. Its influence on the total changes in the absorptance a_s depends in many respects on the surface state, namely, on the conditions of the electron excitation sink.

The kinetic curves of changes in the total absorptance were approximated by the dependence

$$\Delta a_s = \Delta a_{s1}[1 - \exp(-t/\tau_1)] + \Delta a_{s2}[1 - \exp(-t/\tau_2)] \tag{2}$$

where Δa_{s1} and Δa_{s2} are the maximum values of the absorptance a_s in the first and second stages, τ_1 and τ_2 are time constants of the processes of forming the absorption centers, and t is the exposure time, in hours.

The following mathematical dependences of changes in the absorptance on the EMR exposure time were obtained for the three types of coatings with different PPB concentrations in the TiO_2 pigment under conditions of minimization of the variance.

$C = 0$ mass %:

$$\Delta a_s = 0.190[1 - \exp(-t/37.93)] + 0.160[1 - \exp(-t/4.25)] \tag{3}$$

$C = 5$ mass %:

$$\Delta a_s = 0.134[1 - \exp(-t/7)] + 0.3[1 - \exp(-t/170)] \tag{4}$$

$C = 30$ mass %:

$$\Delta a_s = 0.076[1 - \exp(-t/2.19)] + 0.120[1 - \exp(-t/53.16)] \tag{5}$$

From Eqs.(3–5), it follows that the maximum values of a_s depend on the PPB concentration in the pigment: $a_{s\infty} = 0.35$ for $C = 0$ mass %, $a_{s\infty} = 0.434$ for $C = 5$ mass %, and $a_{s\infty} = 0.196$ for $C = 30$ mass %. With increase in the PPB concentration in the pigment, the maximum concentration of the absorption centers formed on exposure to EMR decreases. For a PPB concentration of 30 mass %, the concentration of the absorption centers decreases by a factor of 1.735 compared to the coating with the untreated pigment. In addition, the time of reaching $a_{s\infty}(t_\infty)$ for coatings with the undoped pigment are greater than that for coatings with the doped pigment: $t_\infty(C = 0$ mass %$) = 226$ h, $t_\infty(C = 5$ mass %$) = 1088$ h, and $t_\infty(C = 30$ mass %$) = 320$ h.

Conclusions

Our investigations have demonstrated that the doping of powders, TiO_2 pigments having the rutile structure, with PPB positively affects the photostability of coatings based on them. These coatings have high reflectance for the entire solar spectrum, which is only slightly smaller than the reflectance of the coatings with the undoped pigment, whereas the photostability to radiation imitating the solar spectrum is better. The improvement is observed at any oxidant concentrations up to 30 mass %, but this effect is most pronounced at a concentration of 15 mass %. It has been found that higher photostability of the total absorptance is caused by the reduction of the intensity of radiation absorption by electron color centers and charge carriers (polarons), the absorption bands of which lie in the range 700–2100 nm. Forecasting demonstrated that the degradation kinetics comprises the fast component determined by the surface processes and the slow component determined by the photolysis of the pigment and polymer binder lattice. The results of forecasting demonstrated that the change in the total absorptance of the coating with the pigment doped with 30 mass % of PPB is lower by a factor of 1.735 than that of the coating with the undoped pigment. Based on the results obtained, we conclude that oxidizing of TiO_2 powders with the rutile structure is a perspective technology of preparing reflecting coatings of space vehicles orbiting at low altitudes, and we hope that it can be used for coatings of space vehicles orbiting at high altitudes where these coatings are subjected to flows of electrons and protons with wide energy spectra in addition to electromagnetic solar radiation.

Based on the foregoing investigation, we can conclude the following:

1) The reflecting coatings based on TiO_2 powders having the rutile structure doped with PPB at different concentrations (5, 10, 15, and 30 mass %) have been prepared. They have high initial reflectance for the entire solar spectrum.

2) It has been found that the photostability of coatings based on doped TiO_2 powders is higher than that of coatings based on undoped powders.

3) It has been demonstrated that the doping reduces the concentration of color centers caused by defects of the anionic sublattice on exposure to radiation.

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