A COMPREHENSIVE STUDY OF NEW THERMAL CONTROL PAINTS

Anatoli V. Grigorevskiy[1], Larisa V. Kiseleva[1], Jacob Kleiman[2] and Zelina Iskanderova[2]

1 - JSC «Composite», 4, Pionerskaya str., Korolev, Moscow region, 141070, Russia, tel./fax +7 (495) 513-2020, E-мail: info@kompozit-mv.ru

2 - Integrity Testing Laboratory Inc., 80 Esna Park Drive, Units #7-9, Markham, ON, L3R 2R7 Canada, E-mail: jkleiman@itlinc.com

ABSTRACT

Thermal control paints ECOM-LG-2 (white) and ECOM-LG-B (black) were developed based on lithium liquid glass binders mixed with various pigments for application on external surfaces of spacecraft that operate in radiation and temperature hazardous orbits. The developed paints were applied to different space-qualified substrates and were tested in space flights and ground-based environmental simulators where they were exposed to the space environmental factors persisting at GEO and LEO orbits.

The results of the flight and ground-based testing had shown that the temperature range of ECOM-LG-2 and ECOM-LG usage can be extended up to 1000 K (727 C). The thermal optical characteristics of ECOM-LG-2 and ECOM-LG (αs and ε) were found to change very little with increasing temperature. Both paints, ECOM-LG-2 and ECOM-LG demonstrated excellent radiation resistance properties and could be used for the geostationary Earth orbit, highly elliptical and interplanetary orbits. The results from LEO flight-exposed and ground tested coatings show that they can be used successfully for spacecraft design in near-Earth orbit.

1.0 INTRODUCTION

With the thermal protection conditions in the interplanetary and near-solar space missions becoming more and more demanding, the requirements to the thermal control coatings (TCC) used for spacecraft's external shields are also increasing. Most of previous interplanetary missions have been to relatively cold parts of the Solar System. However, a number of missions like the BepiColombo by ESA-JAXA that will send a planetary probe close to the Sun and Mercury where it will endure temperatures in excess of 350°C or the Parker Solar Probe to be launched by NASA on a course that swings past Venus and loops around the sun before diving ever lower into the star's brutally hot atmosphere that will need to withstand temperatures outside the spacecraft that reach nearly 1,400°C are now either launched or going to be launched shortly. To cope with this, the spacecraft's external items, such as the antennas, solar arrays, Sun sensors and multilayer insulation, have temperature-resistant outer layers and protective coatings, need to be individually qualified to prove their capability.

Thermal control coatings, including paints, are used on outer surfaces of spacecraft to maintain their thermal balance in a specified temperature range. TCC's are characterized by the following characteristics [1]:

- Optical characteristics (solar absorptance - α_s and thermal emissivity - ε);
- Dielectric characteristics (volumetric resistance - ρv and surface resistance - ρs);
- Adhesive characteristics for different types of substrates;
- Operating temperature range (usually in a range from - 150 ^{O}C to + 150 ^{O}C);
- Outgassing characteristics, which should meet the requirements of relevant National and International standards (GOST R 50109, ASTM E595, ECSS-Q-70-02, QJ 1558);

2.0 EXPERIMENTAL TECHNIQUE

The irradiation of the TCC samples by protons, electrons and solar UV was performed on the UV-1/2 test facility at Kompozit shown in Fig. 1 [2].

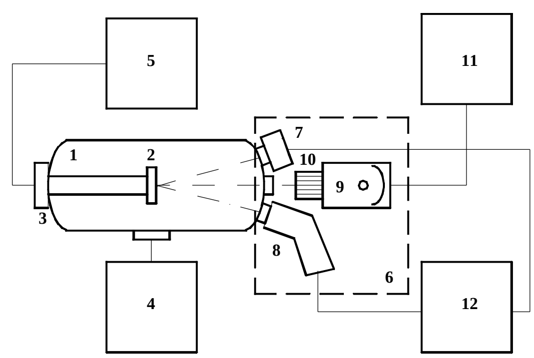

Fig.1. Schematic diagram of computerized test facility UV-1/2.
1 - vacuum chamber; 2 –sample holder; 3 – thermostat; 4 - pumping and vacuum monitoring system; 5 - measurement unit; 6 - space simulators; 7 - electron accelerator; 8 - proton accelerator; 9 - simulator of solar radiation (UV-source); 10 – focusing optics; 11 - solar simulator control box; 12 – accelerators control box.

The UV-1/2 test facility was designed to study the functional properties of materials and coatings under separate and combined irradiation by protons, electrons and UV in the comparatively low energy part of the GEO radiation spectra. The facility operates at a vacuum level as low as 10^{-5} Pa, with electrons and protons at energies up to 50 keV, solar electromagnetic radiation up to 10 SEE (solar exposure equivalent) and temperature T = ±150 ^{o}C. The reflectance spectra covering the wavelength range from 0.2 μm to 2.5 μm were collected before and after

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irradiation experiments on a «cary 500» spectrophotometer. absorptance as was calculated using the reflectance spectra and data on extraterrestrial solar spectral density - s(λ) [3].

The infrared Fourier spectrometer with «Mid-IR Integrate IR», based on integrated sphere with a gold diffuse-reflecting coating was used for the measurements of spectral emission factor in a wavelength range from 3 to 20 μm. The thermal optical properties, αs and ε were measured, using a solar irradiance reflectometer ИФ-1 and infra-red reflectometer ТРМ-3. Spectral emittance was measured at Vavilov State Optical Institute (Saint-Petersburg) for the temperature range up to 1000 K [4].

The enamels were prepared by mixing the selected liquid lithium glass binders with 4-6% of pigments in a porcelain ball mill. To obtain homogeneous composition, depending on pigmentation level, dispersivity and hardness of the pigment, milling times of up to 1-1.5 hrs were used. For the ECOM-LG-2 (white), a lithium liquid glass binder was used with a $BaSO_4$ pigment. For the EKOM-LG-B (black) a lithium glass binder was used with some selected metal oxides pigments.

The prepared enamels were applied on Ni, space grade Al-Mg alloy, CFRP (epoxy) composite and C-C substrates that were treated with sandpaper and degreased with acetone prior to the deposition of the enamels.

3.0 RESULTS AND DISCUSSION.

Thermal control paints ECOM-LG-2 (white) and ECOM-LG-B (black) were developed for applications on external surfaces of spacecraft that operate in radiation-hazardous orbits [5,6]. Figure 2 shows the dependence of solar absorptance of the developed ECOM-LG-2 (white) coating on the simultaneous irradiation by protons, electrons and solar UV. The horizontal axis represents values of proton irradiation fluence Fp. To reproduce the real situation in GEO environment, the electron irradiation fluenct in these experiments, Fe, was five times higher than the proton fluence, i.e. Fe=5·Fp. The equivalent of solar irradiance was 100 ESH.

For comparison, on the same graph are shown the data for a few other white TCC' like the ECOM-1 coating, consisting of a special acrylic binder with ZnO_{Ga} pigment, the TP-15 coating, based on a potassium liquid glass binder with ZnO pigment, and an optical solar reflector (OSR), based on silica glass with back-sprayed Al .

Figure 3 presents similar data for the black ECOM-LG-B coating and a few other black TCC's that include the Z-306 coating, based on a polyurethane binder with soot and silica [7], the EKOM-2 paint, based on an acrylic binder with thermally-resistant pigments, the PUI Black Conductive paint [8] and the PUI Black Polyurethane paint [8];

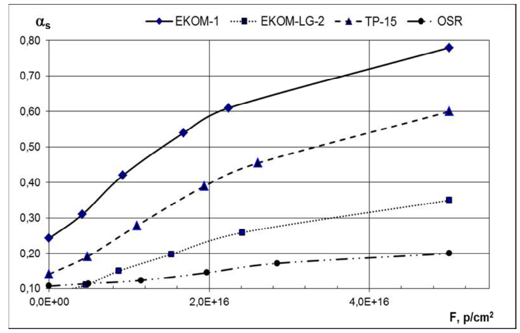

Fig. 2. Dependence of solar absorptance of ECOM-LG-2 on Fp at simultaneous irradiation by protons + electrons + solar UV.

As can be seen from Figs. 2 and 3, the inorganic ECOM-LG-2 and ECOM-LG-B, based on the lithium liquid glass, demonstrated very good radiation resistance properties and can be used in radiation-hazardous orbits.

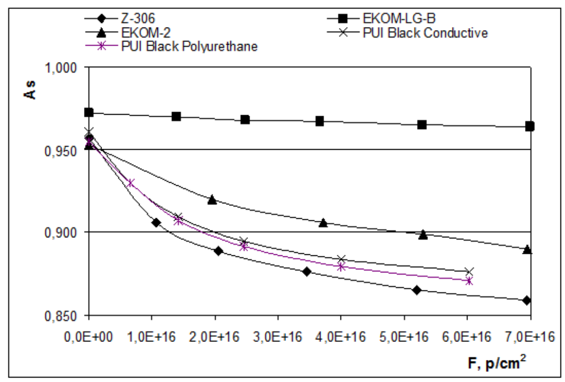

Fig.3. Dependence of solar absorptance of ECOM-LG-B on Fp at simultaneous irradiation by protons + electrons + solar UV.

Thermal cycling tests of ECOM-LG-2 and ECOM-LG-B in the temperature range of ± 150 °C showed that their major characteristics including adhesive, electrophysical and optical properties did not change significantly after 100 cycles.

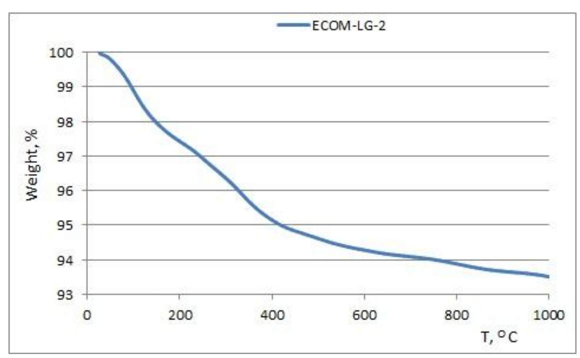

Fig.4. Thermogravimetric analysis of ECOM-LG-2

The resistance of the TCCs' to higher temperatures was initially evaluated by performing thermogravimetric analysis of powders of dried and crushed lithium liquid glass and then – on ECOM-LG-

2 and ECOM-LG-B enamels. Figures 4, 5, and 6 show thermogravimetric analysis results for ECOM-LG-2 and ECOM-LG-B for up to T = 1000 ^{O}C and lithium glass for up to T = 1500 ^{O}C.

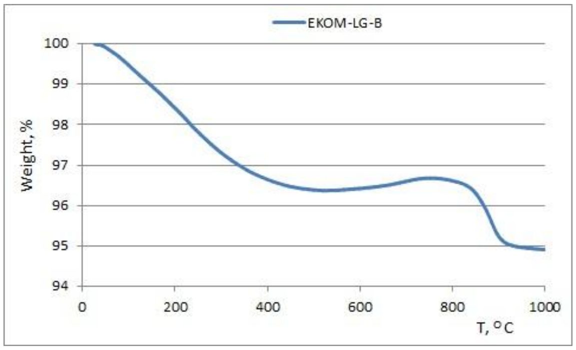

Fig.5. Thermogravimetric analysis of ECOM-LG-B

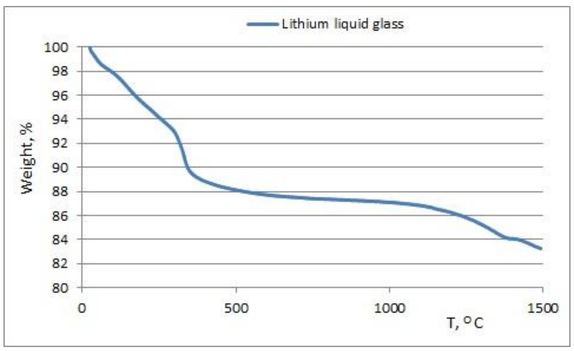

Fig.6. Thermogravimetric analysis of lithium liquid glass-based binder

Nickel and aluminum AMg6 alloy substrates were painted with ECOM-LG-2 and ECOM-LG-B and used in a series of experiments to evaluate the influence of temperature on their thermal optical properties. The AMg6 alloy samples with ECOM-LG-2 and ECOM-LG-B coatings were annealed at different temperatures (110, 300, 500 °C) in air for 1 hour, with α_s and ε measured afterwards at room temperature. Tables 1 and 2 show the test results.

Table 1 Solar absorptance of TCC samples after annealing in air for 1 hour

T of annealing, °C	20	110	300	500
	Solar absorptance, α_s			
ECOM-LG-2	0,08	0,08	0,08	0,08
ECOM-LG-B	0,96	0,96	0,96	0,96

Table 2 Total emittance of TCC samples after annealing in air for 1 hour

T of annealing, °C	20	110	300	500
	Emittance, ε			
ECOM-LG-2	0.955	0.950	0.935	0.925
ECOM-LG-B	0.950	0,945	0,950	0.945

Analyzing the data in Tables 1 and 2, it can be concluded that thermal optical properties of ECOM-LG-2 and ECOM-LG-B change very little after annealing up to 500 ^{O}C.

The samples on nickel substrates were used directly for emittance measurements at preset temperatures without the annealing step.

Table 3 shows the results of emittance measurements of these samples for both the ECOM-LG-2 and ECOM-LG-B painted coupons. As can be seen from Table 3, the emittance of ECOM-LG-2 paint decreased by almost 15% with increasing temperature. The emittance of ECOM-LG-B, however, remained relatively unchanged.

Table 3 Total emittance measurements of ECOM-LG-2 and ECOM-LG-B samples at various temperatures.

	Emittance, ε				
ECOM-LG-2	0.95	0.809	0.810	0.815	0.817
ECOM-LG-B	0.92	-	0.903	0.908	0.918
T of annealing, ^{O}C	20	300	500	600	700

Figure 7 presents the dependence of spectral emittance of EKOM-LG-2 paint, in the range 2 to 25 μm, at three different temperatures, i.e. at 293K (~20 C), at 577 K(~304 C) and 974 K (~700 C). Figure 8 presents similar data for the EKOM-LG-B paint at 293K (~20 C), 801K (~528 C) and 974 K (~701 C).

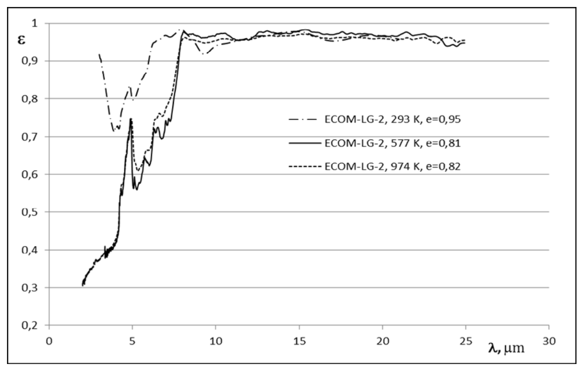

Fig.7. Spectral emittance of EKOM-LG-2 (white coating) measured at three temperatures.

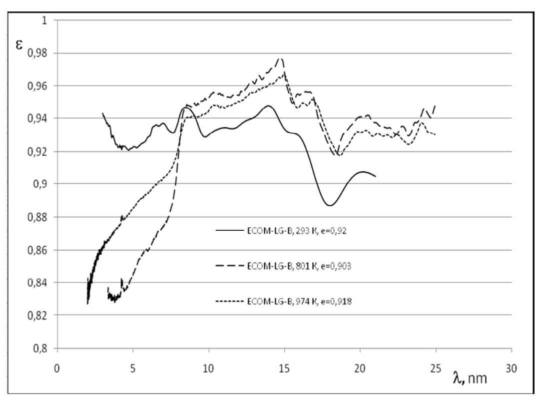

Fig .8. Spectral emittance of EKOM-LG-B (black coating) measured at three temperatures.

4.0 FLIGHT EXPERIMENT

A full-scale LEO space flight experiment was performed to evaluate the irradiation resistance of

ECOM-LG-2 and ECOM-LG-B to LEO space environment conditions [9]. A removable cassette with the enamel-based TCC samples was exposed on the outer surface of MIM2 RS ISS module for almost 1.5 years. The total solar irradiation time of the samples was 90 ESH (equivalent of solar irradiance). The total time was calculated taking into account the variations in solar irradiation incidence angle to the surface normal of the active area.

The fluence (integrated flux) of atomic oxygen during the operating time was calculated at 2.54×10^{19} atoms/cm^2 taking into account the orbital altitude, the solar activity index and the variations in the flux incidence angle to the surface normal of the active area.

Evaluation of the atomic oxygen fluence, based on erosion results from exposed polyimide film that was also mounted on the cassette gave a value of $2.5\text{x}10^{19}$ atoms/см^2, showing good coincidence with the calculation results.

A number of samples (marked in Table 4 as ill №XX) were mounted on the back side of the cassette and were shielded from the Sun exposure. The atomic oxygen fluence for the back side samples was estimated at $5\text{x}10^{18}$ atoms/sm^2, based on the measurement of the AO flux from the erosion of exposed polyimide film. The sample locations on the SKK cassette is shown in Table 4.

Table 4. Location of samples in the SKK cassette

Paint	Sample №	Sample size	Sample type	Location on the SKK cassette
EKOM-LG-2	№181 №185	30x30mm	flight (ЛО) witness-(OC)	front side -
	ill №14 ill№12	∅45	flight (ЛО) witness- (OC)	back side -
EKOM-LG-B	№142 №141	30x30mm	flight (ЛО) witness-(OC)	front side -
	ill№52 ill№54	∅45	flight (ЛО) witness-(OC)	back side -

4.1 Total reflectance spectra in the wavelength range from 0.2 to 20 μm

Reflection spectra measurements in the wavelength range from 0.2 to 20 μm were performed using two spectral measurement tools: "Cary 500" spectrophotometer, in a spectral range from 0.20 to 2.5 μm, and infra-red Fourier spectrometer “Frontier” from 2.0 to 20 μm. The systems were equipped with integrated spheres for acquiring the total reflected signal.

Figures 9 and 10 show the results of total reflectance measurements conducted in the range from 0.2 to 2.5μm for the ECOM-LG-2 and the ECOM-LG-B samples, respectively. The calibration of the "Cary 500" instrument was done using a witness-sample made of Spectralon ("Spectralon I.D": SRS-99-020).

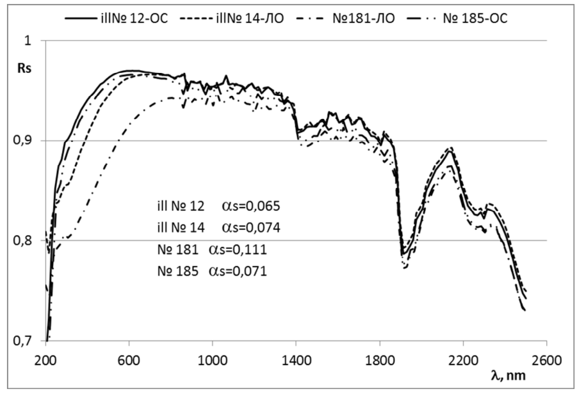

Fig.9. Total reflectance spectra of ECOM-LG-2 samples in the wavelength range from 0.2 to 2.5 μm (before (witness) and after flight experiments)

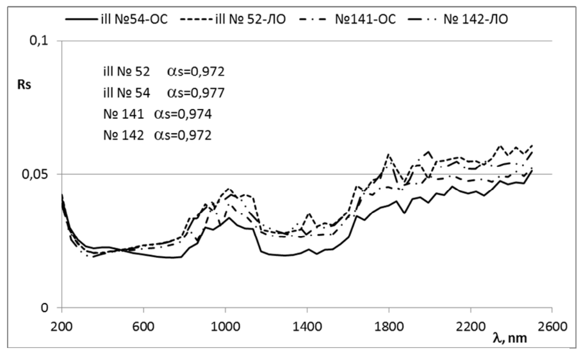

Fig.10. Total reflectance spectra of ECOM-LG-B samples in the wavelength range from 0.2 to 2.5 μm (before (witness) and after flight experiments)

Analyzing the spectra in Fig. 9, it is clear that the reflectance values of the two white flight TCC samples located on the front side of the cassette slightly decreased in the wavelength range 0.25 -1.2 μm, with the rest of the spectra remaining unchanged. The reflectance spectra for white witness TCC samples remained unchanged.

Analyzing the spectra in Fig. 10, it can be suggested that reflectance curves of the black TCC samples underwent an insignificant change that is within the limits of the measurement error.

The observed changes may be due either to the UV- solar irradiation influence or to a buildup of a contamination layer.

4.1.1 Reflectance spectra in the wavelength range from 3 to 20 μm

Reflectance spectra measurements in the wavelength range from 3 to 20 μm were performed on a "Frontier" infra-red Fourier spectrometer. The spectrometer was equipped with an integrated sphere attachment "Mid-IR IntegratIR" with a gold diffusely-reflecting coating. Figures 11 and 12 present the results of these reflectance measurements.

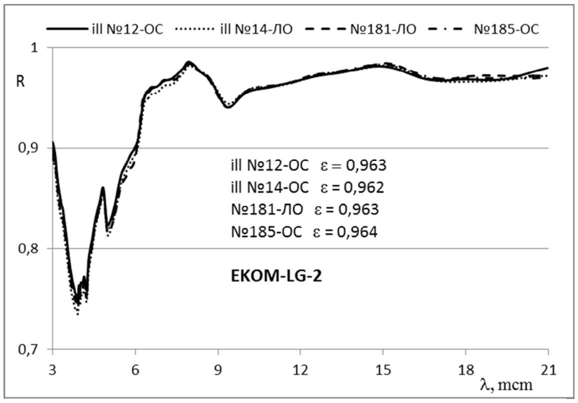

Fig. 11. Reflectance spectra of ECOM-LG-2 samples in the wavelength range from 3 to 20 µm (before (witness) and after full-scale exposure)

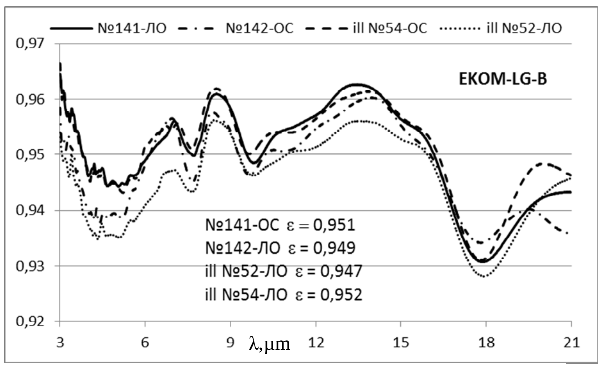

Fig.12. Reflection spectra of ECOM-LG-B samples in the wavelength range from 3 to 20 µm (before (witness) and after full-scale exposure)

4.1.2 Comparative Evaluation of α_S and ε Data Obtained on Different Spectrometers

The data for each of the thermal optical properties, α_S and ε, of the white and the black enamels were measured using the following methods:

Total Absorptance

- The total reflectance R(λ) was measured, as described above, in the wavelength range from 0.2 µm to 2.5 µm using the "Cary 500" spectrophotometer with an integrating sphere and the obtained values were corrected to take into account the spectral distribution of extraterrestrial solar radiation curve;
- A relative measurement method using a portable solar reflectometer IF-1. This method also uses reference samples for comparison and will be described in details elsewhere.

Solar Emittance

- The solar emittance (ε) measurement was performed on an infrared spectrometer by measuring the spectral reflectance R(λ) in the wavelength range from 3 µm to 20 µm and converting it to the emittance values by subtracting from 1 (assuming that the absorptance in the materials is negligible). This method also uses spectral distribution of black-body radiation for preset temperature;
- relative measurement method using a portable infra-red solar reflectometer TRM3. This method also uses reference samples for comparison and will be described in details elsewhere.

Tables 5 and 6 present the summary of this comparative evaluation study. A satisfactory coincidence in the results obtained on the stationary and portable instruments can be suggested.

Table 5. Summary of a comparative evaluation of solar absorbance a_s measured by two methods

Sample name	Sample №	a_s (CARY 500)	a_s (IF-1)
ECOM-LG-2	№181- ЛО	0,111	0,087
	№185- ОС	0,071	0,071
	ill №14- ЛО	0,074	0,069
	ill №12- ОС	0,065	0,065
ECOM-LG-B	№142- ЛО	0,972	0,973
	№141- ОС	0,974	0,974
	ill №52- ЛО	0,972	0,971
	ill №54- ОС	0,977	0,977

ЛО - flight sample, ОС - witness-sample

Table 6. Summary of a comparative evaluation of emittance **ε** measured by two methods

Sample name	Sample №	e (IR-Fourier spectrometer	e (TRM-3)
ECOM-LG-2	№181- ЛО	0,963	0,930
	№185- ОС	0,964	0,935
	ill №14- ЛО	0,962	0,950
	ill №12- ОС	0,963	0,950
ECOM-LG-B	№142- ЛО	0,949	0,955
	№141- ОС	0,951	0,965
	ill №52- ЛО	0,947	0,960
	ill №54- ОС	0,952	0,965

ЛО - flight sample, ОС - witness-sample

In summary, the analysis of all thermal optical data indicates that there was a good repeatability between calculated values and measured values of αs and ε. The flight tests show that solar absorbance αs of white TCCs (mounted on the front side of the cassette) increased, but solar absorbance αs of black TCCs decreased under the influence of solar UV-radiation. However emittance ε of the mentioned samples stayed practically unchanged. Thermo-optical characteristics of the TCC samples mounted on the back side of the cassette did not change.

4.2 Dielectric properties

Volume resistivity measurements of the flown samples were performed according to the Russian GOST Standard #6433.2-71 by using a voltmeter-ampere meter method [10].

The volume resistivity measurements were performed at room temperature (22°C) and at a relative humidity of 82%. Table 7 contains the measurement results.

Table 7. Summary of volume resistivity measurements.

Material name	Sample number	Resistivity, MΩ	Coating thickness h, µm	Volume resistivity ρ_v, MΩ·m
ECOM-LG-2	№181 ЛО	235	149	120
	ill №14 ЛО	290	157	145
	№185 ОС	207	165	98
ECOM-LG-B	№141 **ОС**	200	171	92
	№142 ЛО	220	158	109
	ill №52 ЛО	200	204	77

ЛО - flight sample, ОС - witness-sample

5.0 GROUND-BASED TESTING OF EKOM-LG-2 IN AN OXYGEN-PLASMA FACILITY

An additional study has been conducted to evaluate the durability and functional performance of the inorganic white charge-dissipative space paint EKOM-LG-2 in an O-plasma facility. Oxygen plasma sources are considered as a very useful ground-based testing tool for "screening" materials in imitated LEO environment. It had been shown that environmental conditions generated in O-plasma systems, i.e. the presence of a variety of oxygen species, including atomic oxygen, are quite severe and can be used for testing trials.

In our studies we used successfully both O-plasma facility (screening) and fast atomic oxygen beam sources as LEO simulation environments. For instance, this approach was used in a Project of comparative ground-based testing and performance evaluation of 67 space flown materials from US, Russia and Europe [11].

All oxygen plasma exposure tests have been conducted in a low-temperature, inductively coupled radio frequency plasma facility. The facility operated at 13.56 MHz and settings that were found to adequately reproduce the LEO AO environment in ground-based simulation, i.e. RF Power ~ 200 watts, oxygen pressure ~ 100 mTorr ± 5%, oxygen input ~ 100 sccm ± 5%. Minimum averaged equivalent AO fluence, used in this study, was ~ 1.9 x 10^{20} at/cm^{-2}, that has been achieved in a ~ 6 hrs plasma exposure.

The tested samples included painted aerospace grade AlMg6 substrate coupons and CFR plastic (epoxy) composite coupons. Kapton100HN and Kapton500HN samples were used in all experiments as witnesses. The measurement of surface resistivity (SR) of all samples before and after testing was done, using a contact method and a specially designed unit probe, described in [12]. All measured SR values of original samples stayed in the range (1.5-2) x 10^8 Ohm/sq. The test and Kapton witness samples were placed on a glass holder and positioned in the middle of the 8 L plasma facility chamber. To avoid plasma undercutting, the edges of all witness samples were covered tightly by glass slides. The chamber was pumped down and left under vacuum for ~24-48 hours to outgas the samples. Mass change of the samples was measured with an electronic microbalance with a 10 µg resolution.

The mass change due to outgassing was found to be very small, almost negligible, for the paint samples on the metal substrates when compared with both KaptonHN witnesses. However, the outgassing from the pained composite samples was ~ 7-9 times higher then from the painted metal coupons. We consider that this result is due to the outgassing from the composite itself, mainly through the back and unpainted edges of the composite coupons.

The O-plasma exposure has been performed in 2 stages. Firstly, the samples were exposed to an equivalent AO fluence of 2x10^{20}cm^{-2}, with SR and mass loss measurements conducted at the end of the exposure. After that the samples were additionally exposed to O-plasma, up to a total equivalent AO fluence of ~ 1.2x10^{21}cm^{-2}. It is important to mention, that the SR dropped after the first AO dose, becoming ~10-15 times lower then of the original samples, reaching values of ~ (7-9) x 10^7 Ohm/sq. After the full exposure dose, the SR dropped additionally, reaching values in the range of ~5 MOhm/sq for the paint on aluminum alloy and ~ 10MOhm/sq for the paint on composite substrates. The mechanism of this phenomenon is presently not clear yet. However, in our opinion, this is a strong indication, that this paint will keep the SR in a charge dissipative range, if to be used as an external space coating on both metal and composite substrate materials during long-term LEO missions.

The weight of the samples after the first stage exposure dropped slightly, but the drop was still smaller then for KaptonHN witness coupons (both calculated as an equal averaged mass loss from a surface unit), and then the weight of painted samples remained practically unchanged, as measured after the second exposure stage.

Thermal optical characteristics have been measured as described above, and the results are presented in Fig.14 and Table 8. As can be seen from Fig.14 and Table 8, the solar absorptance, as well as the thermal emittance, remained practically unchanged after the O-plasma testing. The conducted testing and the obtained results indicate that the white charge dissipative ECOM-LG-2 space paint, developed for GEO applications, can be used successfully also for LEO long-term missions.

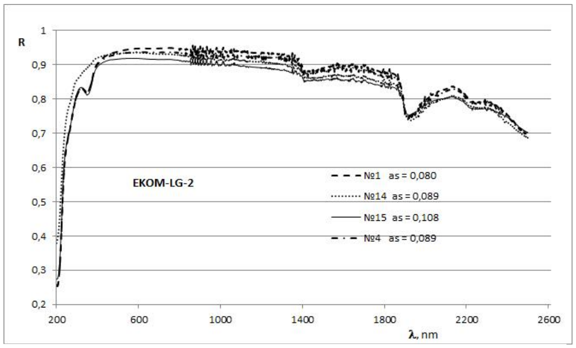

Fig. 14. Reflectance spectra of ECOM-LG-2 samples (before and after O-plasma experiments)

№ 14 – original sample, №№ 1, 4, 15 - samples after O-plasma exposure up to AO fluence ~ $1.2x10^{21}cm^{-2}$.

Table 8. Measurement results of emittance of TCC EKOM-LG-2 before and after O-plasma exposure

Material name	Sample number	ε
ECOM-LG-2	№1	0.950
	№4	0.955
	№15	0.950
	№14	0.950

№ 14 – original sample, №№ 1, 4, 15 - samples after O-plasma experiments

6.0 CONCLUSIONS

As a result of the ground and flight tests of ECOM-LG-2 and ECOM-LG-B inorganic thermal control paints the following conclusions could be made:

- The temperature range of ECOM-LG-2 and ECOM-LG can be extended up to 1000 K (727 C);
- The thermal optical characteristics of ECOM-LG-2 and ECOM-LG (α_s and ε) change only slightly with increasing temperature;
- Both paints, ECOM-LG-2 and ECOM-LG, have good radiation resistance properties and could be used for the geostationary Earth orbit, highly elliptical and interplanetary orbits;
- The results on LEO flight-exposed and ground–based tested coatings did show that they can be used successfully for long-term in Low Earth orbit.
- The experimental data indicate how the spectroscopic and dielectric properties of the material samples have changed during the ground-based and flight tests. The test results contain information which can help to understand physical and chemical processes of the TCCs under various space environment factors and high temperature influence. These testing results also enable to find the ways to extent the operating temperature range of the TCCs up to 1500 K.

7.0 REFERENCES

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