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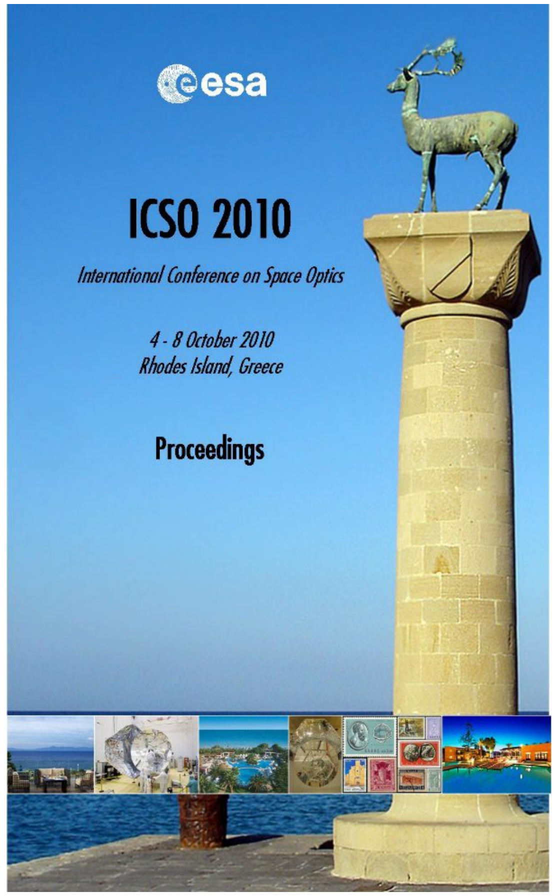

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ANALYSIS OF OPTICAL PROPERTIES BEHAVIOUR OF CLEARCERAM, FUSED SILICA AND CAF_2 GLASSES EXPOSED TO SIMULATED SPACE CONDITIONS

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INTRODUCTION

Optical instrumentation on-board satellites suffer degradation due to the hostile conditions of space environment. Space conditions produce instrumentation performances changes causing a decrease or a cancellation of their features. Particularly, space environment conditions have a significant influence on the optical properties of glasses which are part of space optical systems.

Space environment characteristics which effects on the optical system have to be taken into account are: outgassing, volatile components, gas or water vapor which form part of the spacecraft materials, vacuum, microgravity, micrometeorites, space debris, thermal, mechanical and radiation environment and effects of the high atmosphere [1].

This work is focused on analyzing temperature variations and ultraviolet (UV) and gamma radiation effects on the optical properties of several glasses used on space applications.

Thermal environment is composed of radiation from the Sun, the albedo and the Earth radiation and the radiation from the spacecraft to deep space. Flux and influence of temperature on satellite materials depend on factors as the period of year or the position of them on the space system. Taking into account that the transfer mechanisms of heat are limited by the conduction and the radiation, high gradients of temperature are obtained in system elements which can cause changes of their optical properties, birefringence… Also, these thermal cycles can introduce mechanical loads into material structure due to the expansion and the contraction of the material leading to mechanical performances degradation [2].

However, it is the radiation environment the main cause of damage on optical properties of materials used on space instrumentation. This environment consists of a wide range of energetic particles between keV and MeV which are trapped by the geomagnetic field or are flux of particles that cross the Earth environment from the external of the Solar System [3].

The damage produced by the radiation environment on the optical materials can be classified in two types: ionizing or non-ionizing. This damage may produce continual or accumulative (dose) alterations on the optical material performances, or may produce alterations which not remain along the time (transitory effects). The effects of the radiation on optical materials can be summarized on changes of optical transmission and refractive index, variation of density and superficial degradation [4-6].

Two non-invasive and non-destructive techniques such as Optical Spectrum Analyzer and Spectroscopic Ellipsometry [7] have been used to characterize optically the three kinds of studied glasses, CaF_2, Fused Silica and Clearceram.

The study of the temperature and radiation effects on the glasses optical properties showed that the gamma radiation is the principal responsible of glasses optical degradation. The optical properties of the Clearceram glass have been affected by the gamma irradiation due to the absorption bands induced by the radiation in the visible spectral range (color centers). Therefore, an analysis about the behavior of these color centers with the gamma radiation total dose and with the time after the irradiation has been carried out in the same way that it is performed in [8].

EXPERIMENTAL DETAILS

A. Samples specification

Three different glasses from SCHOTT Lithotec, SICO and OHARA have been analyzed under space conditions simulation: a CaF_2 (VIS) glass whose refractive index is 1.434 at 589nm; a Fused Silica glass whose refractive index is 1.458 at 589nm; and a ceramic glass, Clearceram, whose refractive index is 1.546 at the same wavelength. All of them are not hardness glasses, i.e. no cerium doped. The samples are polished as parallel-plane disks of 20mm diameter and 5mm thick.

The number of studied samples has been 7 per glass divided as follows: one of them was used as a blank, another one was exposed to UV radiation, one more was subjected to thermal cycling and the four remaining samples were exposed to four different total dose of gamma radiation.

B. Tests

Simulation of the thermal and vacuum environment was performed at LINES-INTA facilities. This laboratory has a 10,000 Class clean room where is located the vacuum chamber in which **thermal cycling test** was carried out. The vacuum chamber includes a base plate and a shroud, independently controlled to reach the thermal range from -90º C to +100º C. It operates through a pump to achieve vacuum levels of 10^{-2} mbars and through a cryogenic unit achieves a high vacuum level of 10^{-6} mbars.

The conditions of the thermal test consisted of 5 temperature cycles between -60ºC and +100ºC (starting point +20ºC) with a slope of 1ºC/min. Glasses were subjected to a vacuum level of 10^{-6}mbars.

UV radiation test was performed in SPASOLAB facilities at the Instituto Nacional de Técnica Aeroespacial (INTA). SPASOLAB is an European Space Agency (ESA) certified laboratory in photovoltaic solar cells for space applications. The UV irradiation was carried out in a vacuum chamber with an AM0 solar spectrum simulator. The test was performed with an acceleration factor that relates real time to equivalent exposure time at AM0 solar spectrum. The spectrum of the used lamps extends from the UV to visible (VIS) and infrared (IR). Test conditions followed *the Photovoltaic Assemblies and Components Standard* (ECSS-E-20-08) and were: 148.25hours of illumination total time, an acceleration factor of 7.11, 1054.1 ESH (Equivalent Solar Hours) of equivalent exposure time, the baseplate temperature was 19.5ºC and the chamber vacuum level was 10^{-6}-10^{-5} mbar. The light uniformity achieved in the exposure of the sample was better than 85%.

Gamma irradiation test was performed at the Centro de Investigaciones Energéticas Medioambientales y Tecnológicas (CIEMAT) facilities in Spain. γ-rays (gamma rays) come from Co^{60} sources placed in a water pool at room conditions. They are distributed at the bottom of the pool in such a way that the radiation uniformity over the glasses was greater than 90%. This test was divided in four steps of γ-radiation total dose with the same dose rate of 11krad/h. These steps simulated different radiation environments typical of space missions. The four steps of radiation were 50krad, 200krad, 800krad and 2Mrad. A different sample of every kind of glass was exposed to each step of radiation total dose, i.e. the samples which were irradiated in each step were different.

C. Optical characterization

Analysis of the glasses optical properties was performed before and after each test. Changes of the optical performances induced by the radiation were obtained by comparison with the blank data.

Transmission measurements were carried out using an Optical Spectrum Analyzer OSA 320 from Instrument Systems in a wavelength range of 350 nm – 850 nm at normal incidence.

The spectroscopic ellipsometry is a very sensitive and accurate optical measurement technique that uses polarized light to characterize thin films, surfaces and materials microstructure. The ellipsometric technique measures the change in polarization state of light reflected from a sample. The measured values are expressed as the ellipsometric angles psi (Ψ) and delta (Δ). These ellipsometric parameters are related to the ratio of Fresnel reflection coefficients r_p, r_s for p- and s- polarized light, respectively. Ellipsometry is used to measure thin film thickness and optical constants -i.e. complex refractive index- but it is also utilized to determine surface and interfacial roughness and optical anisotropy [7]. Two different ellipsometers were used for spectroscopic ellipsometric measurements. Glasses optical properties measured before and after the thermal cycling test were carried out using a Rotating-Polarizer Spectroscopic Ellipsometer ES-4G from SOPRA. The spectral range analyzed was 300-800nm and the incidence angle was 75º. A variable angle spectroscopic ellipsometer (VASE) from J. A. Wollam Co. Inc. was the instrument used for measuring the ellipsometric parameters before and after UV and gamma radiation tests. The measurements were carried out at two different incident angles, 60º and 70º, analyzing a spectral range from 200 to 1000nm.

RESULTS AND DISCUSSION

A. Analysis of the optical properties of the glasses exposed to thermal cycling

Transmission and ellipsometric measurements performed after thermal cycling have slightly changed with regard to the measurements carried out before test for all the studied glasses. These variations are inside experimental uncertainty. Fig. 1. displays the comparison between the transmission curves of the Fused Silica glass before and after thermal cycling.

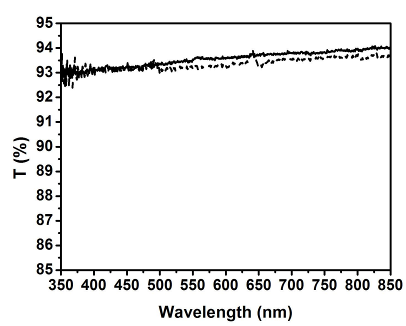

Fig. 1. Transmission measurements before (solid lines) and after (dash lines) thermal cycling for Fused Silica glass.

In addition, it had been performed interferometric measurements in order to determine surface deformations or microcracks of the glasses. However, the interferograms measured before and after thermal test were similar, so wavefront deformation was not relevant.
It is concluded that the optical and mechanical properties of the analyzed glasses are not influenced by temperature variations.

B. Analysis of the optical properties of the glasses exposed to UV radiation

Variations of ellipsometric parameters and transmission spectrum between the measurements performed before and after UV radiation test are small and inside experimental uncertainty, so these changes can be considered negligible. Fig.2. shows the ellipsometric parameters of the CaF_2 glass measured before and after UV radiation exposure.
The optical constants determined through ellipsometric and transmission data evidenced that the optical performances of the glasses are not affected by UV radiation.

C. Analysis of the optical properties of the glasses exposed to γ-radiation

After exposure of glasses to gamma irradiation, only one of them, the Clearceram, showed a darkening which was increasing as function of the radiation total dose. This coloration is due to the new electronic configurations created by the interaction of the ionizing radiation with the material causing visible light absorption. Formation of these color centers depends on the composition and structure of the irradiated material [5], [8], [9].
Variations of the transmission and ellipsometric measurements confirmed that the optical constants of the Clearceram glass have been changed after the gamma irradiation test.
Transmittance of CaF_2 and Fused Silica glasses remains constant after γ-rays while the Clearceram transmission decreases with the radiation total dose achieving a null transmission at the UV spectral range for radiation total dose higher than 800krad. The ellipsometric parameters showed that the variation of the real part of the refractive index after gamma irradiation is negligible while the extinction coefficient increases with the radiation total dose (Fig. 3.).
Changes of the Clearceram transmission and the complex part of the refractive index are due to the color centers generated by γ-radiation. This induced absorption is expressed as function of the complex part of the dielectric coefficient, ε_2, which is connected with the absorption coefficient, α [10]. Complex part of the dielectric coefficient has been fitted through Gaussian shaped absorption peaks. Total absorption is represented by the color centers generated by the γ-radiation and absorption which is in the glass before irradiation (Fig. 4.). Therefore, the total absorption can be described as a sum of individual absorption peaks with an energy, E (eV), a width at half maximum high, Br (eV), and an amplitude, A. The peak amplitude increases with the γ-radiation total dose while the energy and width of the peak are independent of radiation dose (Table 1).

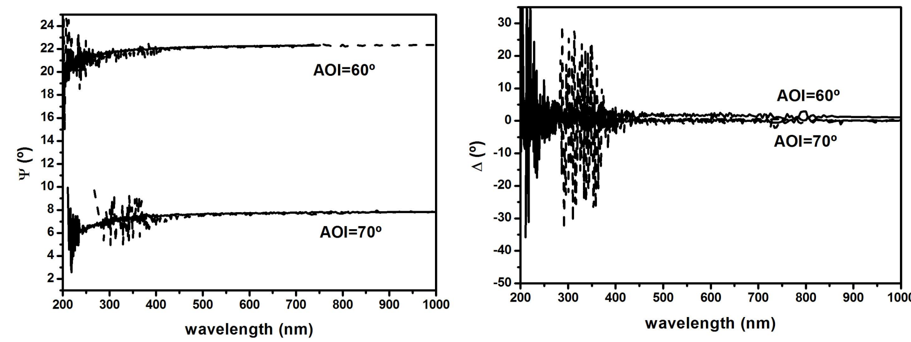

Fig. 2. Ellipsometric parameters at two incidence angles for Ca F_2 glass: before UV radiation (solid lines); and after UV radiation (dash lines).

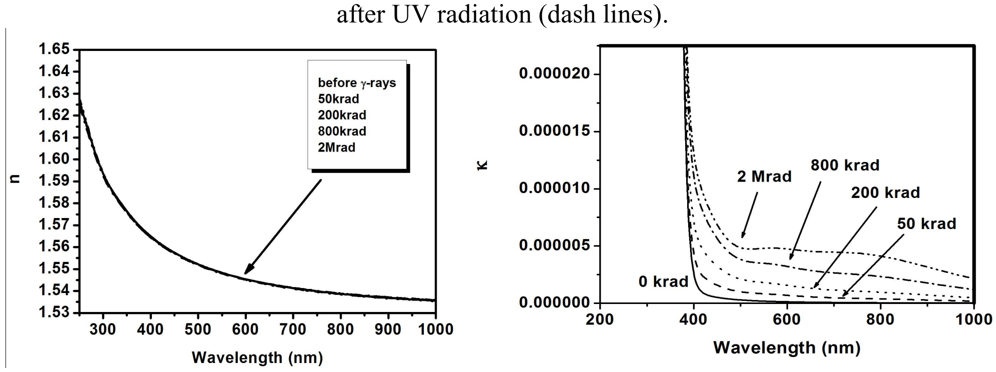

Fig.3. Real and complex part of the refractive index for Clearceram glass before and after gamma radiation.

The growth of the absorption peaks amplitudes as function of γ-radiation total dose (Fig. 5) has been fitted through a first order exponential function described in [8].

Transmission and extinction coefficient of a Clearceram sample exposed to 800krad of gamma radiation have been analyzed along several months after irradiation in order to evaluate the behavior of the glass optical properties with time. The analysis showed that the amplitude of the induced absorption peaks decreases (Fig. 6); that means that the color centers generated by radiation are meta-stable. This natural relaxation process of the absorption peaks amplitudes is perfectly fitted by a function with two exponential terms which represents two different relaxation rates as it is showed in [8].

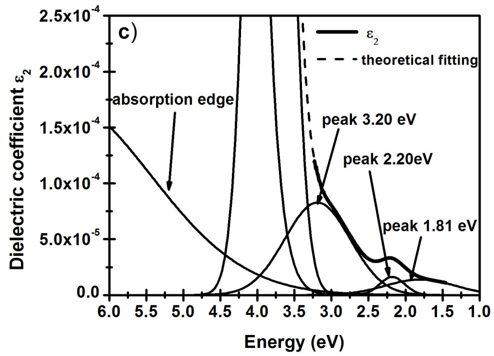

Fig. 4. Decomposition of ε_2 into absorption bands after 800Krad of γ-radiation for Clearceram glass. Radiation generated three absorption peaks which energies are centered in 3.20eV, 2.20eV, 1.81eV.

Table 1 shows the amplitude (A), energy (E) and width (Br) of the Gaussian absorption peaks induced by gamma radiation.

Clear Ceram	Peak 1	Peak 2	Peak 3
E (eV)	3.20 ± 0.04	2.20 ± 0.04	1.81 ± 0.20
Br (eV)	1.08 ± 0.06	0.42 ± 0.03	1.15 ± 0.04
After 50krad A	$(1.13\pm0.01)\cdot10^{-5}$	$(1.7\pm0.2)\cdot10^{-6}$	$(1.9\pm0.2)\cdot10^{-6}$
After 200krad A	$(3.70\pm0.01)\cdot10^{-5}$	$(6.8\pm0.1)\cdot10^{-6}$	$(5.70\pm0.06)\cdot10^{-6}$
After 800krad A	$(8.28\pm0.02)\cdot10^{-5}$	$(1.63\pm0.03)\cdot10^{-5}$	$(1.40\pm0.02)\cdot10^{-5}$
After 2Mrad A	$(9.75\pm0.02)\cdot10^{-5}$	$(2.01\pm0.02)\cdot10^{-5}$	$(2.15\pm0.01)\cdot10^{-5}$

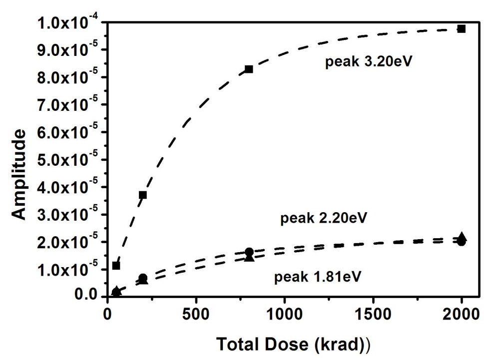

Fig. 5. Absorption peaks amplitude vs. γ-radiation total dose (symbols). Dash lines are the fitting with the first order exponential function.

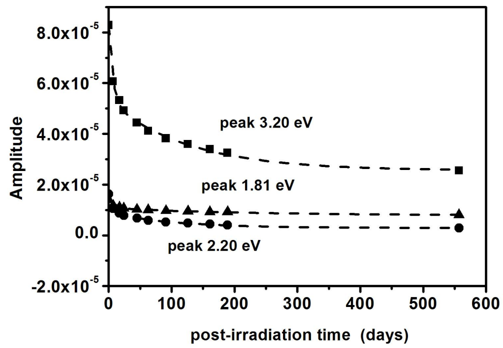

Fig. 6. Temporal evolution of the absorption peaks amplitudes generated after 800krad of gamma radiation total dose. Dash lines are the fitting with a compound exponential function with two exponential terms.

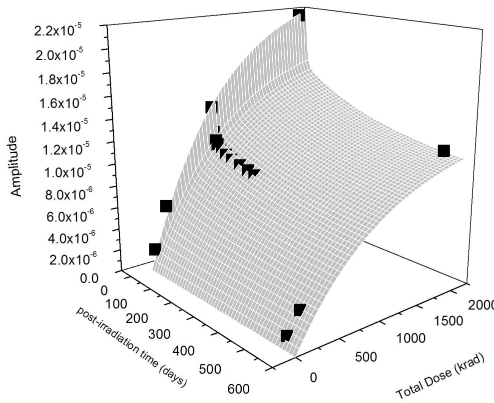

Fig. 7. Fitting of the Clearceram absorption peak of 1.81eV of energy (black point) using the two-variable function (mesh).

The behavior of the absorption peaks induced by γ-radiation on Clearceram as a function of radiation total dose and post-irradiation time has been analyzed. The model proposed to predict the optical properties changes of the glass consists of a two-variable function (1) determined from the studies above expounded, amplitude vs. radiation dose and amplitude vs. time. Also it is assumed that the natural relaxation process is independent of the γ-radiation total dose because of the absorption peaks energy depends on the glass structure and on the photons energy of the gamma source; and that the concentration of the generated color centers increases with the dose but not the nature of their electronic configuration. This last assumption involves the absence of "cross-link" defect kinetics [11]. Therefore, the post-irradiation relaxation process of the glass can be described by the next relationship:

$$A(t,D)=\left[Q_0+P_0\left(1-e^{\left(\frac{D}{P_1}\right)}\right)\right]\left[1-P_2-P_3+P_2\cdot e^{\left(\frac{-t}{P_4}\right)}+P_3\cdot e^{\left(\frac{-t}{P_5}\right)}\right] \qquad (1)$$

where 't' is the time after irradiation, 'D' is the radiation total dose, 'P_i' are the fitting parameters. 'Q_0' is the initial absorption of the glass before being exposed to radiation conditions. 'Q_0' is zero for all the peaks generated after irradiation.

Fig. 7, allows predicting the behavior of the Clearceram optical properties after being exposed to a total dose of γ-rays at any post-irradiation time.

CONCLUSION

Samples of three types of glasses, Fused Silica, CaF_2 and Clearceram, have been exposed to space environment conditions such as high variations of temperature and UV and gamma radiation. Optical properties of these glasses have been analyzed before and after exposure to space conditions using non-invasive and non-destructive optical characterization techniques as Spectroscopic Ellipsometry and Optical Spectrum Analysis.

The optical performances of the CaF_2 and Fused Silica glasses have not been affected by the thermal cycling, UV and gamma irradiation test. On the other hand, the Clearceram optical properties change after exposure to gamma radiation. Variations of the Clearceram glass refractive index real part can be considered negligible after irradiation. However the complex part has been affected due to the absorption generated by the γ-radiation in the visible spectral range. This induced absorption has been fitted by individual Gaussian absorption peaks which correspond to the color centers generated by the ionization of the Clearceram glass. A study about the behavior of the absorption peaks amplitudes versus γ-radiation total dose has been performed with the result of a first order exponential function which models changes of glass absorption by effect of gamma radiation dose. A second study was carried out where it is achieved an exponential function which fits the natural relaxation process of the Gaussian absorption peaks after 800krad gamma irradiation. This relaxation process follows two different decay rates, the faster decay occurs in the first month. A two-variable function which simulates the behavior of the glass with the radiation total dose and the time has been described. This function allows predicting the optical performances losses of a Clearceram glass after gamma radiation exposure. This proposed model is useful to analyze and characterize optical materials for space applications.

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