COMPARATIVE ASSESSMENT OF STAINED-GLASS WINDOWS MATERIALS BY INFRARED THERMOGRAPHY

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Abstract

This paper reports the analyses of infrared thermography images of two stained-glass windows with the objective of the *in-situ* characterization of this type of artworks. The analyses were carried out by active thermography. The observations revealed that glasses absorbed the long-wave IR radiation emitted by the halogen lamps and their apparent surface temperature progressively increased. After switching the spotlight off, they experienced a progressive decrease of temperature. Silver stained glasses presented the same thermographic behavior than uncolored glasses because silver nanoparticles were too small or the yellow layer was too thin to produce a different response than the base glass with the IR radiation. The apparent surface temperature of enamels and grisailles depended on their thickness and color. Lead comes maintained an almost constant surface apparent temperature, except those painted that behave in a similar way than enamels. Metallic tin–lead welds experienced the most important variation of the surface apparent temperature in reflection mode due to the energy reflected by the surface of the weld. Glass defects such as big bubbles were also observed.

Keywords: Glass, grisaille, enamel, infrared thermography

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1. Introduction

Infrared (IR) thermography is a non-destructive and contactless technique which measures the IR radiation re-emitted from the measured targets. An IR camera produces apparent surface temperature images based on calculations from the received IR radiation and black body emission laws [1, 2]. The photo-thermal signal depends on parameters governing the heat diffusion: thermal conductivity, thermal emissivity, thermal diffusivity, temperature, specific heat, and density. In addition, these parameters can be correlated with the following characteristics of the object: aspects of the surface, presence of delamination, presence of cracks, internal structure of the material, progress of a physical and chemical transformation, drying and sedimentation, etc. Active thermography is a methodology for thermographic inspection in which the studied objects are thermally excited by an external heat source and the different thermal behaviors are analyzed mathematically using the Kirchhoff, Planck, and Boltzmann Laws and the different models of heat transfer. Conversely, passive thermography consists on the inspection of the object without being purposefully excited with an external heat source. The focus of this latter methodology is the location of thermal anomalies in the object based on a single image.

IR thermography has been used for more than 25 years to detect subjacent defects in buildings, such as fissures, cracks and humidity, as these features cause contrasting thermal responses. In cultural heritage, the most usual application of IR thermography is moisture location in historic buildings; other common applications in historic constructions are the assessment of previous conservation treatments such as cleanings, consolidations and restorations; and the identification of hidden structures behind the painting of the walls [3-16]. Frescos have been also widely studied with IR thermography to detect previous interventions, hidden damages and, in some cases, to discover the cause of their deterioration (humidity, unknown structures in the wall, etc.) [17-24]. In case of mosaics, IR thermography permits to evaluate the mortar and the suitability of the consolidative materials and treatments [24, 25]. The application of IR thermography is not only focused on constructions, it has been also applied on paintings on canvas or wood [26-29], tapestries [30], books [31, 32] and archaeological artifacts [33, 34] to evaluate their conservation state, to detect hidden damages and to improve their conservation strategies. However, studies on historical glasses are scarce and they are focused on the detection of grisaille detachments [34].

The main drawback for applying IR thermography on glasses is their behavior on the infrared region of the electromagnetic spectrum (wavelength in the range of 0.75–1000 μm). Silicate glasses are mostly

transparent to solar radiation (short-wave infrared) and opaque to thermal radiation (mid- and long-wave infrared). This latter radiation is mostly absorbed, thereby heating up the glass. Much of the absorbed radiation is then re-radiated from the glass inward and outward [35]. This behavior favors that glasses seem opaque and cold in comparison with other building materials. Moreover, the high reflectance of glasses, mainly at incidence angles higher than 45 degrees, can produce erroneous temperature estimations [36, 37]. Nevertheless, IR thermography has been applied successfully to evaluate windows for industrial purposes [38-41], to research fire endurance of tempered glass [42], to study the mechanical defects and the elastic deformations on glass sheets [43-47], to analyze the efficiency of solar cells [37, 48] and to detect damages on fiber optics [49, 50].

Stained-glass windows are composed by glasses, which can be painted with enamels and grisailles, mounted on lead comes to form window panels. These comes are usually welded to each other using a melted tin–lead alloy. The characterization of these materials is commonly carried out during their restoration, when the panels are dismantled and transported to the workshop. The *in-situ* characterization with a non-destructive and contactless technique would be helpful to evaluate the conservation state of the stained-glass windows without being dismantled. The normal procedure for stained-glass windows assessment implies the mounting of scaffoldings and the observation piece by piece; however, the Stimulated Infra-Red Thermography (SIRT) permits the inspection of a wide surface in a very short period of time.

The main goal of this research was to assess for the first time the behavior of the different materials from the stained-glass windows (glasses, enamels, grisailles, lead comes and tin-lead welds) by passive and active infrared thermography with the future objective of the integration of this technique for usual analysis in the characterization of stained-glass windows.

2. Materials and methods

2.1. Stained-glass windows

Two different stained-glass windows were visualized by IR thermography: firstly, “Saint George and the dragon”, a contemporary small size stained-glass window (31 x 31 cm) from a Spanish private collection was analyzed (Fig. 1 a). The second stained-glass window analyzed was a 20th century stained-glass window formed by three panels (158 x 460 cm, each one) elaborated in the Maumejean workshop between 1942 and 1943 which represents the allegories of Sciences with a classic iconography. This stained-glass window is located in the building of the headquarters of the Spanish National Research

Council (CSIC, Madrid) (Fig. 1 b). The window is placed with north orientation in a central courtyard of small dimensions, so it does not receive sunlight directly. Both stained-glass windows present different types of glasses, enamels and grisailles.

Fig. 1. Stained-glass window analyzed: a) “Saint George and the dragon” (Private collection, Spain), b) CSIC headquarters (Madrid, Spain) stained-glass window.

2.2. IR thermography

The characterization of the surface thermal behavior of glasses, enamels, grisailles, lead comes and tin-lead welds were carried out by infrared thermography. The thermographic analysis of both the stained-glass window entitled “Saint George and the dragon” and the 20th century stained-glass window from the CSIC headquarters (Madrid, Spain) was carried out with a FLIR ThermoCAM™ B4. This is a LWIR (Long-wave Infrared) Camera with 7.5 to 13 μm IR wavelength detection range, which corresponds to a -20 to +130 $^{\circ}\text{C}$ temperature detection range. Temperature accuracy is 0.08 $^{\circ}\text{C}$ and the camera was equipped with a 25 $^{\circ}$ FOV (Field of View) lens. As the analyses did not aim to acquire accurate temperature measurements but to detect thermal anomalies and perform active thermography calculations, emissivity was set to a constant value (0.96) in both transmission and reflection modes. The camera was set at 1 m distance. For thermal excitation, two 500-watt halogen lamps located at 1 m from the glass were used. The acquisition lasted 20 min: 10 min during heating and 10 minutes cooling after turning off the heating source with a frequency of acquisition of one capture per minute. “Saint

George and the dragon" glass window was analyzed in the laboratory in February 2012, being room temperature around 20 °C throughout the experiment and the 20th century stained-glass window was analyzed on site in the CSIC headquarters (Madrid, Spain) in August 2011, being room temperature around 30°C.

In addition to the apparent surface temperature evolution of discrete pixels with time, Principal Components of Thermography (PCT) analyses were performed.

PCT analyses were made on each of the series of images recorded at equal times. The surface apparent temperature of each pixel in these series of images is a function of space and time. PCT analyses are based on searching linear combination of images within the series that diagonalize the covariance matrix (i.e. the matrix representing the covariances of temperature-time, temperature-space and time-space). The result of each linear combination is another image that is called PCT, with an associated eigenvalue. The larger the eigenvalue of a certain combination, the larger is the contribution to the total variance of the process. The linear combination of images contributing the most to the total variance is the PCT-1 component and in descending order of contribution PCT-2, PCT-3, etc.

In each PCT image, the scale represents the covariance; therefore darker (lower) negative values indicate areas with a negative covariance in relation to that specific component. The set of PCT allows categorizing what areas of the object are being affected more than others are by the thermal excitation as well as recognize the contribution of different processes in the thermal excitation (materials, defect, etc.) By analyzing each PCT areas behaving analogously in relation to a process can be recognized. PCT images were obtained through the application *ir_view* (v. 1.7.5) developed with Matlab [51]. PCT analyses have been used previously to analyze materials in heritage buildings [52-54], but have not been performed previously for glass windows.

The color of the glasses was measured with the portable colorimeter "Blue line" CM-310 from Metrotec in reflective light mode placing the white standard under the analyzed area.

3. Results

3.1 Analyses of the evolution of the apparent surface temperature

3.1.1. Glasses

Glasses are almost transparent to short-wave IR radiation, but they are opaque to mid- and long-wave IR radiation [35]. Usual halogen lamps, as used in this study, emit from 0.35 to 4-5 μm depending on the filament, with the maximum emission around 1.0 μm [55, 56]. The transmittance of glasses to

radiations from 0.3 μm up to 2.5 μm is around 0.9 and the reflection is around 0.07, which means that they are practically transparent to the radiation [36]. Nevertheless, the glass transmittance for radiations with wavelengths between 2.5-4.0 μm decrease to ~ 0.4 , and for wavelengths higher than 5.0 μm the transmittance is almost null [36, 57]. The wavelength range of the thermographic camera (7.5 to 13 μm) coincides with the absorption bands of the bridging and non-bridging Si-O bonds [58]. This radiation is absorbed by the glasses, heating them up.

According to this fact, the behavior of glasses of the stained-glass windows depended on the thermographic mode applied (Fig. 2 a). In transmission mode, glasses presented a significant increase in their surface apparent temperature due to the heating induced by the absorption of the silicate bands, and also due to the radiation transmission through them. However, in reflection mode, glasses showed the increase of the surface apparent temperature due to the warming of the glass surface and the reflection of the IR radiation. The heating was more intense in transmission mode than in reflection mode because of the higher influence of the lamp radiation, which depended on the distance of the camera. Therefore the cooling, when the spotlight was switched off, was slower in reflection mode (Fig. 2 b).

Fig. 2. a) Surface apparent temperature map evolution in transmission and reflection mode at 0, 10 and 20 minutes of essay in “Saint George and the dragon” (Private collection, Spain). b) Thermal variations of an orange glass in transmission and reflection mode.

3.1.2. Silver stained glasses

The stained-glass window “Saint George and the dragon” presents three silver stained glasses with different yellow tones. Historically this coloration was obtained applying a mixture of silver salts, clays or ochres, water and Arabic rubber on one of the glass surfaces and firing them at temperatures between 500 and 650 $^{\circ}\text{C}$. During firing, silver cations penetrate into the glass by ion exchange with the sodium or potash cations, and then are reduced to their metallic state nucleating into clusters. The final color of the glass varies from pale yellow to dark orange and it depends on the oven temperature, the firing time,

the type of paint and even the glass composition because these parameters influence the size, shape and distribution of the silver nanoparticles [59-61].

Despite their different coloration (CIE-L*a*b* coordinates of Yellow1: $L^* = 72.98$; $a^* = -2.01$; $b^* = 34.03$; Yellow2: $L^* = 85.59$; $a^* = 7.95$; $b^* = 37.88$; Yellow3: $L^* = 53.13$; $a^* = -4.98$; $b^* = 55.71$), the three silver stained glasses presented a thermographic behavior similar to the base glass because silver nanoparticles were too small or the yellow layer was too thin to produce a different response than the base glass with the IR radiation (Fig. 3).

Fig. 3. Thermal variations in “Saint George and the dragon” (Private collection, Spain) measured in reflection mode of uncolored glass and yellow silver glasses.

3.1.3. Enamels

An enamel is a brightly colored glass formed by a highly fusible glass with a small amount of pigment. The powdery enamel is applied onto the glass pane with a brush and then fired. The result is a thin homogeneous layer of glass of a bright color on the glass surface [62, 63]. Enamels present different colors depending on their thickness and colorants, and could be transparent or translucent depending on the firing regime.

During the illumination with the halogen lamps, the areas painted with enamels experienced a higher increase of their surface apparent temperature in comparison with the glass support (Fig. 4).

Fig. 4. Thermal variations in “Saint George and the dragon” (Private collection, Spain) measured in reflection mode of a glass without surface layer and glasses painted with a thin, medium and thick layer of orange enamel.

A relation between the thickness of the enamel layer and the surface temperature was observed. The surface apparent temperature of the glasses painted with the thin layer of enamel showed a similar behavior than glasses without surface layer (Fig. 4). However, those glasses painted with thicker layers from the same orange enamel presented slightly higher surface apparent temperatures (Fig. 4). This behavior is probably related to the low transmittance of glassy materials to mid- and long-wave IR radiation which induced the heating of the surface layers.

The second variable which modifies the surface apparent temperature of the enamels is their color. For opaque materials, light colors present a higher reflectance than dark colors [35]. The enamels presented the same behavior. Enamels with darker colors increased more the surface apparent temperature in comparison with those ones with lighter colors (Fig. 5 a and b). The areas without enamels showed the lower increase in temperature.

Fig. 5. a, b) Linear thermal variation in the stained-glass window from the CSIC headquarters (Madrid, Spain) measured in reflection mode after 10 minutes of irradiation (Average of 3 pixels in vertical).

3.1.4. Grisailles

A grisaille is a dark paint applied to draw the contours and details of the figures and to produce the effect of shades and volumes. Grisailles are made by powdering a highly fusible glass with pigments such as iron oxide, but it also often contains copper, tin, and/or manganese oxides. During firing the grains of fusible glass melt and fix the pigment grains onto the glass surface [62, 64]. They usually present dark colors such as black or brown.

Grisailles showed the most important increase of surface apparent temperature during the illumination because of their lower albedo, and subsequent higher absorption of thermal radiation (Fig. 6 a and b). A similar effect was observed in a glass piece with grisaille from the French Cathedral of “Chartres” analyzed by photothermal analysis [34].

In the glass decorated with a dragon, the area with the highest temperature increase was the wing and the neck on the dragon because there was a higher concentration of grisaille lines. In addition, they can also affect the surrounding elements increasing their surface apparent temperature.

Fig. 6. a) Detail of “Saint George and the dragon” (Private collection, Spain). b) Thermal variations of glass, grisaille, green enamel and red enamel in reflection mode (from the points indicated in the Fig. 6 a).

3.1.5. Metallic materials

Metallic materials presented a completely different thermal behavior. In transmission mode, the low heat capacity of metals produced an almost constant surface apparent temperature (Fig. 2 a). In reflection mode, the lead came showed a slight temperature rise because the emissivity coefficient of non-oxidized lead is 0.057-0.075 [65], while tin–lead welds presented an important increase of the surface apparent temperature (~ 40 °C) mainly due to the reflection of the energy emitted by the halogen lamp on the fire-polished surface of the welds (Fig. 7 a).

The 20th century stained-glass window presented the metallic parts painted black, and their behavior was similar to dark enamels (Fig. 7 b).

Fig. 7. a) Thermal variations in "Saint George and the dragon" (Private collection, Spain) measured in reflection mode of a glass, lead came and tin-lead weld. b) Thermal variations in the stained-glass window from the CSIC headquarters (Madrid, Spain) measured in reflection mode of a glass, dark enamel and painted lead came.

The metallic frame also induces an increase in the surface temperature from the border of the glass to the center contrary to the windows with wood frame [38]. This different effect could be due to the different transmission of the heat in the frames.

3.1.6. Glass defects

Infrared thermography was also applied to identify defects on glasses such as bubbles. They were observed without illumination and after 10 minutes of irradiation in transmission and reflection mode. Large bubbles were better observed without artificial illumination due to the reflection of the environmental IR radiation in the irregularities of the glass surface because, after illumination, the glass surface was heated and it masked the bubble (Fig. 8). According to Campa *et. al* [66], bubbles can increase more the surface temperature during the heating, however, in this case, the glass hide it.

Fig. 8. A large bubble in the glass observed by IR thermography without illumination and after 10 minutes in transmission and reflection mode.

3.2. Principal Components of Thermography analyses

The PCT analysis was applied to the thermography images obtaining the percentage of contribution to the total covariance in each case (Figs. 9, 10 and 11). All images showed extreme positive and negative values corresponding to the reflective behavior of the lead comes (Figs. 9 a, 10 a and 11 a), which need to be excluded from the image analyses that will focus only on glass. In the series of images, PCT-1 shows most of the information, being PCT-2 and PCT-3 redundant or lacking any sort of information. The exception would be a pattern revealed in the PCT-3 from the book spine detail (Fig. 10 c) which could be a different reflection of the glass surface or may be associated to a thickening of the putty beneath.

In the 1940's stained-glass window from the CSIC headquarters (Madrid, Spain), the painted glasses were the areas which showed higher values of covariance (lighter colors) as opposed to unpainted glass (dark colors) and a correlation between the thickness of the enamel and contribution to the total covariance can be observed (Fig. 9 and 10).

In the case of the contemporary panel, the factor affecting the most to the total variation of the series of images was the presence of grisailles, which were accurately observed in the PCT-1 image (Fig. 11 a). The presence of the red and green enamels used in the dragon and the knight was observed in the PTC-2. However, the orange enamels, which covered some of the glass pieces, did not show an especial response on the PCT analyses.

Fig. 9. Principal components of thermography analyses of the open book detail in the 20th century stained-glass window (Madrid, Spain) in reflection mode. a) PCT-1, b) PCT-2, c) PCT-3.

Fig. 10. Principal components of thermography analyses of the book spine detail in the 20th century stained-glass window (Madrid, Spain) in reflection mode. a) PCT-1, b) PCT-2, c) PCT-3.

Fig. 11. Principal components of thermography analyses of the small stained-glass window (Private collection, Spain) in reflection mode. a) PCT-1, b) PCT-2, c) PCT-3.

4. Conclusions

Two stained-glass windows were successfully characterized with infrared thermography. Each material showed a different behavior. Glasses were heated due to the absorption of the mid- and long-wave IR radiation emitted by the halogen lamps which lead a progressively increase of apparent surface temperature followed by a progressive decrease after switching the spotlight off. In transmission mode, the apparent surface temperature is higher due to the transmission of the radiation through them. Silver stained glasses presented the same thermographic behavior than base glasses because silver nanoparticles were too small or the yellow layer was too thin to produce a different response than the base glass with the IR radiation. Enamels and grisailles experienced a higher increase of their surface apparent temperature in comparison with the glass support, which depended on the thickness, and color

of the surface layer. Metallic materials presented an almost constant surface apparent temperature in transmission mode due to its low heat capacity; although in reflective mode the energy reflected by the surface of the metal induced the fictitious increase of the surface apparent temperature. The thermal behavior of metal was different when they were painted; then, their behavior was similar to enamels. Glass defects such as big bubbles were also detected by infrared thermography. PCT analysis of thermal images proved to be successful in detecting the influence of paintings and grisailles in the thermal response of glass windows. The differences between the behavior of early 20th century glass windows in relation to contemporary one may be a result from the evolution of composition and techniques, as painting and its thickness is the main factor affecting the thermal response in 1940's glass windows, while grisailles (and not enamels) are the main driving factor for change in contemporary glass windows. The complementary study with metrology could analyze the real temperature of the surface and not only apparent temperature; however, IR thermography has proved to be a useful tool for the *in-situ* analysis of historical stained glass windows.

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