Surface degradation of coating measured by means of emissivity change

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Abstract

We present an indirect application of thermographic measurement on determination of surface properties of protective polymeric coating painted on glass. The application can serve as a nondestructive, noninvasive and noncontact technique for assessment of surface roughness of the coating and help to assess a degree of its degradation.

Key words: polymeric coating, IR thermal imaging, emissivity, surface roughness

1. Introduction

Increasing general availability of thermal imaging cameras makes it possible to use them in areas beyond scope of typical engineering applications. Advent of thermography in the field of cultural heritage protection several decades ago has been mostly associated with detection of surface or subsurface defects and anomalies like presence of moisture etc. In this paper, however, a method for use of a thermocamera for surface quality assessment as a necessary step in a proper care for historical monument is outlined.

The most valuable glass mosaic in the Czech Republic is mosaic of the Last Judgment on the St. Vitus Cathedral located within Prague Castle (see Figure 1). The mosaic was created at the request of Emperor Charles IV by Venetian masters and finished in 1371. Due to its composition, chemical durability of medieval glass is often low and therefore it is necessary to protect its surface, because surrounding environment steadily damages it. Further damage accumulation is to be eliminated or at least suppressed by additional protective polymeric coating which is expected to last for a long time. Therefore the most stable and environmentally resistant coating has to be selected among the prospective candidates based on accelerated test.

In addition to finding the optimal chemical formula for the protective coating, also a technique for long term monitoring is being developed as an integral part of the project called "Technology of maintenance and conservation of the Last Judgment mosaic and methods of medieval and archaeological glass conservation". The development concentrates mostly on optical methods determining reflectivity and gloss of the surface as a measure of undergoing physical and chemical degradation processes. Also IR thermal imaging is considered, because it offers many advantages in comparison to the optical methods. In fact, these two approaches suitably complement each other and thus are both adopted.

In the following text a first study on application of IR thermography for assessment of state of coating surface is presented. The paper is organized as follows: after a brief summary of advantages of thermal imaging methods over optical methods, some details of investigated material and testing procedure are presented. An influence of surface roughness on thermal emissivity is then shown on a case study for opaque material and then evaluation of coating on a glass specimen is assessed. Finally, the pros and cons of the technique are discussed.

2. Motivation and description of the presented technique

Surface inspection is common and crucial task in many research activities. Nondestructive and noncontact measurement is required in many application. Concerning monitoring methods, the traditional photo documentation is not suitable for quantitative analysis of the surface state. Optical methods evaluating reflectance and gloss of the surface promise more reliable results. But due to local variation of glass pieces, significant effort has to be exhibited to comply with a requirements of the technique, i.e. to find a suitable location on a specimen or mosaic. Therefore also other approaches are simultaneously examined.

3. Relation between surface roughness and emissivity

It can be shown that in a micrometer range, in which thermocameras operate, the surface of the coating appears fully reflective first, and only gradually loses its mirror-like appearance as various defects accumulate. This process is the core of the technique for determination of reflectivity as a proxy to surface roughness measurement.

The surface is considered optically flat if deviation from ideal state does not exceed a fraction of wavelength in which it is observed. Therefore glass pieces that fail to be studied by optical method due to a number of irregularities, in infrared range can be seen as specular, mirror-like. In another words, what looks dull and diffuse in visible light can be still shiny in middle short waves range.

Therefore a surface that appears smooth and specular in longer-wave radiation while in shorter-wave the same surface looks rough. The basic trend is inversely proportional relation between observation wavelength and specularity [1]. Therefore reflectivity and emissivity are essential for indirect quantitative characterization of the surface. There is also important relation between emissivity, reflectivity of the surface as a measure of is roughness [2]. The shinier the surface, the lower emissivity is detected and vice versa. Soft diffuse surface, called Lambertian, is defined as a surface reflecting incident radiation isotropically in every direction evenly.

If successfully implemented, the technique can help conservators in selection of most weatheringresistant material for coating and also to help in monitoring the mosaic on site.



Figure 1. Mosaic of Last Judgment with detail of angel head

4. Properties of medieval glass and protective layer

Original medieval tesserae were prepared from glass containing oxides used to decrease melting temperature. This way it was easier to produce the glass, but on the other hand these oxides make the glass prone to degradation in reaction with environment. The protection of the mosaic is based on multilayered films of organosilanes fluorinated polymers and polymers (for more detail on protective concept of the layers see [3, 4]). The coating should protect the glass pieces from environmental forces like acidic rain, chemical action of pollutants adhering on surface and abrasion due to dust particles propelled by wind.

Glass tesserae are produced by elaborate breaking of larger piece of glass and this process yields to characteristic wavy surfaces. Dimensions of these surface feature exceed significantly thickness of the coating and further complicate the situation for monitoring.

5. Experimental procedure

First, the technique described in ISO standard for proper measurement of surface temperature, was applied on test specimens in different stage of wear that manifests itself as a decrease of specularity. Surface roughness was found to influence surface emissivity this way. Emissivity and reflectivity of the surface in infrared range can be determined using technique similar to the standardized method for emissivity measurement [5] based on difference between real and apparent surface temperature measured by thermocamera. Assumed true temperature of the studied surface is known, if one observes reflection of an object kept at different temperature (e.g. by means of Peltier cooler), resulting measured apparent temperature can be corrected in thermocamera (FLIR SC7600 with a cooled detector) by adjusting the value of surface emissivity.

Accumultation of surface damage can be determined directly by various mechanical measurement of roughness, or indirectly, based on optical properties of surface. This relation between surface topography and reflectivity or emissivity of material is well described in literature [2], where it is also possible to find another methods exploiting this principle [6, 7, 8].

The monitoring technique should prove the stability of coating. The degradation of the coating can manifest itself in the form of transition from transparency to translucency, various types of delamination and also as an increase of surface defect presence. All these defect decrease aesthetical appearance of the mosaic pieces and have to be avoided.

The intention of this work was to verify the technique on "standard" opaque specimen first and then on coated glass.



Figure 2: Set of coins as a progressive sequence of surface weathering, photography and SEM micrographs

Surface variation vs. emissivity



Figure 3: Surface variation vs. emissivity

6. Correlation emissivity – roughness for opaque specimens

Set of coins (Figure 2) of identical denomination selected at different level of weathering was used to simulate process of long term aging or weathering (combined soiling, oxidization and scratching). A direct relation between the weathering of surface and emissivity was found. The origin of visual difference between coins was studied by means of scanning electron microscope (SEM TESCAN Mira II). Set of micrographs from SEM show detailed surface appearance of specimens at the scale decisive for emissivity in IR. Increase of surface roughness in the most worn ones is associated with an increase of emissivity. This way a relation between visual appearance and SEM micrographs was established. In the second step, relation between emissivity of the surface and its roughness was determined. In secondary electron detector, that enhances topographic features of surface by 'shadows' cast on the surface. Variability of this shadows (expressed as mean of local variations) reflects roughness as microtopography, is found in strong correlation with emissivity measurement (Figure 3). This confirms validity of original proposal to apply determination of emissivity to estimation of surface roughness as a measure of surface degradation.

7. Correlation apparent temperature – aging for semitransparent specimens

Unfortunately, the approach was not possible to apply also on coated glass (microscope slides) specimens because of their significant transparency that hinders a procedure balancing radiation contributions from specimen itself and from surrounding sources, but assuming being nontransparent for thermal radiation.

Due to high degree of transparency of thin microscope slide, only a small fraction of energy radiated from the surface of glass belongs to glass itself or the object reflected in its surface. As the settings of thermal imaging camera does not allow for adjustment of transparency of object, it is not possible to use adjustment directly for assessing the surface properties.

Emissivity distribution for various angles of incidence and reflectance is determined and function of emissivity is a measure indication whether the surface is specular or reflective.

Fortunately, there is also a relation between emissivity an observation angle, the closer observation direction is to the normal of the studied plane, the more pronounced is component of reflection. Adopted approach departures from characteristic function of emissivity in relation to incidence angle and surface normal [9]. Measurement was carried out on polymer coated microscope slides. Specimens of controlled roughness were prepared in laboratory by means of artificial aging. The polymeric layer on glass specimen – microscope slide - was prepared by dip coating technique. Degradation of the coating was invoked by accelerated aging tests carried out at high level of RH in humidity chamber and by UV light in QUV Weathering Tester.

Emissivity of object surface can be favorably expressed in polar coordinates. In this representation, Lambertian surface is depicted by a circle, indicating even distribution of radiation to every directions [9].

Emissivity of the surface is also a function of observation angle. If the surface is observed in direction of its normal, the emissivity is the highest, and as elevation of direction of observation decreases, the emissivity also gets lower. In the case of diffusive surface the emissivity is the same for every elevation angle. The relation of angular dependency of emissivity can be utilized to comparatively determine surface quality on the scale from the full specularity to diffusivity.

Position on this scale then play a role of indicator of surface degradation – originally smooth surface of protective coating gets damaged, which means it is scratched, roughened, loses its transparency etc.

Because of the reasons mentioned above, it is not possible to directly measure emissivity of transparent material, but apparent temperature alone can be used as a proxy to process of surface degradation. The procedure can be as follows: for a several pairs of equal angles of incidence and reflectance the apparent temperature are measured by thermocamera. The smaller deviation of apparent temperature for various angles, the closer the surface is to fully diffusive state. Therefore the surface can be characterized by only one value. The high R² value resemble sound interpolation. The tendency of data points to grow with undamaged surface is shown on the Figure 4.



Figure 4: Dependence of maximal apparent temperature of specimen on its aging

8. Discussion and conclusions

Prior using this technique in the field, calibration tests on control specimens have to be carried out, establishing correlation between roughness and emissivity.

Attempt to repeat measurement also on handheld bolometer FILR i5 was unsuccessful as it was virtually impossible to point to the same location for time necessary to set proper emissivity in camera in order to obtain the same value of apparent temperature as the real temperature from contact measurement. Based on this preliminary study it can be concluded: The presented methodology is to be also elaborated as a field test. The technique for inspecting the mosaic, promises to overcome limits of optical methods. In spite of the technique being developed and calibrated based on cooled research thermocamera, even the simplest handheld devices can be used with satisfactory results provided it is fixed on tripod and therefore became tool in conservator's practice. IR thermography can be used as and complementary method. Two separate wavelength ranges extends possibilities of surface quality monitoring to wider variety of surfaces and their damages.

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