COMPOSITES SURFACE STATE ASSESSMENT BY LIF METHOD

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Abstract. The application of fibre reinforced composites has been increasing in many branches of industry. One of the techniques used for composites joining is adhesive bonding. The joints can be made during both manufacturing and repair. The reliability of the bonding is crucial for safe usage of the structure, therefore it is important to assess the pre-bond state of the surface. The performance of adhesive bonds depends on the physico-chemical properties of the adhered surfaces. This research is focused on characterization of the surfaces before bonding. In-situ examination of large surface materials determine the group of methods that are preferred. The analytical methods should be non-destructive, enabling large surface analysis in relatively short time. In this work a Laser Induced Fluorescence (LIF) method was tested that can be potentially applied for surface analysis. This is a noncontact method allowing for inspection of relatively large surfaces in short time. In the reported work LIF intensity and time characteristics were investigated. The investigated samples were either surface contaminated or overheated. It was shown that laser induced fluorescence method can be useful for non-destructive evaluation of CFRP surface and some of the investigated cases can be easily detected.

Introduction

Adhesive bonding is one of the methods of joining composites. For example, it is used in aeronautics and wind turbines manufacturing. There is a extensive research towards effective methods of bond quality assessment [1]. The performance of adhesive bonds depends on the physico-chemical properties of the bonded surfaces. Previous destructive research proved that thermal degradation of composite material or contamination of the surface with hydraulic fluid (Skydrol) weakens the adhesive bond of CFRP [2]. So it is important to assess the surfaces before bonding. An overview of the non-destructive testing (NDT) methods used for the assessment of the surface before adhesive bonding was presented in [3]. In the research reported in this paper we focus on, the Laser Induced Fluorescence (LIF) which is one of the NDT techniques that can be applied for in-situ material examination. Initial results for CFRP surface assessment using LIF was presented in [4]. Promising results, especially for thermally degraded samples were observed. In the research reported here wider set of cases such as: spot overheating, different times of exposition to heat source and higher temperatures were investigated.

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1. Experimental

In-situ examination of large surfaces such as passenger aircraft fuselage parts determine the group of methods that are preferred. The analytical methods should be non destructive and enabling large surface analysis in relatively short time. In this work research focus is on a spectroscopic method that can be potentially applied for large surface material analysis - LIF. This technique allow to analyze large objects using surface scanning technique [5]. Additionally spatial distribution of fluorescence intensity can be monitored with CCD imaging camera equipped with bandpass optical filters.
LIF spectra were recorded using laboratory system equipped with laser excitation sources: DPSS cw Nd:YAG 532 nm, 0.2 – 2 W output power (Spectra Physics) and pulsed Nd:YAG 532 nm, 6 ns pulse diration (Briliant B, Quantel). The 532 nm excitation was chosen following previous research reported in [4]. Excitation at this wavelength showed maximal sensitivity to surface state condition. Fluorescence detection system was based on 0.3 m Czerny-Turner type monochromator (SR-303i, Andor) and spectroscopic ICCD camera (DH-740 Istar, Andor). In the detection path excitation laser radiation was blocked by a band pass filter (OG550, Shott). The fluorescence spectra were recorded in 10 randomly chosen points for each sample in order to avoid local surface inhomogeneity. Additionally, the spatial distribution of laser induced fluorescence was recorded with digital camera equipped with cut-off bandpass filter (OG570, Shott).
Thermal processing of samples was carried out by means of heat gun producing stream of hot air 5 mm in diameter and temperature adjustable in the range from 200 to 480 ^{0}C as well as hotplate with temperature adjustable in the range from 20 to 500 ^{0}C.
Contamination of composite samples with hydraulic fluid/water mixture was realized by preparing a Skydrol water mixture that was stirred at 70°C for 4 weeks. Then the aqueous phase was extracted and mixed with water in order to create 3 solutions that differ with the pH level. The samples were immersed in these solutions for 40 days.
Three type of prepreg CFRP samples were investigated. Woven samples were analyzed in thermal processing research and samples with unidirectional layers were investigated in terms of surface contamination.

2. Results

2.1 Effect of exposition to hot gas stream of constant temperature in different time

The influence of duration of heat source exposure was investigated. The research was conducted on woven CFRP samples with 4 layers and 3 mm thickness. The heat gun was set to constant temperature of 450°C. Four times of exposure were tested 5, 10, 20 and 30 s. The photographs of the heat exposed area under the LIF laser illumination are presented in fig. 1. The woven structure of the composite is visible. The increase in the intensity in the visible light is clearly observed. The photographs were digitized in order to quantify the increase in fluorescence. The results are depicted in fig. 2. The shortest exposure (5s) almost did not influence the investigated surface. With increasing time of heating a clear circular shape forms indicating the overheated area. In a horizontal 1D view the heat influence is even more obvious (fig. 3). The 5 and 10 s heating have very low influence, while for 20 and 30 s heating, one observe a significant fluorescence intensity increase. Moreover the periodic surface structure caused by the presence of carbon fibers is reflected as local maxima observed in fig. 3.

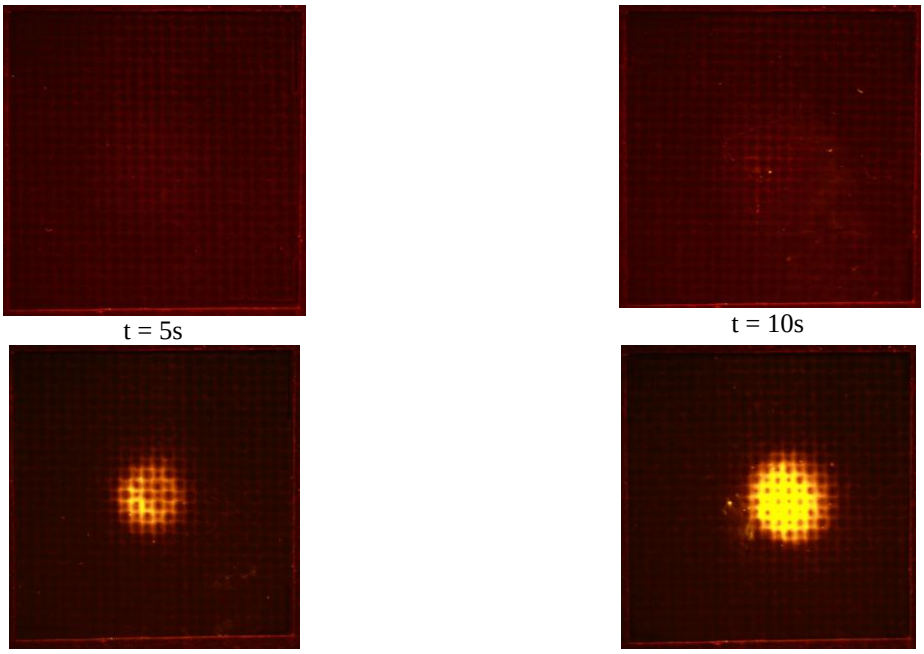


Fig. 1. Fluorescence intensity distribution registered at samples exposed to heat source at 450°C for 5, 10, 20 and 30 seconds. Picture made with photo camera. The displayed area is 50 mm x 50 mm.

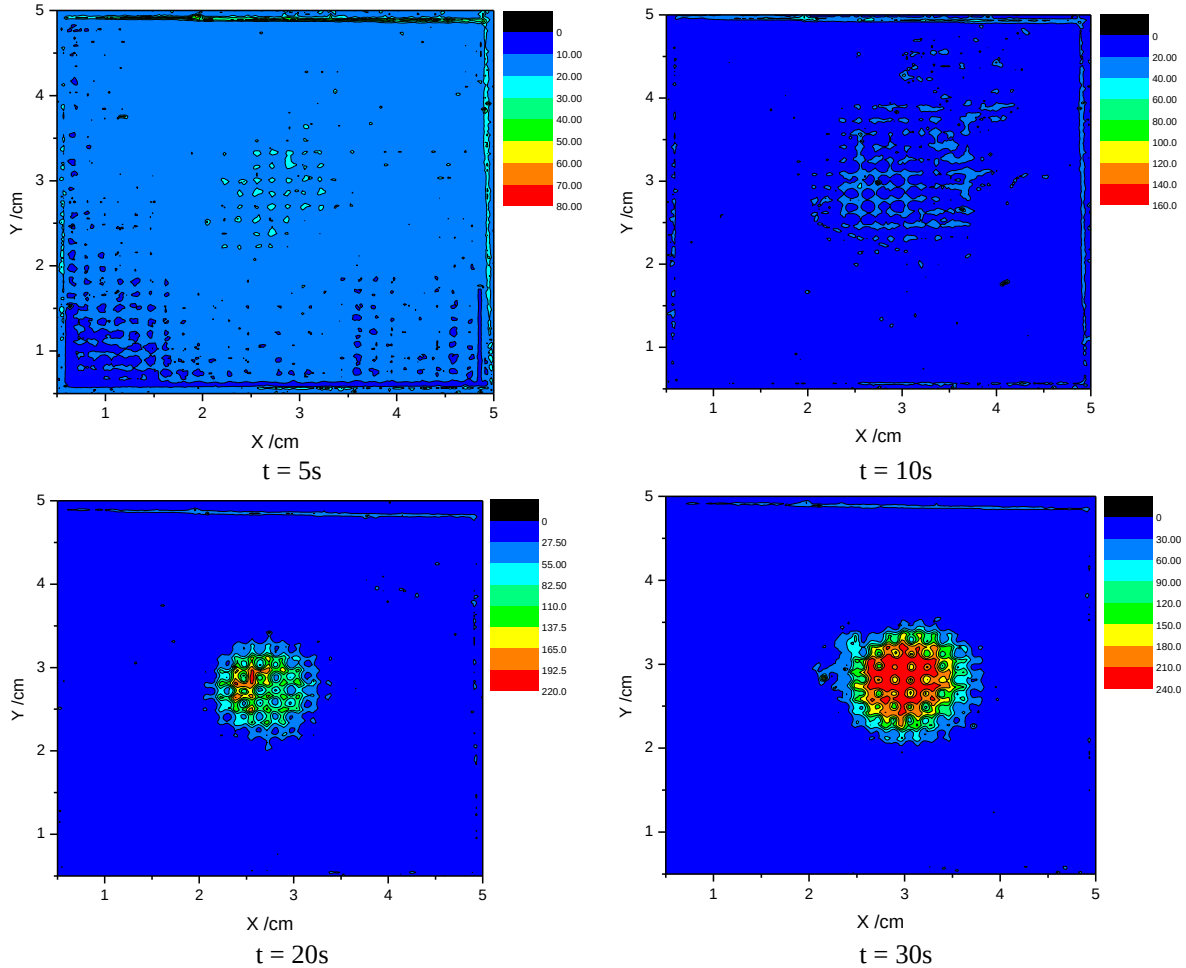


Fig. 2. Fluorescence intensity distribution registered at samples exposed to heat source at 450^{0}C for 5, 10, 20 and 30 seconds. Processed photographs from fig. 1.

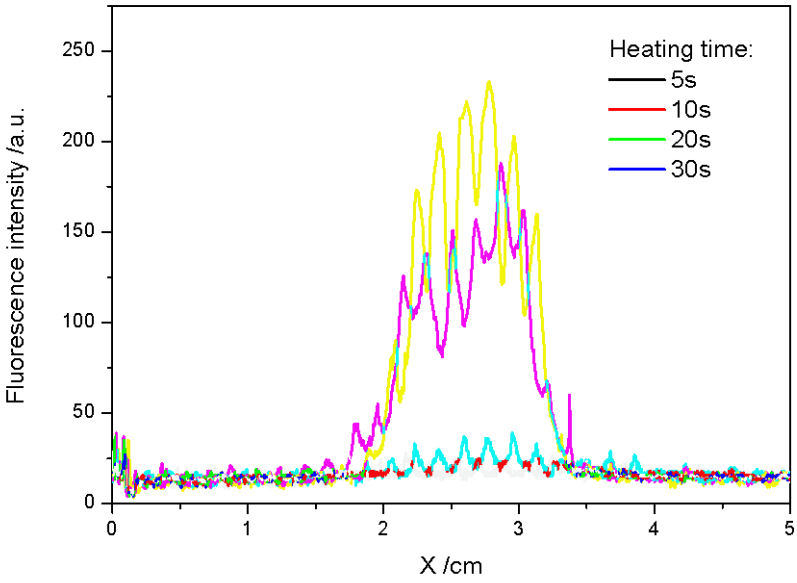


Fig. 3. Dependence of intensity distribution on time of exposition to heat source. 1D view based on 2D images presented in fig. 2.

In the next step the LIF response was measured with spectrometer. For each time of exposition the response was measured in 10 random points. The response is very non-uniform. The intensity varies from point to point. As an example the response for 10 s heating was plotted in fig. 4. The same behaviour was observed for the other heating times (5, 20 and 30 s). This non-uniformity in the materials has to be taken into account because it can lead to measurement errors.

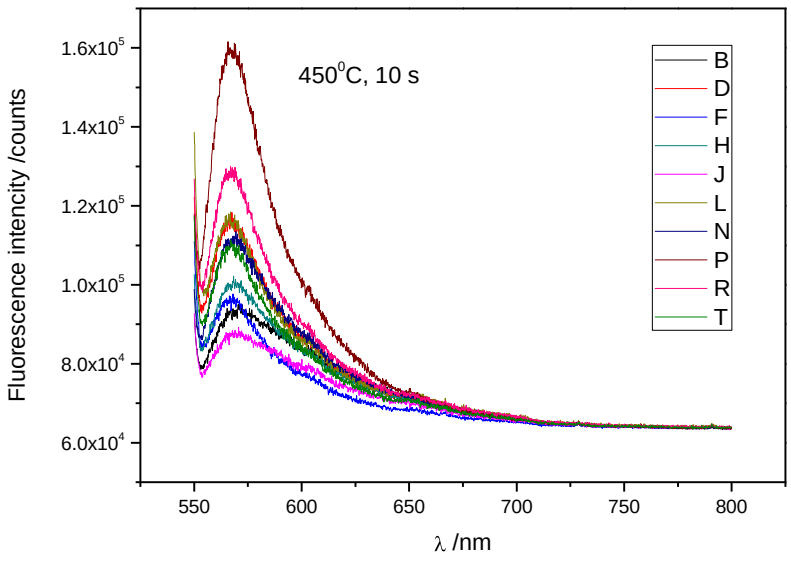
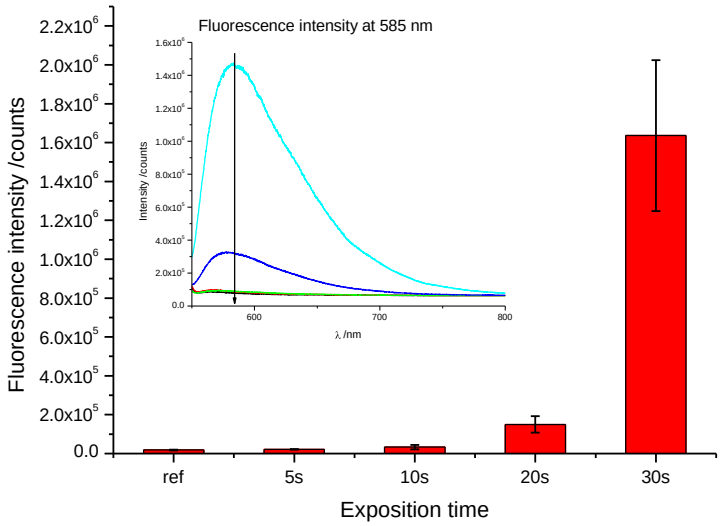
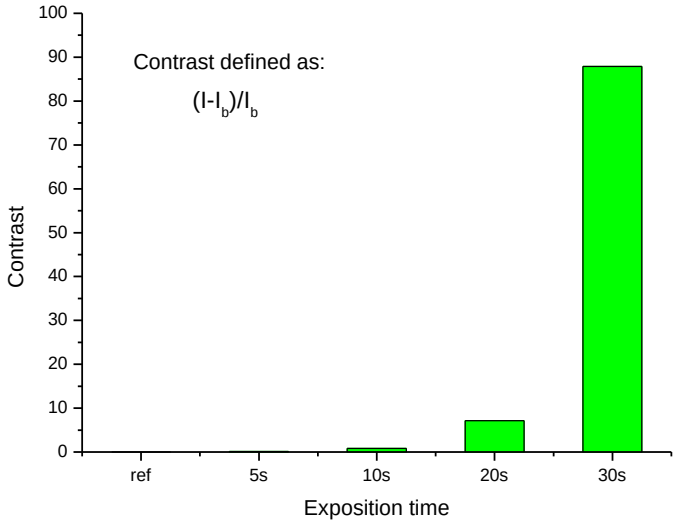


Fig. 4. Intensity measured at 10 points on the sample under 10s exposition to heat source; non-uniform distribution is observed; similar non-uniform response was also noticed for 5, 20 and 30 s.

The mean fluorescence intensity over the 10 points was presented in fig. 5. The intensity value was calculated as a peak value at 585 nm (fig. 5a). Significant changes in the intensity occur for 20 s heating and longer. Increasing the exposure by 10 s (up to 30 s) results in intensity value increase by an order of magnitude. In order to comparatively compare the obtained values a contrast value was calculated in relation to the reference measurement. The result was depicted in fig. 5b).



a)



b)

Fig. 5. Mean fluorescence intensity in relation to heat exposition time calculated as: a) peak value at 585 nm; b) calculated contrast value.

It should be noted that there are differences in results obtained by photographs by spectra registration. In spectroscopic results there is a significant increase of intensity between the 20 and 30 s cases (fig. 5a), while in photograph analysis (fig. 3) only a slight increase was observed. The discrepancy can be explained by the nonlinear characteristic of the digital camera. It is linear only for middle range of intensities while for very low or high intensities the characteristic is exponential. Nevertheless, the photography analysis is useful for analysis of spatial distribution of the fluorescence phenomenon.

2.2 Effect of thermal processing in different temperatures

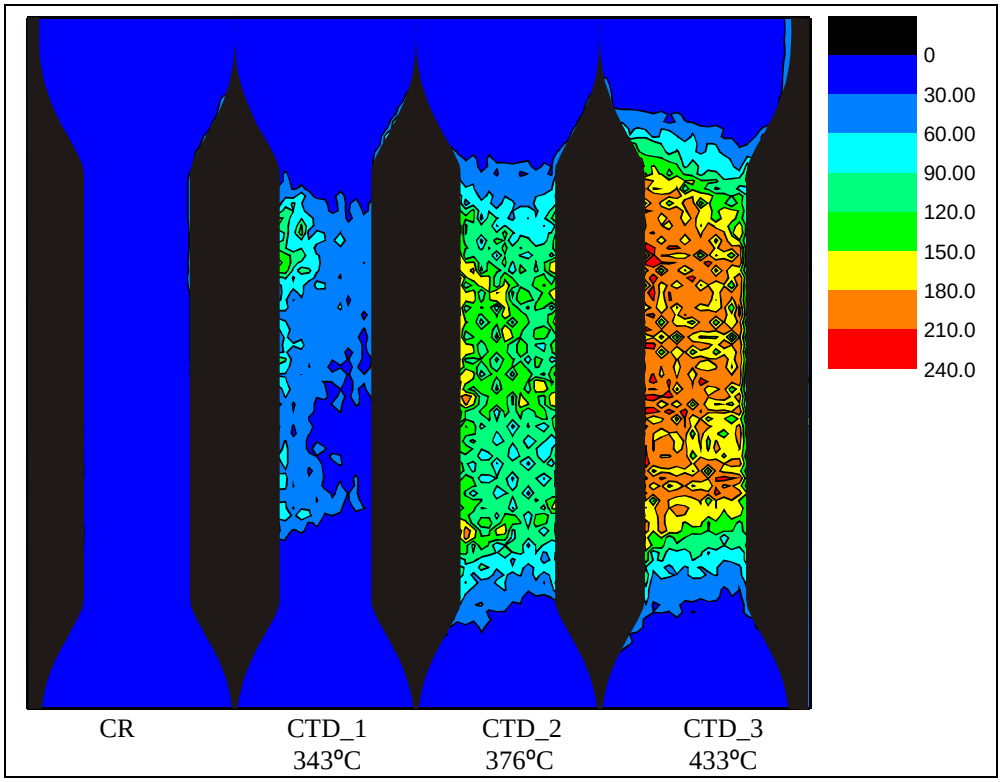


Fig. 6. Fluorescence intensity distribution computed from the photographs for reference sample and three heated on a hot plate at 343, 376 and 433°C.

The second part of investigation was devoted to the influence of the temperature to which the composite was exposed to. The research was conducted on woven CFRP samples with 2 layers and 1.5 mm thickness. The samples were prepared in dogbone shape for further destructive testing (fig. 6). They were heated on a hot plate. The level of thermal degradation was determined by mass drop of sample. The sample were given following symbols:

1. CR – reference,
2. CTD_1 – exposed to 343^{0}C for 180 s (start of degradation, 2% mass drop),
3. CTD_2 – exposed to 376^{0}C for 180 s (5% mass drop),
4. CTD_3 – exposed to 433^{0}C for 180 s (corresponds to fastest degradation),
5. CTG – exposed to 136^{0}C for 600 s (10^{0}C above glass transition temperature).

Characteristic temperatures were taken from Thermogravimetric Analysis TGA (mass drop temperatures) and Dynamic Mechanical Analysis DMA (glass transition temperature). The analysis of the digital camera photographs indicate the higher intensity for higher heating temperature. The CTG sample gave the same results as REF so it was not plotted in fig. 6. For the spectroscopic analysis four samples of each kind were measured. The CTD_x samples characterize with clearly higher intensity than the CR and CTG samples (fig. 7). This is due to much higher temperature of heating. Within each set of the samples prepared in the same conditions one can observe non-uniformity in the result. The CTD_1 and CTD_2 samples are hardly distinguishable. But there was little difference in the temperature to which they were exposed to. The CTD_3 samples gave the highest intensity. In order to compare the samples an average values of intensity were calculated and plotted

in fig. 7. The correlation of the exposure temperature with the intensity value is clearly visible.

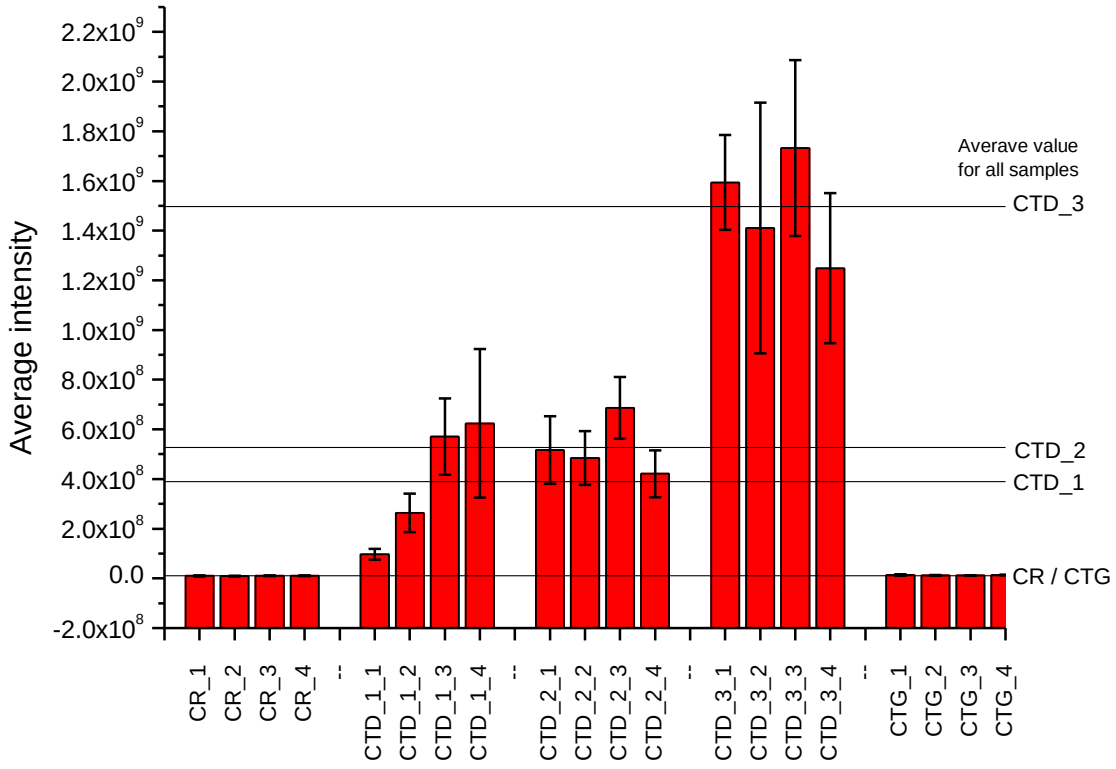


Fig. 7. Changes of the intensity for 4 sample of each kind together with the mean value.

2.3 Effect of surface contamination

The results showed in previous sections indicted that LIF intensity is a sensitive feature for thermal degradation assessment. However, initial studies showed that the fluorescence intensity seems to be insensitive to hydraulic fluid contamination. For this reason a temporal characteristic was analyzed for samples contaminated with aqueous phase of hydraulic fluid/water mixture resulting in three different pH levels (4, 3 and 2) of the solution. The hydraulic fluid used for surface contamination was Skydrol. Pulsed laser was used for samples surface excitation. The results for fluorescence decay time (t) indicate that the uncontaminated samples (REF) have the highest value of decay time. Two uncontaminated samples were measured and they gave comparable results (fig. 8). The presence of contamination causes a drop in the decay time for all cases (pH 2, 3 and 4). Moreover there is a decrease in decay time with the decrease of pH value (fig. 8). Taking into account the standard deviation of the measurements the pH 3 and 4 cases are comparable. The clearly different result (the lowest decay time) is observed for pH 2.

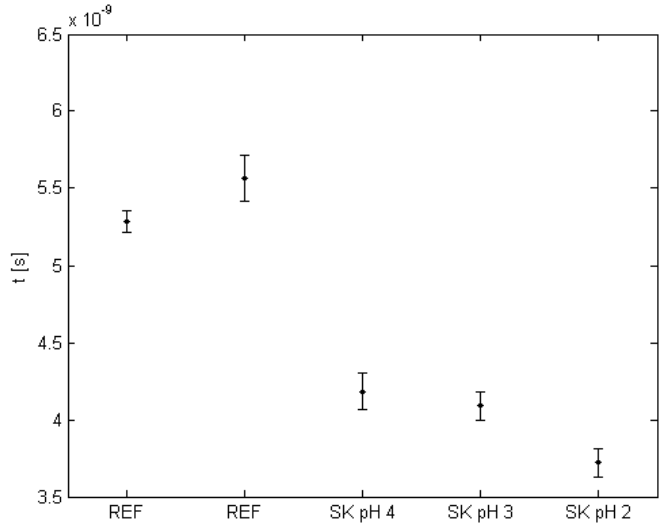


Fig. 8. Graphical presentation of fluorescence decay for varying level of Skydrol contamination

3. Conclusions

In the reported research LIF method was successful used for non-contact and non-destructive assessment of the CFRP surface. The LIF intensity is sensitive to the level of thermal degradation while the temporal characteristics is sensitive to surface contamination with hydraulic fluid/water solution. Presented results are promising and encourage for further development of the method and search for other areas of application in NDT of composite structures.

Acknowledgments

This work has been supported by the grant no. PBS1/B6/8/2012 (project KOMPNDT) of Polish National Centre for Research and Development (NCBIR).
The research leading to these results has received partial funding from the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 266226.
The measurements have been done using equipment of Photophysics Department headed by prof. G. Sliwinski and with his kind agreement.

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