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► **To cite this version:**

Jérôme Neauport, Jessica Fournier, P. Grua, Evelyne Fargin, Veronique Jubera, et al.. Luminescence study of defects in silica glasses under near-UV excitation. *Physics Procedia*, 2010, 8, pp.39-43. 10.1016/j.phpro.2010.10.009 . cea-01692824

**HAL Id: cea-01692824**

**<https://cea.hal.science/cea-01692824>**

Submitted on 25 Jan 2018

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VI Encuentro Franco-Español de Química y Física del Estado Sólido  
VI<sup>ème</sup> Rencontre Franco-Espagnole sur la Chimie et la Physique de l'État Solide

## Luminescence study of defects in silica glasses under near-UV excitation.

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### Abstract

In this paper, we report the results of photo-luminescence experiments on high purity silica glass. Two different types of defects located on optical pieces are investigated: indented sites and laser damage craters. Visible luminescence excited at the wavelength of 325 nm (3.81 eV) is observed using confocal microscopy. Each type of defect produces qualitatively different luminescence spectra. In the case of indented sites, the well known Oxygen Deficient Center (ODC) band peaking at 2.75 eV is clearly observed, while for laser damage areas, another classical luminescent object is found: the Non Bridging Oxygen Hole Center (NBOHC) which peaks at 1.9 eV. On both types of defects, the luminescence spectra yield a strong peak around 2.2 eV but its interpretation is still controversial.

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*Keywords* : Photoluminescence, Silica, Defects, Indentations, Laser damage.

### 1. Introduction

The Laser MegaJoule (LMJ) Facility is under construction at the CEA-CESTA in France. This facility will be used for inertial fusion studies. It consists in 176 laser beams focused at 355nm (3 $\omega$ ) on a few millimeter target. High purity silica optics is used to convey the laser beam in the UV. Fused silica optics have a propensity for surface laser damage in the typical fluence range starting around 10 J/cm<sup>2</sup> for 3 ns pulse duration, well below the intrinsic bulk damage threshold which is beyond 100 J/cm<sup>2</sup> for this pulse length. In this context, getting a better understanding of defect of silica is of much importance. In silica glasses, two luminescent defects are well known:

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the ODC (Oxygen Deficient Center) with an emission band at 2.75eV (450 nm) and the NBOHC (Non Bridging Oxygen Hole Center) with an emission band at 1.9 eV (650 nm) [1]. These two luminescent objects have been identified in our experiments, but such fact must be considered as anomalous, since the absorption spectra of ODC's and NBOHC's do not match our excitation wavelength of 325 nm [1]. Such defects are unlikely to trigger damage in the fluence range we consider. Other known defects can be absorbent but are not luminescent at this excitation wavelength.

In order to understand laser damage initiation; we study different types of defects: indentations and laser damage. The main feature of this experimental study is the appearance of a strong luminescence peak at 2.2 eV, not present for pristine material.

## 2. Sample preparation

Samples used in this study are high purity silica glasses (CORNING 7980) polished by SESO, of 50 mm diameter and 5 mm thick.

### 2.1. Indentations

The indentations are made with sphericoconical diamond tip of 10  $\mu\text{m}$  radius. Many parameters are adjustable: the final load  $F_f$  corresponding to the maximum load, the loading rate ( $v_1$ ) which is the speed when loading from  $F_i$  to  $F_f$ , and the time  $t_{F_{\text{max}}}$  during which we maintain the final load on the sample. Our experiments are performed with parameters given in table 1.

Table 1: Experimental parameters for indentations

Indenter radius	$F_f$	$v_1$	$t_{F_{\text{max}}}$
10 $\mu\text{m}$	1.5 N	2000 mN/s	60 s

Despite the fact that our experimental parameters are constant, resulting indentation sites are not equivalent from site to site as shown on Figure 1. We chose indentations shown on Figures 1b and 1c for our luminescence study. They are referred to as indentation 1 and indentation 2.

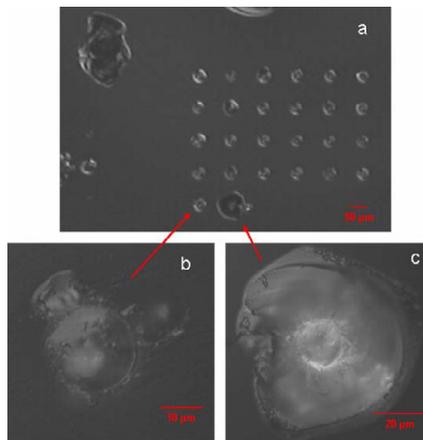


Figure 1. (a) Image of indentations matrix, (b) and (c) enlargement of the two analysed indentations.

2.2. Laser damage:

We create laser damage in our samples with a Nd:YAG laser, at 351 nm wavelength, 2.7 ns pulse duration, and a 6mm beam diameter. The laser fluence is about 40 J/cm<sup>2</sup>. For each damage site, we use a multi-shot technique (one to seven shots per site) in order to obtain different sizes of damages, as seen on Figure 2.

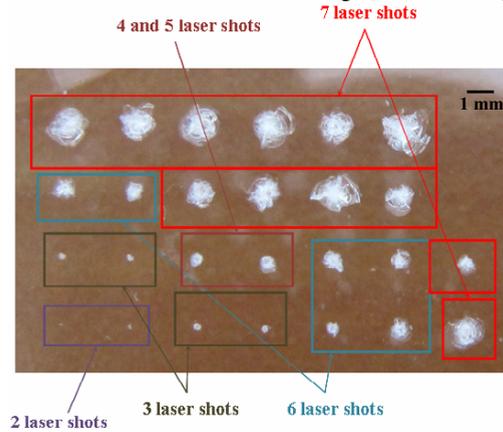


Figure 2. Image of laser damage matrix.

3. Experiments

Photoluminescence spectra are made on a LABRAM HR-800 spectrometer, a high resolution Raman spectrometer, permitting luminescence confocal microscopy. Luminescence is excited by a continuous laser at 325 nm (3.81 eV) at room temperature. The spatial resolution is about 1µm and the signal is collected with a CCD camera. We use a laser of 100 µW power, except for our reference sample: a silica sample without artificial surface defect, for which we operate at 1mW. The beam waste is controlled with a pinhole opened at 300µm. For all measurements, the obtained spectra are corrected by the equipment function.

4. Results

4.1. Indentations

The luminescence spectra (Figure 3a), obtained for the indentation 1 (Figure 1b) and the indentation 2 (Figure 1c), show different characteristics. The spectrum is shifted to lower energies when the indentation induces more fractures. In order to identify the defects, the spectra (Figure 3b) are decomposed using Gaussian curves. The parameters of the fit are given in Table 2.

Table 2: Parameters used for fitting luminescence spectra of the indentations

	Indentation 1		Indentation 2	
	Position (eV)	Width (eV)	Position (eV)	Width (eV)
1 <sup>st</sup> band	2.33	0.72	2.31	0.74
2 <sup>nd</sup> band	2.75	0.70	2.75	0.70

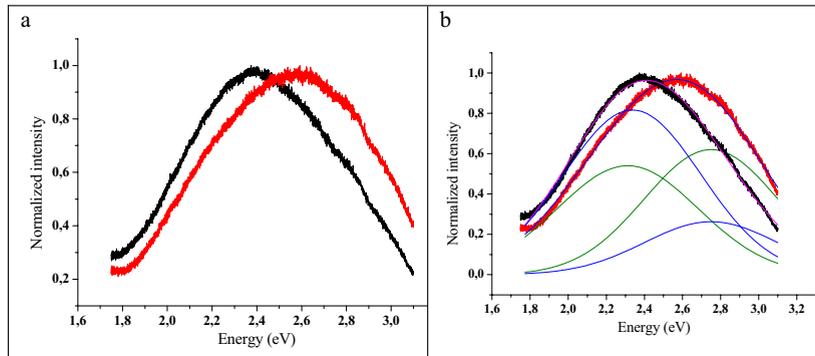


Figure 3. a) Comparison of photo luminescence spectra excited at 325 nm for indentations: indentation 1 (red) and indentation 2 (black); b) Comparison of fitted spectra for the two indentations: indentation 1 (green) and indentation 2 (blue).

The parameters used are in good agreement with those found in literature [2-3]. The spectra fits show that two defects are present in the indentations. The first one around the 2.3 eV band is not attributed to a known defect. The second band at 2.75 eV is attributed to the Oxygen Deficient Center [1].

#### 4.2. Laser damage

First results obtained on laser damage seem to indicate that the luminescence behaviour of defects is strongly correlated to the nature of damages (figure 4a). Strong differences are observed in the relative intensity of the emission bands. In all the cases, the emission bands can be fitted by three Gaussian curves (Figure 4b: only the Gaussian fit for the two laser shots is shown), parameters are given in Table 3. The corresponding curves are in good agreement with parameters found in literature [2-3]. Three luminescence bands are present in laser damages in different ratios. The first band at 1.89 eV corresponds to the NBOHC defect [1-5]. The second band at 2.25 eV is, to our knowledge, not related to a known defect. Finally, the third defect at 2.75 eV corresponds to ODC defect [1,2,4,5].

Table 3: Parameters used for fitting luminescence spectra of the laser damage craters

	2 laser shots		4 laser shots		5 laser shots	
	Position (eV)	Width (eV)	Position (eV)	Width (eV)	Position (eV)	Width (eV)
1 <sup>st</sup> band	1.89	0.15	1.89	0.15	1.89	0.15
2 <sup>nd</sup> band	2.26	0.41	2.25	0.42	2.24	0.34
3 <sup>rd</sup> band	2.75	0.40	2.75	0.40	2.75	0.40

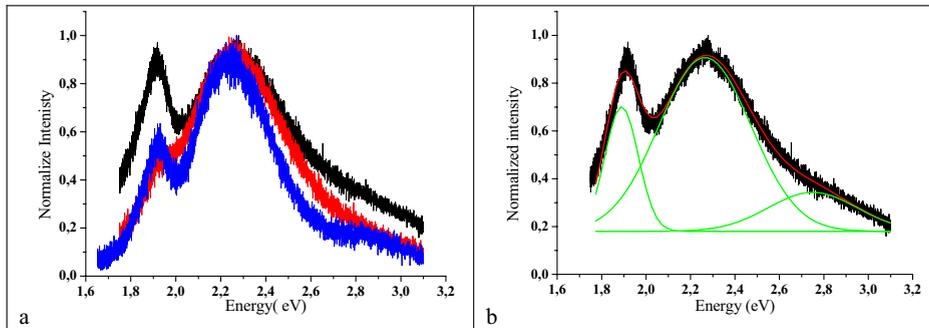


Figure 4. a) Comparison of luminescence spectra for 2 laser shots (black), 4 laser shots (red), and 5 laser shots (blue); b) Fitted luminescence spectra for two laser shots (black: experimental data, green: partial fits, red: final fit).

## 5. Discussion

We show that indentations and laser damage craters give rise to different spectra. In indentations, only two defects are present: ODC and an unknown defect emitting around 2.2 eV. The luminescence bands are also larger than for laser damage. In laser damage, there are three luminescence bands which are ODC, NBOHC and the unknown defect emitting at 2.2 eV. Concerning this defect, two hypotheses are mainly proposed. The first one concerns the Self Trapped Exciton [2,6]. This defect is strongly luminescent under 150 K but at room temperature it may still have a weak luminescence. The second hypothesis involves an  $E'_{\delta}$  center located in small silicon clusters [3-5], since a correlation between the Electron Paramagnetic Resonance signal of  $E'_{\delta}$  center and the luminescence band around 2.2-2.3 eV was evidenced from the study of  $\gamma$ -irradiated amorphous silicon.

## 6. Conclusion

This study shows that a luminescence is found in both cases studied (indentations and laser damage). In the two cases, ODC defect is found at 2.75 eV and an unknown defect around 2.2 eV. In laser damage a third defect is found at 1.89 eV and attributed to NBOHC defect. The 2.2 eV is not clearly ascribed now. New investigations are required in order to interpret accurately this 2.2-2.3 eV luminescence band.

## Acknowledgements

We would like to thank H. Bercegol for active contribution to these results, T. Cardinal and A. Garcia for fruitful discussions concerning luminescence spectroscopy, and T. Donval for assistance with laser damage accomplishment.

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