Nanosecond multiple pulse measurements and the different types of defects

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ABSTRACT

Laser damage measurements with multiple pulses at constant fluence (S-on-1 measurements) are of high practical importance for design and validation of high power photonic instruments. Using nanosecond lasers, it has been recognized long ago that single pulse laser damage is linked to fabrication related defects. Models describing the laser damage probability as the probability of encounter between the high fluence region of the laser beam and the fabrication related defects are thus widely used to analyze the measurements. Nanosecond S-on-1 tests often reveal the "fatigue effect", i.e. a decrease of the laser damage threshold with increasing pulse number. Most authors attribute this effect to cumulative material modifications operated by the first pulses.

In this paper we discuss the different situations that are observed upon nanosecond S-on-1 measurements of several different materials using different wavelengths and speak in particular about the defects involved in the laser damage mechanism. These defects may be fabrication-related or laser-induced, stable or evolutive, cumulative or of short lifetime. We will show that the type of defect that is dominating an S-on-1 experiment depends on the wavelength and the material under test and give examples from measurements of nonlinear optical crystals, fused silica and oxide mixture coatings.

Keywords: Nanosecond laser damage, S-on-1 damage tests, fatigue effect, statistical fatigue, material modification fatigue, short lived defects, fabrication defects, light induced defects.

1. INTRODUCTION

In order to make meaningful realistic laser damage tests that do not require huge budgets one usually chooses the S-on-1 protocol.[1] The S-on-1 protocol uses a high number of pulses of constant fluence for the irradiation mimicking thus operation of a typical high power photonics setup or instrument, which is expected to work with constant performance over a long time (and thus a large number of laser pulses). Like the 1-on-1 protocol,[2] the S-on-1 protocol is based on the measurement of damage probabilities P at different levels of peak fluence F_0 in the beam. The measurement, or estimation, of a probability is always based on a number of statistically independent repetitions of the same experiment. In the case of damage probability measurements we irradiate n sufficiently spaced sites on a sample and decide after inspection under a microscope how many of them, say k, were damaged. Using the maximum-likelihood principle and the numbers n and k of this stochastic experiment with binary outcome, we can then deduce the best hypothesis for the damage probability $P = k/n$ and its uncertainties ΔP^+ and ΔP^- corresponding to a chosen level of confidence.[3]

For multi pulse damage tests like the S-on-1 tests we also need to choose how many pulses we want to use in conditions where no damage is detected. This maximum pulse number to use on a single test site, S, will depend on the aim of the experiment: (i) If one only wants to know if the material shows a fatigue effect, a relatively small number of pulses can be sufficient (some hundreds of pulses). (ii) If the test is used to make a validation or to acquire data for an extrapolation of the damage threshold at high pulse numbers, the necessary pulse numbers are much higher.

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According to the ISO standard, the S-on-1 damage probability (for a maximum of S pulses per site) is given by the number of sites that damaged at pulse number S <u>or before</u> ($k = k(N_d \leq S)$). The S-on-1 damage probability is thus the complement of the survival probability of the sample if S laser pulses are used.

Using an online damage detection system, one can check for the appearance of damage after each laser pulse and thus record the damaging shot numbers N_d for all damaged sites. The most frequently used online damage detection systems are based on imaging of the irradiated site [ref] or on diffusion measurements [ref] but any pump-probe style measurement giving a response that allows to identify the occurrence of damage may be used. The damaging shot numbers N_d are very information-rich data, as they allow to retrieve any N-on-1 damage probability curve with N ranging from 1 to S. One obtains thus a two dimensional data set $P(F_0, N)$ that can be represented in different ways.

1.1. Representations of S-on-1 laser damage data and definition of the fatigue defect

For example one can plot all N-on-1 damage curves $P(F_0)$ with the parameter N changing between the curves (Figure 1a). This yields typically in a plot with many curves (N) each containing rather few points (Fluences).[4]

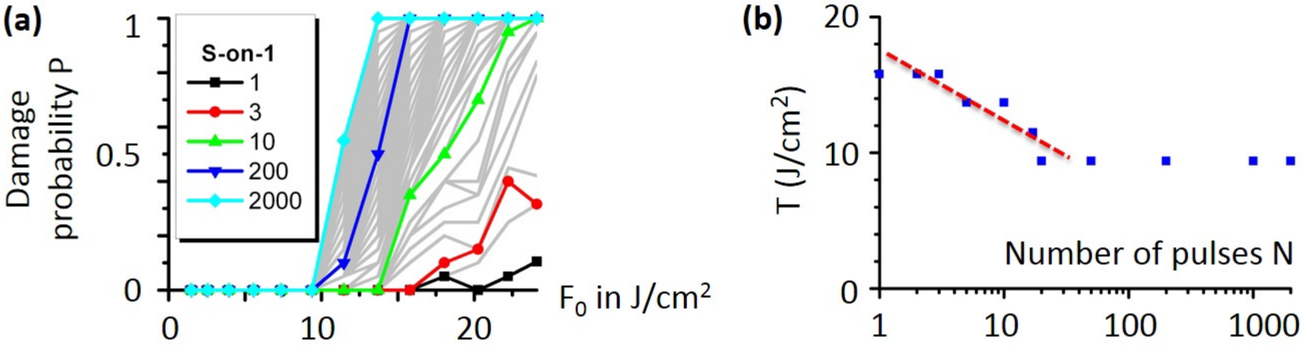

Figure 1: 2000-on-1 data in the bulk of KTP. (a) Full representation of the data set as 2000 $P(F)$ curves. The lowest curve (black squares) is the 1-on-1 curve. (b) Reduced representation of the data set. Only the experimental damage thresholds T are shown as function of the maximum used pulse numbers N. The initial decrease of the $T(N)$ curves indicates a strong fatigue effect. (16 J/cm² for 1-on-1; 9J/cm² for 50-on-1 (= 56% of the 1-on-1 value).

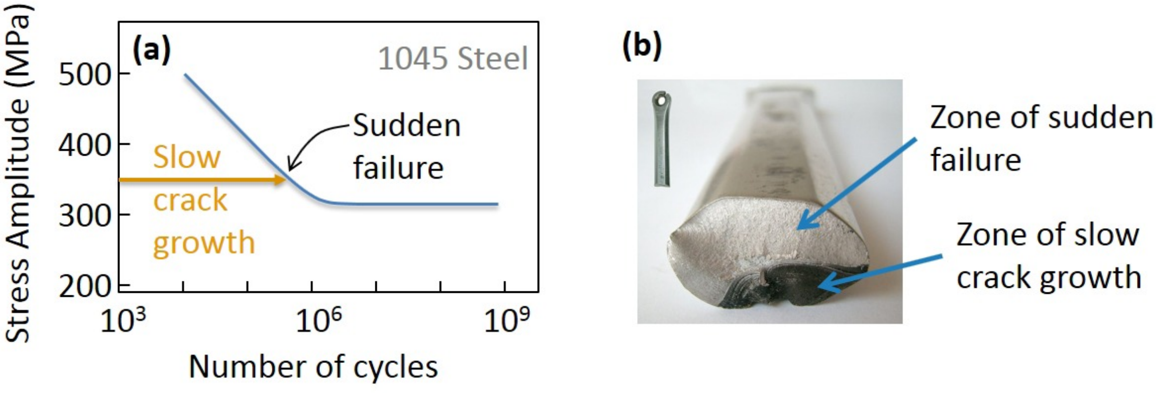

Figure 2: Information on mechanical fatigue damage. (a) Mechanical fatigue damage in Steel.[6] A chosen stress amplitude is applied and removed for a high number of cycles. Doing the first cycles a small crack is initiated. The crack then grows slowly until the work piece beaks suddenly. (b) Photograph of an aluminum part that was subject to fatigue failure.[7]

As this plot is usually difficult to read. One often finds a reduced representation that plots the N-on-1 damage thresholds T (the highest fluence for which still $P = 0$) as a function of the pulse number N (Figure 1b). It is this plot that defines the "fatigue effect": if $T(N)$ decreases, one says that there is a fatigue effect.[5] The name of the effect has been chosen in analogy with mechanics, where a metallic work piece that is charged and uncharged repeatedly can break after many cycles, even when a load that is much smaller than the load it can withstand for a single loading/unloading cycle (Figure 2).

Two more, very helpful representations of S-on-1 data exist: (i) Plotting a P(N) curve for each fluence yields some curves with many points that can be used to evaluate if statistical fatigue is present and, if this is the case, one can extract the single-pulse damage probability $p_1(F_0)$ by fitting this plot (Figure 3a). (ii) Marking each damaged site in the N_d - F_0 -plane yields a scatter plot that is a reduced representation of the data (it does not contain the undamaged sites), but that nevertheless allows to estimate which kind of fatigue is present in the sample and yields also information on simultaneous failure modes (Figure 3b).

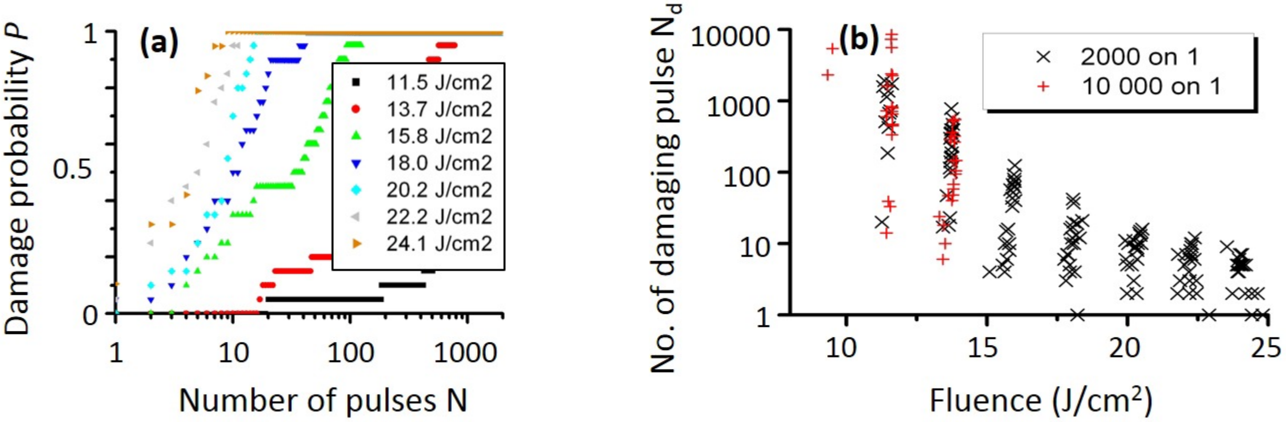

Figure 3: Two more representation of the same dataset as in Figure 1. (a) Full representation of the data set as 7 $P(N)$-curves, one for each Fluence. (b) Reduced representation of the data set. Only damaged sites are shown. Each damaged site is represented by a point in the N_d-F-plane. (Additionally data of a 10 000-on-1 test has been added.

1.2. Precursor encounter models for single pulse nanosecond laser damage

Nanosecond laser damage is generally admitted to be caused by fabrication defects in the optical material. The idea came up after studying the morphology of laser damages close to the threshold [ref laurent et Feit] and comparing the electric field strength at the laser damage threshold to the electric field strength at dielectric breakdown. The idea is now widely accepted since models that hypothesize the laser damage probability to be the probability to find a damage precursor in the high fluence region of the laser beam succeed to describe the dependency of 1-on-1 laser damage tests on the laser beam size.[8,9] Several variations of these "precursor encounter models" exist that differ mainly in the shape of the statistical distribution function $g(T)$ describing the distribution of the precursor thresholds in the ensemble.[9–11] All of them consider a homogeneous distribution of the damage precursors in the host material and suppose a deterministic reaction (the occurrence of damage) if a precursor is irradiated at a fluence exceeding its threshold.

$$P(F_0) = 1 - \exp\left(-\int_0^{F_0} g(T)\, A\left(\frac{T}{F_0}\right) dT \right) \tag{1}$$

Equation 1 gives the general expression of $P(F_0)$ for these models. $A(T/F_0)$ is the surface area that is irradiated by a fluence exceeding the threshold T if the peak fluence of the beam is F_0. In particular, for a spatially Gaussian test beam one obtains:

$$A\left(\frac{T}{F_0}\right)=\begin{cases} 0 & ,\text{if } \frac{T}{F_0}>1 \\ -\frac{\pi}{8}d^2\ln\left(\frac{T}{F_0}\right) & ,\text{if } \frac{T}{F_0}\leq 1 \end{cases} \quad (2)$$

With d the $1/e^2$ diameter of the spatially Gaussian laser beam.

2. MATERIAL MODIFICATION FATIGUE – THE ANALOGY TO MECHANICS

In mechanics, the mechanism causing the fatigue effect was readily discovered: During the first cycles a small crack forms and grows slowly until the work piece is weak enough to suddenly break during the last and damaging cycle. In laser damage, the most widely spread hypothesis follows a similar argumentation line.[5] One suspects that the first pulses (the incubation pulses) cause cumulative material modifications that "weaken" the material up to a state where it can be broken by the last and damaging laser pulse. However, most of the time this hypothesis is proposed solely based on decreasing $T(N)$, i.e. also in cases where no direct evidence of cumulative material modifications during the incubation pulses is presented. Even if experimental evidence for cumulative material modifications is provided,[12] the precise physical mechanisms that finally lead to damage are not easy to determine in the case of laser induced damage and determining them usually needs long and detailed investigations. Two groups of references should be mentioned in this context (refs[13,14] and references therein), but others have also been reviewed.[5]

3. MODELS FOR FATIGUE LASER INDUCED DAMAGE

The successful single-pulse damage models for the nanosecond regime encourage us to model the optical material under test as a host material with homogeneously distributed damage precursors. We will ground the following analysis of possible models for fatigue laser induced damage on this image of the optical material.

3.1. Precursor encounter models and S-on-1 tests

First of all we might ask ourselves if we cannot apply the precursor encounter models to multi pulse irradiation too. The answer is no, because all precursor encounter models are based on the assumption of homogeneously distributed damage precursors in the optical material. During multi pulse irradiation however, all pulses except the first will interact with a sample that has been modified locally by the preceding pulses. Additionally, these models understand the laser damage probability as the probability of encounter between the high fluence region of the laser beam and a damage precursor. During multi pulse irradiation however, the probability of encounter between the pulses and the region that has been modified by the preceding pulses is 100% (if laser fluctuations are neglected).

Thus, two fundamental assumptions of the precursor encounter models are no longer valid in a multiple pulse experiment with material modification and in consequence the model should not be applied "as is" to the analysis of S-on-1 data.

Nevertheless the precursor encounter models predict an increasing damage probability if we consider the fluctuations of pulse energy and focal point position in a sample that is not modified by the laser pulses.[4,15] In this case the sample stays homogeneous (the model stays valid) and the surface over threshold probed up to pulse number N, $A(T/F_0, N)$, increases with increasing N. According to this model, the damage probability increases significantly with increasing N if the peak fluence in the beam F_0 is close to the damage precursor threshold T_P. It however fails to explain the experimentally observed increase in damage probability for fluences F_0 that clearly exceed T_P.[4]

We thus need to look for other models describing S-on-1 laser damage.

3.2. Cumulative material modifications

Considering the composition of the sample (a host material containing damage precursors) and the idea that cumulative material modifications lead to S-on-1 damage, we should distinguish two cases. Either the damage precursors get worse, or the host material is modified and becomes more fragile than the damage precursors.

3.2.1. Modification (worsening) of fabrication defects

Concerning the first case, we should try to figure out how an existing damage precursor would react when being irradiated by laser pulses that are sufficiently strong to modify it, but that do not exceed the threshold where the interaction would damage the host material.

Limiting this discussion to damage precursors in the bulk, the main 'suspects' of being laser damage precursors are submicronic metallic inclusions,[16] submicronic inclusions of non-stoichiometric dielectrics,[17] and clusters of point defects (like color centers).[18] Some of these defects are accompanied by strain in their vicinity, which could lead to mechanical failure.[13]

As the irradiation definitely has an effect on the material, it either generates electron-hole pairs (if absorbed in a dielectric) or/and causes heating of the conduction band electrons (if absorbed in a metallic inclusion). Most of these conduction band electrons will relax non-radiatively generating 'gentle' heating. We speak about 'gentle' heating, in the sense that no laser damage is caused by the pulse we are looking at. (Extreme heating would lead to thermal run-away [ref] or strong thermal stresses, which would lead to damage during this pulse.) Gentle heating, which is not triggering damage, will either be without any influence to the material or favor diffusion (of metallic inclusions[19]), annealing of point defects[20] and resorption of frozen stresses. In one word, the thermal compound of the relaxation rather leads to 'laser annealing' than worsening of the damage precursors.

The only mechanism that may worsen existing damage precursors concerns point defects. Point defects generate stresses in their vicinity and stresses favor the generation of point defects during the relaxation of electron-hole pairs. Additionally, point defects easily provide conduction band electrons and the presence of conduction band electrons early in the laser pulse will favor absorption and thus laser damage.

Hence, for defect-clusters, a worsening by the incubation pulses cannot be completely excluded but a well-studied crystal, KDP, gives a counterexample: Damage precursors in KDP are believed to be defects clusters,[18,21] however KDP shows strong laser conditioning.[22] So defects clusters too do not always worsen during pre-irradiation with non-damaging fluences.

In summary, it seems probable that fabrication defects (the single-pulse damage precursors) do not worsen during the incubation pulses of an S-on-1 test.

3.2.2. Creation of new defects in the host material

In the second case we suppose that new defects are created in the host material and that these new defects cause multi-pulse laser damage at fluence levels lower than the 1-on-1 threshold.

The creation of new defects by low fluence irradiation has been reported in several different materials[5,12,14,20,23,24] especially if the photon energy of the laser approached the band gap of the material. But, even if experimental evidence for cumulative material modifications is found, the link of these modifications with fatigue laser damage may be quite indirect[12] and thus rather difficult to prove.

Recent work on fatigue laser damage in synthetic fused silica using UV wavelengths indicates the importance of self-focusing for the damage mechanism.[25] Similarly, one of the most complete investigations on nanosecond laser damage also includes self-focusing as the final step in their model.[14] More generally, the light-induced modifications of the material may in fact modify the propagation of the laser beam by different physical mechanisms (modification of the refractive index, thermal self-focusing, nonlinear self-focusing) and thus cause a catastrophic enhancement of the peak fluence in the material. For lasers with spatially Gaussian beams and good positional stability, the signature of these mechanisms is an influence of the beam diameter on the fatigue strength, i.e. the relative threshold decrease observed at a certain number of pulses (for example T(1000)/T(1)).[14]

Material modification fatigue as discussed here above is thus most likely to be caused by generation of new defects in the host material. These defects have long lifetimes and accumulate pulse after pulse until the effects they have on the laser beam become sufficiently strong to cause damage. They may cause focusing of the beam and cause damage downstream or absorb themselves a sufficient portion of the light to induce thermal run-away and damage. Preexisting single-pulse damage precursors are probably not directly responsible for fatigue laser damage.

3.3. Statistical fatigue

However not all experiments for which a fatigue effect was observed can be understood by cumulative laser-induced material modifications (Figure 4). The presence of a strong fatigue effect at 1064 nm and the absence of fatigue at 532 nm or 355 nm would mean that the material modification are caused by IR photons but not by 532nm-photons or 355nm-photons, which is difficult to conceive.

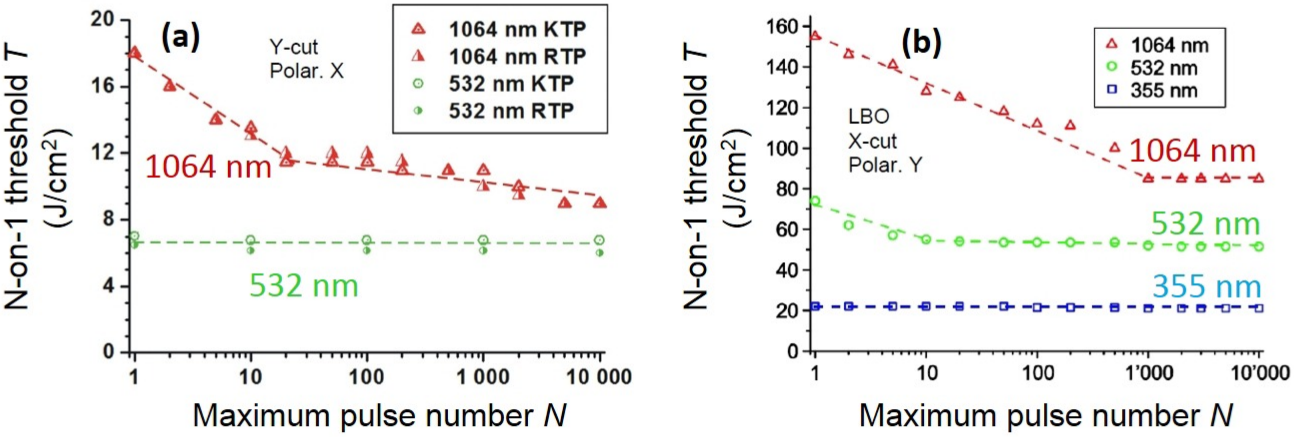

Figure 4: *T*(*N*) data obtained at different test wavelengths in the bulk of nonlinear optical crystals. (a) KTP and RTP at 1064 nm and 532 nm. (b) LBO at 1064 nm, 532 nm and 355 nm. In all three materials the fatigue effects disappears at shorter wavelength which is contradictory to light-induced, cumulative material modifications causing the fatigue effect.

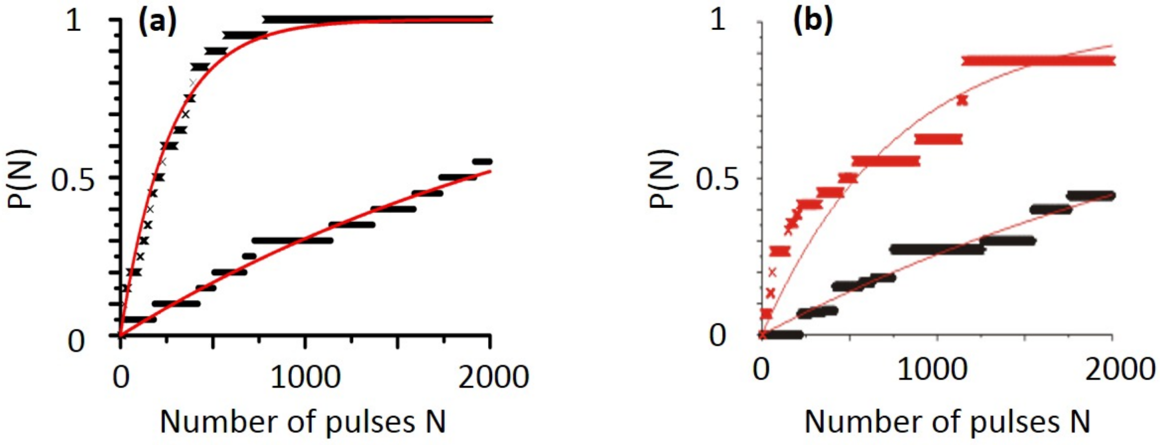

Figure 5: *P*(*N*) data obtained in the bulk of KTP and the corresponding fits using the statistical fatigue model.
(a) Measurement with a multiple longitudinal mode (MLM) laser. (b) Measurement with a single longitudinal mode (SLM) laser.

The situation can be understood if one assumes that there exists a single pulse damage probability p_1 at 1064 nm, and that the interaction becomes deterministic for shorter wavelengths. In case of statistical interaction, the single pulse damage probability p_1 is independent of the pulse number N, meaning that there are no material modifications. An S-on-1 damage experiment can then be understood as statistically independent resampling and the $P(N)$ curves can be modeled by the simple formula $P(N) = 1 - (1 - p_1)^N$ (Figure 5a).[26]

The stable single pulse damage probability may possibly be caused by the laser statistics of multi longitudinal mode (MLM) lasers or it may be caused by the light matter interaction itself.[27] Experimental data with validated single longitudinal mode (SLM) operation (each pulse has to be SLM) is still a bit rare (Figure 5b), but statistical fatigue seems to be very frequent for IR experiments.[26–28] By the way, this statistical model has first been proposed in the early days of laser damage research by Bass *et al.*[29] and has been abandoned later on (for a discussion see refs[4,5]).

In terms of defects, the constant single pulse damage probability can be understood as a consequence of the light-induced formation of defects that may cause damage with a certain probability, but that are of short lifetime, so that the material is indistinguishable from the pristine material before the next laser pulse.

4. DISTINGUISHING THE DIFFERENT TYPES OF FATIGUE

The $P(N)$-curves for statistical fatigue and material modification fatigue have completely different shapes, so that it is simple to distinguish the two processes by observing the $P(N)$ data. For statistical fatigue, the $P(N)$-curves start steep and flatten gradually (Figure 6b). For material modification fatigue caused by an ideal laser (and neglecting any single pulse damage), the damage probability is zero as long as the material modifications did not reach the critical point. For a perfectly stable laser and material modifications that are operated in the host material, this critical modification will be reached after the same number of pulses for all tested sites (critical pulse number). The damage probability $P(N)$ thus switches suddenly from 0 to 1 when exceeding the critical pulse number (Figure 6c).[30] If no fatigue effect is present, all sites either damage at the first pulse or do not damage at all and the corresponding $P(N)$ curves will be horizontal lines (Figure 6a).

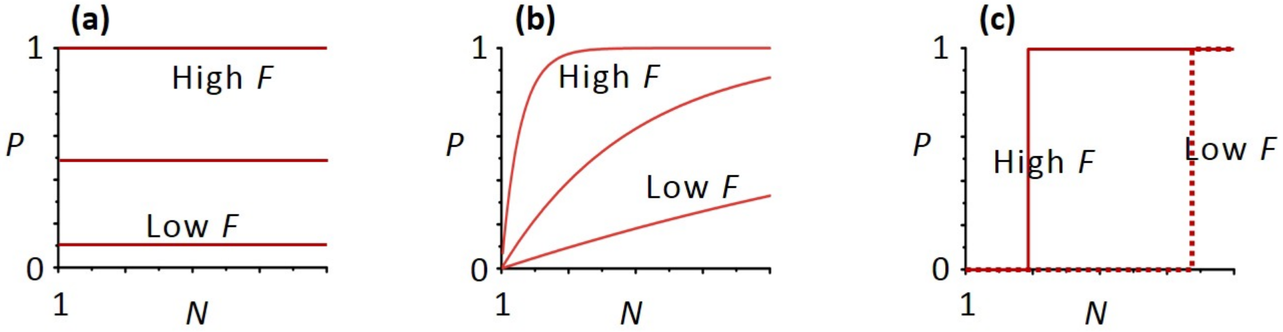

Figure 6: Theoretical $P(N)$-curves for: (a) a sample without fatigue effect; (b) a sample with statistical fatigue and (c) a sample in which cumulative material modifications are at the origin of the fatigue effect.

Simply plotting all damaged sites in the Nd - F0 - plane represents a simpler and faster (but less quantitative) means to distinguish the different situations: For the deterministic material modification fatigue all points form a well-defined curve (Figure 7c). In the absence of fatigue all points are concentrated on the first pulse numbers (Figure 7a) and the gradual increase of the damage probability with the number of pulses during statistical fatigue translates to a scattering of the N_d values over typically more than one decade (Figure 7b).

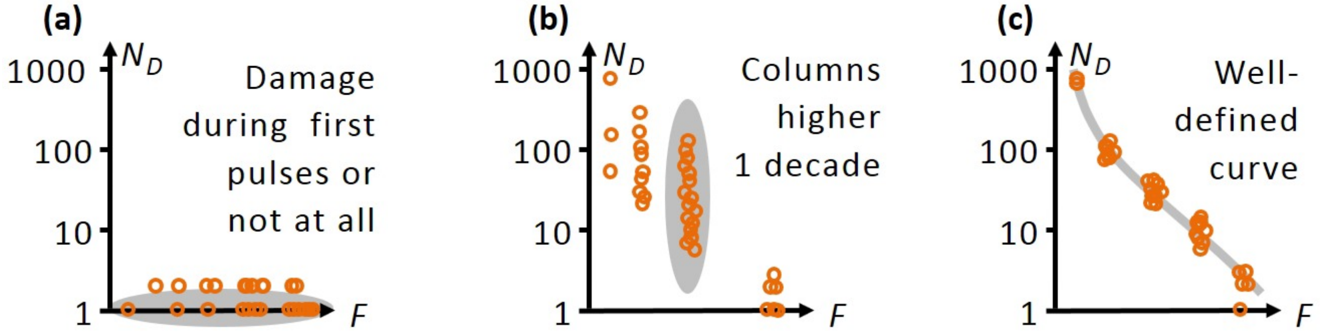

Figure 7: Theoretical N_d(F)-curves for: (a) a sample without fatigue effect; (b) a sample with statistical fatigue and (c) a sample in which cumulative material modifications are at the origin of the fatigue effect.

5. THE BEAM SIZE EFFECT

Data acquired in the UV on thin films with different beam sizes clearly shows that no fatigue effect is observed with sufficiently large laser beams.[30] This is understandable in view of the above discussion. If the laser beam diameter is of the same size as the mean distance between fabrication defects, some sites will always damage during the first laser pulses because the probability of encounter between the beam and the fabrication defects is high. Depending on the irradiation conditions the few sites without fabrication defects may damage later but this does not change the threshold of the measurement that is determined by the fabrication defects.

6. THE WAVELENGTH EFFECT

Measurements at different wavelengths show that for longer wavelengths, where the laser-matter interaction is weak, statistical fatigue is obtained whereas material modification fatigue is obtained for shorter wavelengths and thus stronger and more deterministic laser-matter interaction. An example for this behaviour is shown in Figure 8 where Suprasil I® has been tested at 1064 nm and 355 nm. Plotting the *P*(*N*) curves clearly shows the differences in the fatigue mechanisms dominating the observations in the IR or the UV. We should mention here that both measurements were done with (different) MLM lasers. The laser fluctuations were especially high for the UV laser[25] inducing an enlarged transition from $P = 0$ to $P = 1$ (Figure 8b). The signature of the cumulative material modification fatigue is anyway easily recognizable. Thus this measurement makes also clear that even strong laser fluctuations do not cause statistical fatigue if cumulative material modifications appear with the given wavelength in the sample under test. Similar conclusions can be drawn from measurements in thin films.[30]

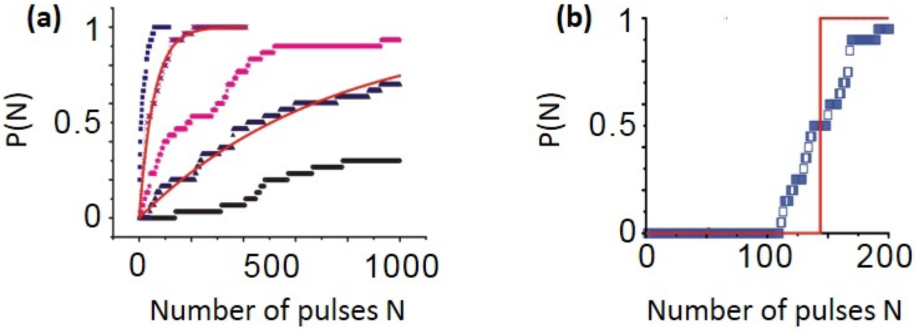

Figure 8: S-on-1 tests in the bulk of high-OH synthetic fused silica (Suprasil® 1). (a) For a test at 1064 nm and (b) for a test at 355 nm. In the IR statistical fatigue is observed and in the UV material modification fatigue is observed.

7. SIMULTANEOUS FAILURE MODES

An advantage of the $N_d(F)$ scatter plots over the $P(N)$-plots is that the first easily allow to recognize the simultaneous presence of different failure modes. An example is given in Figure 9 where an ion beam sputtered silica layer has been tested at 266 nm using a beam diameter of 30 µm.[30] At the given beam diameter one always encounters fabrication defects that damage during the first pulses. These fabrication defects determine the S-on-1 damage thresholds so that in terms of the $T(N)$-curve one would say that there is no fatigue effect. The sites that do not contain fabrication defects however show that material modification fatigue is simultaneously present in this sample.

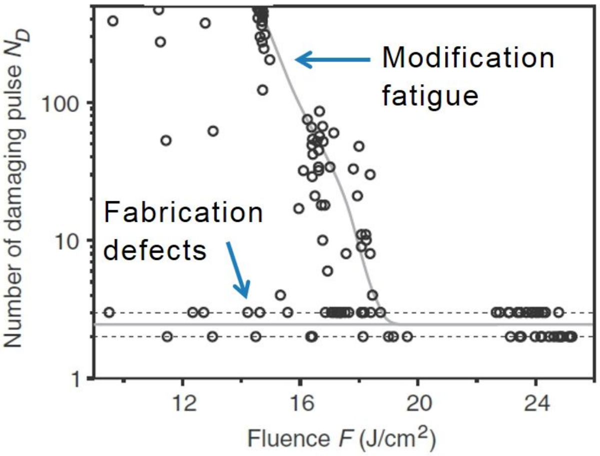

Figure 9: 500-on-1 test of an ion beam sputtered silica layer at 266 nm..

Considering that laser conditioning affects the fabrication defects, one could say that 'fatigue' and 'conditioning' may be present in one and the same sample as these two effects are based on two different types of defects and two different types of physical mechanisms leading to damage.

8. SUMMARY AND CONCLUSIONS

In summary, we provided an overview of nanosecond multiple pulse measurements and discussed different empirical models for these experiments. We concluded that precursor encounter models should not be used to describe S-on-1 measurements showing material modification fatigue. The light-induced material modifications that finally lead to damage are most probably located in the host material and not in the pre-existing fabrication defects. The defects leading to material modification fatigue are light-induced and of long life-time so that they accumulate during the incubation pulses. We also showed that for weak laser-matter interaction, like it is found at IR wavelengths, statistical fatigue is frequently observed. The defects associated with statistical fatigue are light-induced and of short lifetime so that they do not accumulate.

In order to distinguish between material modification fatigue and statistical fatigue one should plot the damage probability P as function of maximum pulse number N (or S). For statistical fatigue these data are well described by the statistical model and for material modification fatigue the incubation pulses (with $P = 0$) are followed by a quick transition from $P = 0$ to $P = 1$. It is also possible to distinguish material modification fatigue and statistical fatigue from $N_d(F)$ scatter plots of the damaged sites, as the N_d values obtained upon statistical fatigue are distributed over at least a decade whereas they are more concentrated (ideally deterministic) for material modification fatigue.

$N_d(F)$ scatter plots of the damaged sites also allow us to quickly understand if there are two parallel failure modes. For example for some sites fabrication defects may cause damage during the first pulses and for the sites without fabrication defects intrinsic light-host material interaction may cause material modification fatigue causing damage at higher pulse numbers.

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