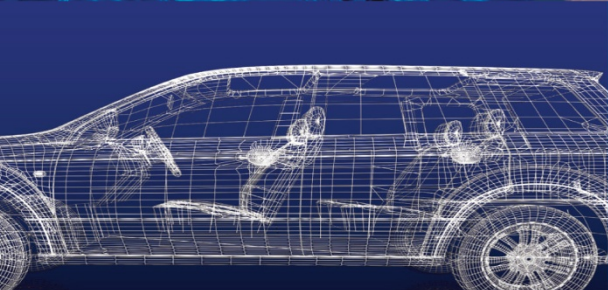
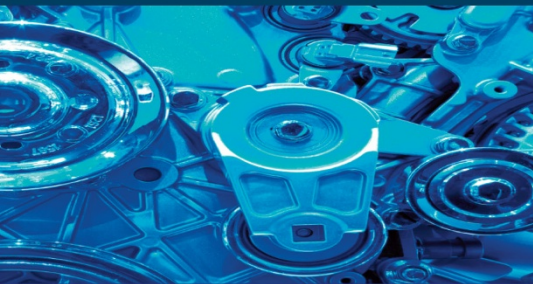


MECHANICAL ENGINEERING AND SOLID MECHANICS SERIES

RELIABILITY OF MULTIPHYSICAL SYSTEMS SET



Volume 2

# Nanometer-scale Defect Detection Using Polarized Light

Pierre Richard Dahoo  
Philippe Pougnet  
Abdelkhalak El Hami

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## Nanometer-scale Defect Detection Using Polarized Light



**Reliability of Multiphysical Systems Set**

coordinated by  
Abdelkhalak El Hami

Volume 2

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Library of Congress Control Number: 2016943672

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British Library Cataloguing-in-Publication Data  
A CIP record for this book is available from the British Library  
ISBN 978-1-84821-936-6

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## Preface

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The various actions decided on at a global level to stimulate sustainable development and to respond to climate issues bring forth increasingly stringent regulations in terms of greenhouse gas emissions and hazardous substances. In the automotive sector these regulations drive industrial companies to develop new mechatronic systems using electricity to replace the various mechanical functions of vehicles. International competition and constant pressure to improve the performance of innovative products compel the companies supplying embedded mechatronic devices to innovate in increasingly shorter lead times to remain competitive.

To improve the performance of embedded systems in terms of volume or mass reduction, or to reduce energy losses, the mechatronic industry implements new packaging methods (such as those based on multimaterials) or incorporates new materials (for instance, carbon nanotubes). Modeling and simulation are used to limit cost, increase durability and reduce lead time to market. The Physics of failure provides the knowledge to predict and reduce potential failures in application and optimize design before activating serial production. In this respect, Reliability Based Design Optimization (RBDO) is a numerical tool used to optimize design and reduce industrial fabrication risks. This approach can only be applied efficiently when the underlying physical phenomena are thoroughly understood and

when the models used accurately represent the conditions under which the device operates.

To model a dynamic system consisting of interacting sub-parts, a simplified system behavior model based on realistic hypotheses and key parameters is first used. Dynamic behavior is controlled by Partial Differential Equations (PDE) based on the characteristics of the system. By incorporating elements or parameters that were initially not included and by improving the PDE (for instance by taking into account non linearities or novel coupling schemes ...) this model is extended and improved leading to an increasingly precise simulation of the real functioning behavior, as used in the process like approach.

Theoretical models are usually built following an analysis of the complex system which leads to equations based on fundamental laws from the bottom-up. Consequences are deduced from realistic hypotheses and known physical laws. Either analytical or digital methods are applied to solve the equations. Whenever possible, experiments are conducted to compare expected results and real data. A top-down approach can also be applied using experimental methods. This approach is based on data obtained by applying specific stresses or external constraints, and from the study of the system response. Data from these tests are compared to simulation results from theoretical or empirical models. Both bottom-up and top-down approaches can lead to some uncertainties in data analysis. This can be evaluated through statistical analysis which provides predictions and margins of error. The objective is to reduce the margin of error in order to obtain realistic predictions and to better understand the properties of active materials.

This book describes experimental and theoretical methods which are developed in fundamental research to better understand the physical chemistry and physical processes in complex systems and which, on the nanometric scale, are the root cause of the outstanding properties of the materials used in innovative technological devices. It presents optical techniques based on polarized light which can be applied to detect material or interface defects which have an impact on their performance. It also describes how to measure the mechanical

properties of nanomaterials and how to analyze experimental data taking into account the range of uncertainties using theoretical models.

This book is written for students at Master and Doctoral levels, teaching academics and researchers in Materials Science and Experimental Studies, as well as engineers and technical staff from industrial sectors involved in systems where embedded electronics, mechatronics and electronic and optical materials are employed.

Chapter 1 describes various approaches which take into account uncertainties and are applied to analyze the static and dynamic behavior of systems and structures. Chapter 2 presents an approach to optimizing the design of a system which matches design cost with the guarantee of functioning without failure in the planned use conditions. This approach is based on taking into account uncertainties and on simultaneously solving two problems: optimizing the production cost of the structures performing the expected functions and ensuring an acceptable probability to fulfill its function. Chapters 3 and 4 give an overview of the classical and quantum theories of light as well as the various methods established to describe the polarization state of light.

Chapter 5 reviews theories on the interaction of light and matter and various condensed phase materials used in industrial applications. The notion of incomplete information about a quantum system is presented using the density matrix to take into account the problem of the interaction of the quantum system with the environment. Chapter 6 describes lasers, sources of polarized light and the experimental methods based on lasers to study either bulk materials using Laser Induced Fluorescence and IR-IR Double Resonance techniques, or the surface of materials using techniques to analyze the reflexion of a probe over the ultrasonic waves created by a pump laser. These methods make it possible to discriminate the different paths through which energy dissipates in materials when defects are present. This approach is used to build theoretical models to understand and analyze the thermal effects in composite materials.

Chapter 7 describes how to apply these methods to model systems before describing the apparatus used to prepare the systems composed of molecules which are trapped at low temperature in a solid matrix (rare gases or nitrogen). The various lasers and infrared detectors used in Laser Induced Fluorescence and Double Resonance techniques are presented. The results obtained on O<sub>3</sub>-GR, CO<sub>2</sub>-GR, and N<sub>2</sub>O-GR systems are analyzed using theoretical models developed to determine the energy relaxation rate constants according to the various paths through which a system may transfer energy. Predictions and extrapolations applying the results of the highlighted transfer mechanisms to other systems are proposed.

Chapter 8 describes the study of the interfaces of assembled materials using the IR spectroscopic ellipsometry technique. This technique is summarized as well as the necessary equipment and the analysis process, which is based on an inverse method applied to the models describing the interaction of light and matter through optimization algorithms. The results obtained on various types of interfaces found in the assembly of mechatronic power devices are presented and discussed. The ellipsometry technique is used to determine the possible modifications that occur in the properties of the materials when they come into contact as a result of physical or physical-chemical processes, as well as to follow the evolution of interfaces as a function of temperature in a dry or humid atmosphere.

Chapter 9 describes how to determine the properties of carbon nanotubes by applying the RBDO approach which correlates theoretical models and statistical methods to characterization and fabrication methods.

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Philippe POUINET  
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June 2016



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# Uncertainties

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Taking into account uncertainty in the design process is an innovative approach. This includes dimensioning the structure of the systems, the use of safety coefficients and the most advanced techniques to calculate reliability. The aim is to design a system that statistically achieves the best performance since the system is subject to variations. For a given risk probability, satisfactory system performance can be targeted which has low sensitivity to uncertainties and respects a minimum performance threshold. From a mathematical point of view, an innovative approach to system design can be considered as an optimization problem under constraints. In this chapter, various methods are presented to calculate systems subject to uncertainties.

## 1.1. Introduction

The methods used to take uncertainties into account are mathematical and statistical tools that make it possible to model and analyze systems whose parameters or use conditions are likely to vary. These methods are used to optimize the design and to balance cost and performance.

These methods are based on:

- the development of an approximate mathematical model of the physical system under study;
- the identification and characterization of the sources of uncertainty in the model parameters;

– the study of the propagation of these uncertainties and their impact on the output signal (response) of the system.

Analysis and estimation of the statistics (moments, distribution parameters, etc.) of the system response are performed in the next step. The methods used to analyze the propagation of uncertainties vary according to the mathematical tools on which they are based. These methods include a reliability based design approach, a probabilistic approach based on design of experiments, and a set based approach.

### 1.2. The reliability based design approach

The reliability based design approach is based on modeling uncertainties. Depending on the methods used, uncertainties are modeled by random variables, stochastic fields or stochastic processes. These methods make it possible to study and analyze the variability of a system response and to minimize its variability.

The most common methods are the Monte Carlo (MC) method, perturbation method and polynomial chaos method [ELH 13].

#### 1.2.1. The MC method

##### 1.2.1.1. Origin

The first use of this mathematical tool dates back to Fermi's research on the characterization of new molecules in 1930. The MC method has been applied, since 1940, by Von Neumann *et al.* to perform simulations in the field of atomic physics. The MC method is a powerful and very general mathematical tool. Its field of applications has widened because of the processing power of today's computers.

##### 1.2.1.2. Principle

The MC method is a calculation technique which proceeds by successively solving a determinist system equation in which uncertain parameters are modeled by random variables.

The MC method is used when the problem under study is too complex to solve by using an analytical resolution method. It generates random draws for all uncertain parameters in accordance with their probability distribution laws. The precision of the random generators is very important because for each draw a deterministic calculation is performed using the number of parameters defined by this generator.

### ***1.2.1.3. Advantages and disadvantages***

The main advantage of the MC method is that it can be very easily implemented. Potentially, this method can be applied to any system, whatever their dimensions or complexity. The results obtained by this method are exact in a statistical sense, that is their uncertainty decreases as the number of draws increases. This uncertainty of precision for a given confidence level is defined by the Bienaymé–Chebyshev inequality. A reasonable precision requires a large number of draws. This sometimes makes the MC method very costly in terms of calculation time, which is the main disadvantage of this method.

### ***1.2.1.4. Remark***

The simplicity of the MC method has made its application popular in the field of engineering sciences. This is a powerful but costly method. Its results are often used to validate new methods that are developed in the framework of fundamental research. It is applied in Chapter 9 in order to characterize carbon nanotubes.

## ***1.2.2. The perturbation method***

### ***1.2.2.1. Principle***

The perturbation method is another technique used to study the propagation of uncertainties in systems [KLE 92, ELH 13]. It consists of approximating the random variable functions by their Taylor expansion around their mean value. According to the order of the Taylor expansion, the method is described as being the first, second or

nth order. The conditions of existence and validity of the Taylor expansion limits the scope of this method to cases where the random variables have a narrow dispersion around their mean value [ELH 13, GUE 15a].

With the perturbation method, the random functions in the expression of the model's response to input parameters are replaced by their Taylor expansions. Terms of the same order are grouped together and, as a result, a system of equations is generated. The resolution is then carried for each order, starting with the zeroth order. The mathematical formalism as well as the general equations for the resolution can be found in the books by El Hami and Radi [ELH 13] and Guerine *et al.* [GUE 15b].

### 1.2.2.2. Applications

There are many applications of the perturbation method. This method makes it possible to study the propagation of uncertainties in static and dynamic systems as well as in linear and nonlinear systems. However, it provides precise results only when the uncertain parameters have a low dispersion [ELH 13, GUE 15a].

Guerine *et al.* [GUE 15b] have used the perturbation method in order to study the aerodynamic properties of elastic structures (stacked flat) subject to several uncertain parameters (structural and geometrical parameters) in the field of modeling and analysis of the vibratory and dynamic behaviors of systems. This work is the first published application of the stochastic finite element method (FEM) combined with the perturbation method for the analysis of aerodynamic stability.

In another study, El Hami and Radi [ELH 13] combine the finite difference method and the perturbation method to model vibration problems in uncertain mechanical structures. This method is used, for example, to determine the probabilistic moments of eigen frequencies and eigen modes of a beam in which the Young modulus varies randomly.

The second order is usually sufficient to determine the first two moments with good precision. In [MUS 99], Muscolino presents a

dynamic analysis method for linear systems with uncertain parameters and deterministic excitations. This method improves the first-order perturbation method, which is limited when the dispersion of uncertain parameters is high. The results obtained are compared to the results of the MC method and to the second-order perturbation methods. The results are closely correlated.

### 1.2.2.3. Remark

The perturbation method consists of expressing all the random variables by their Taylor expansions around their mean values. However, the use of this method is difficult to implement, particularly in the case of systems with many degrees of freedom and in cases where the uncertain parameters have a low dispersion around their mean.

EXAMPLE 1.1.– Application of the perturbation method.

The objective of this example is to demonstrate the advantages of the Muscolino perturbation method to determine the beam response.

A beam which is fixed at its extremities and free to vibrate in the (Oxy) plane is considered (Figure 1.1).

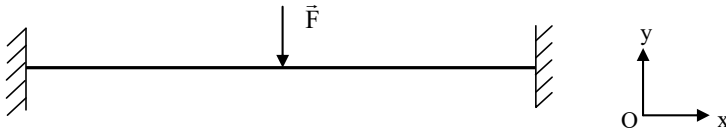


Figure 1.1. Biembedded beam

The mass and stiffness matrices are given by:

$$[M] = \frac{m}{420} \begin{bmatrix} 156 & 22.l & 54 & -13.l \\ 22.l & 4.l^2 & 13.l & -3.l^2 \\ 54 & 13.l & 156 & -22.l \\ -13.l & -3.l^2 & -22.l & 4.l^2 \end{bmatrix}, \quad [K] = \frac{EI}{l^3} \begin{bmatrix} 12 & 6.l & -12 & 6.l \\ 6.l & 4.l^2 & -6.l & 2.l^2 \\ -12 & -6.l & 12 & -6.l \\ 6.l & 2.l^2 & -6.l & 4.l^2 \end{bmatrix} \quad [1.1]$$

The beam has a square section of side  $b$ , which is modeled as a Gaussian random variable.

The stiffness matrix  $[K]$  can be written as follows:

$$[K] = b^4 \cdot [A],$$

where  $[A]$  is a deterministic matrix.

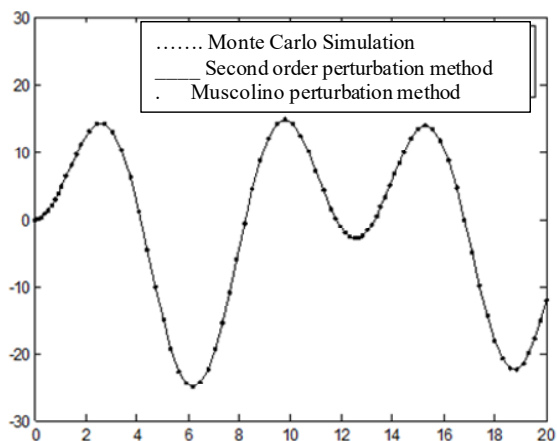
Likewise, the mass matrix  $[M]$  can be written as:

$$[M] = b^2 \cdot [B],$$

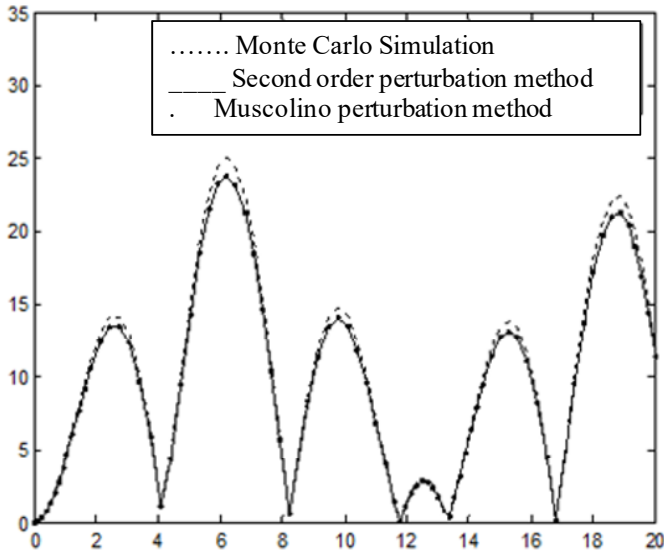
where  $[B]$  is a deterministic matrix.

The beam's response to a force  $F = 600 \sin(800t)$  applied at the beam midpoint is studied. The mean value and standard deviation of the displacement of the beam midpoint are calculated with the second-order perturbation method and the proposed new method. The results are compared to those obtained with the MC method as the reference using 10,000 draws.

The results (Figures 1.2 and 1.3) show that the two perturbation methods give the same results as the MC method.



**Figure 1.2.** Mean of the displacement of the beam midpoint



**Figure 1.3.** *Standard deviation of the displacement of the beam midpoint*

### **1.2.3. The polynomial chaos method**

#### **1.2.3.1. Origins and principle**

The polynomial chaos method is a powerful mathematical tool that was developed by Wiener in the framework of his theory on homogeneous chaos [GUE 15a, GUE 15b]. This method formalizes a separation between the stochastic components and deterministic components of a random function. The polynomial chaos leads to a functional expression of a random response by decomposing its randomness on the basis of orthogonal polynomials.

In a general sense, a set of second-order stochastic variables (with finite variance) can be expressed by a series expansion of Hermite polynomials, orthogonal functions of some Gaussian and independent random variables modeling uncertainty. The deterministic part is

modeled by coefficients  $\bar{x}_j$ , called stochastic modes, weighting the Hermite polynomial functions.

$$X(\xi) = \sum_{j=0}^{\infty} \bar{x}_j \phi_j(\xi) \quad [1.2]$$

The polynomial family,  $\Phi_i$ , forms an optimal orthogonal basis and allows for a convergence of the expansion [ELH 13], in the sense of least squares. However, the convergence's rapidity and the expansion's accuracy in Hermite polynomials are not verified anymore with non-Gaussian processes. In fact, in the case of Gaussian processes, the Hermite basis optimality is a consequence of the Gaussian characteristic of the probability density function (PDF). The mathematical expression of this PDF is then equivalent to the weight function associated with the scalar product defined in this Hermite basis. This principle can be generalized and used to establish a correspondence, called the Askey scheme [ASK 85], among families of orthogonal polynomials and probability distributions. The concept of expansion in a generalized chaos polynomial can then be defined. An exponential convergence is thus demonstrated and generalized to arbitrary probability laws (not necessarily Gaussian) [GHA 99].

### 1.2.3.2. Remark

Polynomial chaos is a concept that is well suited to the modeling of random functions and processes. It is a tool that allows the consideration of uncertainties and nonlinearities in modeling and systems analysis. The numerical schemes by which polynomial chaos is implemented differ in the way they make use of the model, which is subject to uncertainty propagation. The intrusive numerical scheme has the advantage of requiring only one calculation to determine the stochastic methods. This calculation is tedious when the original model contains a lot of uncertain parameters. The calculation's complexity is greater in the case of systems with many degrees of freedom that are highly nonlinear. This is due to the fact that the original model is transformed via its projection on the basis of the polynomial chaos in a system of deterministic equations,



whose dimension and complexity depend significantly on the original model's number of uncertain parameters and degrees of freedom.

In contrast, the non-intrusive scheme has a significant advantage; in that it does not require modifications or transformations of the original model. In [ELH 13], we find numerous applications of this method.

### **1.3. The design of experiments method**

#### **1.3.1. Principle**

The design of experiments (DOE) method makes it possible to implement or simplify, in terms of complexity and cost, an experimental protocol to determine the parameters impacting the performance of an industrial product. The objective of the DOE method is to obtain a design which is almost insensitive to variation in system parameters. By setting the number of experiments to be performed, this method makes it possible to determine the impact of several parameters on the system responses. When applied to a given system, its efficiency depends on the control of the values given as input to the system parameters and on the precision of the measurements of the corresponding responses. Several techniques are based on the DOE concept. The various concepts which can be applied to design sets of experiments are described by Chatillon [CHA 05].

The Taguchi DOE method makes it possible to significantly reduce the number of trials [TAG 86]. This method is implemented by using two matrices conjointly: a control matrix representing the adjustable factors and a noise matrix representing the noise factors (uncertain parameters). The trials are performed for each combination of the factors identified in these matrices. The statistical data such as the mean value and standard deviation of the response signal are measured. To evaluate the quality of the results, the Taguchi method

uses as a quality criterion the signal to noise ratio and a loss function. The method developed by Huang-Chang [HUA 05] which is based on the concept of orthogonal columns makes it possible to simultaneously study multiple design parameters, thus reducing the minimum number of trials.

### **1.3.2. The Taguchi method**

This statistical method is used to set an experimental protocol which renders the main response of a system insensitive to the different values of its parameters. A set of experiments is defined along with the various sets of system parameters that will be used. The number of experiments to be performed depends on the adjustable design parameters, the number of random (uncertain) parameters, possible interactions between these parameters and the effect of these parameters on the response (linear effect or not, etc.).

By taking into account the variability of multiple parameters, the Taguchi method optimizes the system response. Originally this method used the signal to noise ratio as a quality indicator thus combining mean and variance.

The advantage provided by the Taguchi method is to simplify the implementation of the design of experiments. It proposes a selection of experiment matrices; tools for helping to choose the most suitable table and advice to take into account the interactions between the adjustable factors of the design.

Taguchi's tables make it possible to:

- choose the matrix of trials to perform according to the number of factors, modalities and interactions;
- verify, using linear graphs, that the selected table takes into account all the factors, their interactions and is representative of the problem under study;
- identify, using the interaction table, the columns corresponding to the interactions that have not been taken into account.

EXAMPLE 1.2.— Application of the design of experiments in robust design.

The aim of this example is to highlight the advantages of the DOE method in making the system response insensitive to variations of input parameters. A microcontroller component assembled on a rectangular printed circuit board is considered. This microcontroller has 256 pins that are connected to the circuit board by solder joints. The printed circuit is attached to an aluminum alloy case with five screws (one in each corner of the circuit and one in its central region). Applying the Finite Element Method (FEM) a model of the printed circuit board equipped with this microcontroller component is developed. The input parameters are geometric (position of the fifth screw, thickness of the printed circuit) and the physical properties of materials (printed circuit board layers, solder, pin, the composite molding compound of the microcontroller). The response of the model is the strongest stress applied during thermal loading on the solder joints among the 256 solder joints of the component.

To select the only input variables that have a significant effect on the response, a screening design of experiments is performed. As the number of variables is 35, a Plackett–Burman design is developed. This design leads to a selection of only 15 parameters that have an effect on the response.

To obtain the hypersurface of the response in the multidimensional space, a design of experiments, of the hypercube latin (LHS) type is carried out on the selected variables. An LHS design with  $n$  trials is an experimental design for which:

- each parameter is divided into  $n$  levels identified by a set of numbers (the higher the number, the thinner the “mesh” and the more easily the fitted model can reach the optima);
- each parameter can take a level once only.

Each parameter is divided into  $n$  levels that are equally distributed between its minimum and maximum value. A sampling that is representative of the hypervolume is thus obtained. The LHS design is tailored to digital testing because of its simplicity of implementation,

and to the spatial interpolation method (Kriging), which provides the hypersurface of the response.

The considered response is the mechanical stress applied on the solder joint which, among the 256 joints of the component, has the largest median stress. 70% of the trials of the LHS DOE are used to build the Kriging model. These trials are randomly drawn among 200 possible trials. The remaining 30% are used to validate the predictive efficiency of the model.

The obtained response surface makes it possible to approximate the stress on the most exposed solder joint. Fifteen variables are considered in this response model. To identify the variables that have the most impact on the stress, a global sensitivity analysis is performed using the Sobol indices method. Several simulations (total number:  $n$ ) of the input parameters are carried out. The response is calculated using the Kriging model. Then, to study the sensitivity of a given parameter, another draw on all other parameters except this one is conducted. This step is repeated many times by bootstrap. The Sobol indices of the parameters under study are then calculated from the total variance and variances relative to the studied parameter. A statistical distribution for each index is obtained. This distribution is represented by a boxplot and used to estimate a confidence interval on the index value.

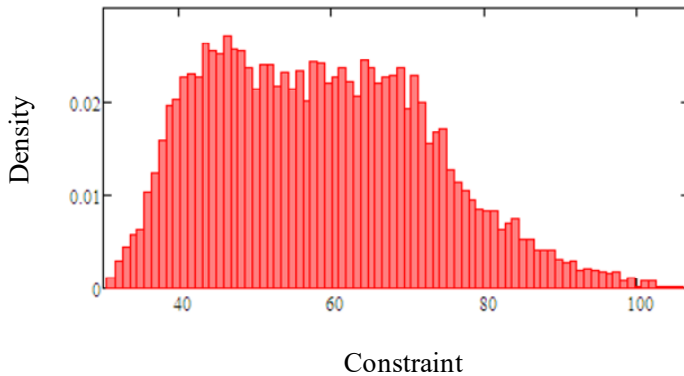
One thousand simulations to simulate the input parameters and 100 calculations of indices are performed. The parameters that appear as the most influential on the response are in the order of importance:

- Parameter X1 (COMP\_Z): the thickness of the solder joint;
- Parameter X4 (EX\_SOLDER): the Young's modulus of the solder;
- Parameter X5 (ALP\_SOLDER): the expansion coefficient of the solder;
- Parameter X14 (ALPX COMP): the component of the expansion coefficient in the design.

Once the influential factors are identified, MC simulations are performed to determine the distribution of the constraint on the solder joint as a function of the variations of the influential factors:

- a nominal value for each non-influential factor is then assigned;
- for each influential factor, a draw of a uniform law in its field of variation is conducted;
- finally, the value of the constraint on the solder joint is calculated by the Kriging model.

The procedure described above is iterated a large number of times (107) to obtain the distribution of the stress applied on the solder joint.



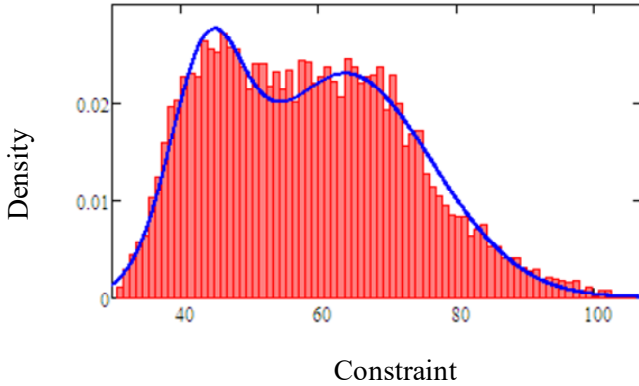
**Figure 1.4.** *Histogram of the stress on the solder joint*

This distribution can be estimated by a parametric model, such as a Gaussian mixture model. The obtained result is shown in Figure 1.5.

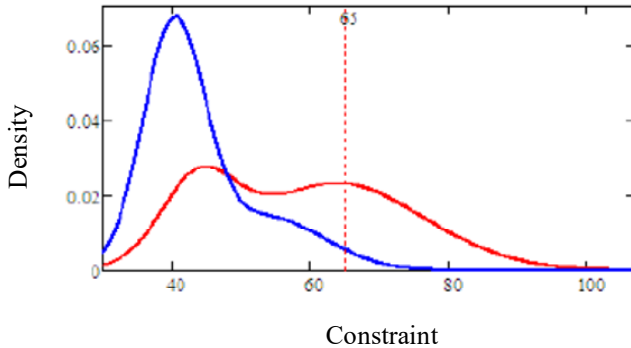
The level of control factors is finally adjusted to reduce the system's sensitivity to sources of variability (noise factors) and adjust the system's response to its target (goal).

The factor ALPX COMP (CTEX of the component) has a strong effect on the constraint (positive influence). This factor can be adjusted by the composite structure of the component coating material. To minimize the stress, small values of Alpx COMP must be

drawn. By reducing the range of variation of the Alpx COMP factor to the interval [5, 7] instead of [5, 23] initially, the average value of the stress and its variability are reduced (Figure 1.6).



**Figure 1.5.** *Density of the stress on the solder joint*



**Figure 1.6.** *Density of the initial stress (red) and optimized constraint (blue). For a color version of this figure, see [www.iste.co.uk/dahoo/nanometer.zip](http://www.iste.co.uk/dahoo/nanometer.zip)*

#### 1.4. The set approach

The methods of the reliability approach described in section 1.3 require as a prerequisite that the probability laws governing the