Continuum mechanical investigations on microstructures

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Continuum Mechanical Investigations on Microstructures

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presented by
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Abstract

The mechanical characterization of microstructures, i.e. structures whose characteristic dimensions are in the μm-range, is the main topic of this thesis. To achieve this goal, an approach is chosen which might be called downscaling. This means that the microstructures are tested and analysed with the same techniques as known from classical material testing, but adapted to the specific problems and needs of small dimensions.

The specimens used during this work consist of two different materials. The silicon samples were produced by anisotropic wet etching. The metallic specimens, either pure Ni or NiFe alloys, were made with the LIGA technique.

One of the main problems with mechanical tests on microstructures is the determination of the geometrical dimensions. The accuracy of the results obtained in any structural analysis is mainly dependent on the accuracy in the dimension measurement. A possible way of how fairly accurate measurements can be achieved is shown, and an error analysis is made.

Two main characterization techniques are used in the present work, static torsional tests and dynamic resonance tests. The dynamic tests allow the determination of the elastic constants by measuring the resonance frequency. This is done for several modes, beam modes as well as plate modes. As some of the materials investigated have a non-isotropic behaviour, an orthotropic model is used. Four out of nine constants have an influence strong enough to allow their determination. To solve the inverse problem, i.e. deduce the elastic constants when knowing the resonance frequencies, an iterative least square procedure is used, involving numerical and experimental data.

During the dynamic tests, higher harmonic excitations of the specimens are observed. This behaviour can be traced back to two different sources. The excitation signal is not a purely sinusoidal signal, but also contains higher harmonics. This is however not sufficient to explain the behaviour of some modes. It is shown that a non-linear model is quantitatively in good agreement with the experimental results. Possible reasons for the non-linearity in the mechanical system are discussed.
Abstract

The static test is performed using a specially designed torsional setup. The main
difficulty consists in the measurement of the tiny torques occurring during the
test. This problem is solved with a self-built differential torque-sensor, allowing
measurements in the µNm-range, with an accuracy of 3% of the nominal value,
and a resolution of less than 0.1 µNm. The working principle of the sensor is
based on the fact that the torque acting in the microsample induces a rotation of a
rigid part of the sensor, which is connected to a spring. By measuring the dis¬
placement of the rigid part at two different points, the torque can be deduced. It is
important to notice that no friction, caused either by torque or longitudinal
forces, occurs in the sensor. The torsional setup leads to results in the form of
torque-rotation diagrams. By analysing these structural responses, it is possible
to calculate the dominating elastic constants, e.g. two shear moduli. This is done
with the same mixed numerical experimental technique as applied during the res¬
onance tests. The results obtained by the two different tests are in good agree¬
ment.

The knowledge of the elastic constants is a necessary condition for the determi¬
nation of failure criteria. Due to stress concentrations in the silicon sample, fail¬
ure always occurs at one of the notches present due to the fabrication process. An
energy criterion, based on surface energy considerations, is formulated with the
help of numerical simulations. For metallic specimens, both von Mises’ and
Tresca’s hypotheses are formulated. The critical equivalent stresses found at the
beginning of yielding lie between the equivalent stresses of von Mises and
Tresca.
Zusammenfassung


Im Verlaufe der dynamischen Versuche konnten höher harmonische Anregungen der Mikroproben festgestellt werden. Dieses Verhalten wird durch zwei verschie-


List of symbols

D  Matrix of relative sensitivity of resonance frequencies with respect to geometrical parameters
R  Matrix of relative sensitivity of elastic moduli with respect to resonance frequencies
S  Matrix of relative sensitivity of resonance frequencies with respect to elastic moduli
F  Global load vector
K  Stiffness matrix
M  Global consistent mass matrix
Q  Global displacement vector
d  Vector of relative dimensional errors
m  Relative resonance frequency error due to erroneous dimensions
r  Total relative resonance frequency error vector
u  Displacement vector
w  Relative resonance frequency error due to frequency measurements
A  Area and vibration amplitude
A_R Amplitude at resonance
B  Coefficient
D_{ij} Plate stiffnesses
E  Young’s modulus
G  Shear modulus
I_i Area moment of inertia with respect to the x_i axis
I_p Polar moment of inertia
M  Moment per unit length
N  Normal force per unit length
Q  Quality factor or shear force per unit length
R  Rayleigh quotient or error function
R_a Surface roughness
S  Surface energy
S* Surface energy per unit thickness
SED Strain energy density
T  Torque or kinetic energy
U  Strain energy or applied voltage
U* Strain energy per unit thickness
List of symbols

W  Work potential of externally applied forces
a, b, c  Dimensionless factors
d  Distance
f  Frequency
f_R  Resonance frequency
h  Height
k_T  Torsional stiffness
l  Length
t  Time
u_i  Displacement in x_i direction
w  Width
x_i  Coordinates
x  Dimensionless displacement parameter
α  Angle or rotational error
γ_S  Specific surface energy
δ  Translational error
ε  Small dimensionless factor
ε_ij  Components of the strain tensor
θ  Rotation
Θ  Dimensionless time parameter
λ  Eigenvalue
ν  Poisson coefficient
Π  Potential energy of an elastic body
ρ  Density
σ_ij  Components of the stress tensor
σ_v  Equivalent stress
σ_y  Yield stress
ϕ  Phase
Φ  Stress function
ω  Circular frequency
ω_R  Circular resonance frequency

(\partial_{x_i})  Derivative with respect to the coordinate x_i
(\partial_{t})  Derivative with respect to time
(\cdot)  Derivative with respect to time
∇  Nabla operator

Literature references are indicated by [ ] brackets
1 Introduction

This is the stuff the dreams of many scientists are made of. Don't worry, I am not making myself guilty of some kind of premature human hybris, as I am not talking about the content of this thesis. By considering the image on the left hand side, one might think that I have in mind a scientist's nightmare about always plodding along the same old paths, feeling upside down. What I am talking about however is the ants, those tiny little creatures you encounter in almost every climate and latitude. They simply have everything so many scientists try desperately and sometimes vainly to reproduce in their labs. They are small entities fully equipped with tactile, olfactive and optical sensors allowing them to scan their environment as they proceed, not to speak of their precise actuators with which they cut, bite, transport and move things around, things which seem huge as compared to the ant itself. What's more, they are intelligent. Maybe not as an individual but as a whole group, they thus constitute a very powerful neural network, able to accomplish complex tasks.

These are features that scientists in the field of microelectromechanics try to replicate in their devices. Before trying to handle such a task, it is however very useful to have some basic knowledge about the materials one is working with. The main aspect from this point of view may well be the mechanical one. This work deals with some of these mechanical aspects. At the centre of it are small structures or more precisely different mechanical properties of small, micrometer-sized structures. To get some knowledge about these characteristic properties, experimental setups have to be designed and methods of interpretation of these experiments have to be developed. It should be mentioned right from the start that the known theories of macromechanics are the key to understanding the mechanics of the structures investigated during this work, though they may have to be adapted or combined in different ways. In this context numerical simulations are a very powerful tool and they help solve problems which would otherwise practically be out of reach.

This work deals with structures whose characteristic dimensions are expressed in micrometers. It does not go beyond this step to investigate what is going on at a molecular level.
1.1 Previous research

Since the first paper of Petersen and Guarnieri [50] in 1979, a fair amount of work has been done to test the mechanical properties of microstructures. As explained in the previous section, work in the micromechanical area is based on and biased by knowledge gained from continuum mechanics on a macroscopic scale. Thus people try to use the same experiments as in conventional tests, simply by downscaling them. These experiments can roughly be separated into two categories, dynamic, non-destructive and static, commonly destructive experiments.

The first category typically uses some kind of resonance frequency measurements to determine elastic moduli. Sometimes shear-moduli as well as residual stresses are obtained from these tests. The classical test specimens are either beams, which come in the form of cantilevers or bridges, and thin membranes subjected to various boundary conditions. The spiritual father of the resonance tests are Petersen and Guarnieri [50], who analysed cantilevers of various materials to determine their Young’s moduli. Working along the same lines were Hök and Gustaffson [22], Putty et al. [53], Zhang et al. [73], Kiesewetter et al. [30] and Ye et al. [72], some of whom used beams and some membranes. Besides Young’s moduli, residual stresses are often calculated in these works. In 1989 Tang et al. [68] are the first to take into account higher order resonance modes. It took some years to see the potential of this procedure. Starting in 1995 there are some people using several torsional and flexural modes to determine Young’s and shear moduli of microstructures, namely d’ Evelyn et al. [10], Mazza et al. [37], Hoummady et al. [24] and Nakano et al. [48]. One of the main problems in all of these works is to match the analytical model to the actual experiment. Considering the non-destructive domain, there are of course always exceptions to the rule, so some people did use dynamic excitation to perform destructive tests, to study crack propagation in microstructures by measuring shifts in resonance frequencies. The first work in this direction was done by Connally and Brown[9], followed by Schlums and Dual [59].

Some work has also been done in the second category, the static tests. The heavyweights in this category, as far as pure quantity and number of papers published is concerned, are bending tests. There are simple reasons for this. It is not due to the fact that bending experiments yield highly precise results or are a better
model than tensile or torsional tests for everyday microstructure-loading conditions. There are so many of them because the structures needed can be easily manufactured and the experimental setups are relatively straightforward, using standard equipment. This does not mean that the interpretation of the results obtained necessarily has to be easy, quite on the contrary.

Howe and Muller [25], in 1983, are the first to work with bending deflections of microbeams. They qualitatively study residual stresses. Several years later Johansson et al. publish two papers [28] and [29], which cover the subjects of internal stress, Young’s modulus and fracture toughness of silicon cantilevers. Weihs et al. study Young’s modulus and yield strength of SiO₂ and Au beams in two different papers, [70] and [71]. Working on the same subject, for different materials, are also Najafi and Suzuki [47], Ericson and Schweitz [13] and Ljungcrantz et al. [36]. It soon dawns upon people that the interpretation of their results is not as straightforward as they first thought it to be. They begin to take into consideration the compliance of the supporting body, either by some analytical model or by finite element simulation, such as Tai and Muller [67], Mullen et al. [46] and Meng et al. [44]. In an overview paper, Sommer and Olaf [63] underline the importance of finite element simulation concerning boundary condition and nonlinear effects due to large displacements. Instead of using microbeams, some people study the load-deflection behaviour of membranes. This can be done in several ways. Allen et al. [2] apply a uniform pressure on one side of a square membrane and measure the deflection on the other side. By assuming a value for the Poisson coefficient v they determine the residual stress analytically as well as Young’s modulus for thin polyimide films. Tabata et al. [65] follow a similar procedure, but they discuss the inconvenience of the assumption of v. Besides they work with composite membranes made of silicon nitride deposited on polysilicon. Hong et al. [23] work on circular membranes, which are deflected by using a nanoindenter, able to measure force and displacement. Kiesewetter et al. [31] also use a point load to deflect a rectangular membrane and study the fracture stress of monocrystalline silicon.

To circumvent the problems related to the bending tests, people thought it might be a good idea to design some equipment for tensile tests, where they would be able to determine directly Young’s moduli as well as fracture stresses. In tensile tests the errors due to inaccurate dimensions are smaller as in bending tests, as dimensions only have a quadratic influence on stresses, compared to a cubic one for bending. Some of the first to do so were Koskinen et al. [33] in 1993 with ten-
sile tests on polysilicon fibres. They were able to measure the force applied but had no means of measuring the actual displacement of the fibre ends. Ilzhöfer and Tsakmakis [26] did some tests on LIGA specimens and measured force and displacement independently. To do so they clamped their specimens in a frame which is rigid with respect to the specimens, but deformable with respect to the tensile testing machine, in which the frame is integrated. Using calibrated strain gauges they measure the forces, whereas inductive sensors allow the measurement of the axial displacement of the frame and of the force sensors. Their difference yields the actual displacement of the specimens. 1996 was a very propice year for the tensile test community, where Sharpe et al. [62] and Mazza et al. [37], [38] presented some neat test equipments and results. Mazza used an elegant method to measure the occurring loads by using a high-precision balance. Both were able to accurately measure displacements and thereby strains by using optical techniques. Sato et al. [57] presented some interesting ideas, but their lever system, involving bending, torsion and tension of different parts, only allows an indirect measurement of force and displacement.

Seeing these thin microbeams, a kind of Pavlov reflex set in with some engineers and they immediately wondered what would happen to those structures under buckling loads. They tried to answer this question in varying degrees of refinement by usually studying microbeams clamped at both ends. Tai and Müller [66], Mullen et al. [46], Meng et al. [44] and Fang and Wickert [14] participated in this quest.

Another large group of researchers is occupied by indentation on thin films or other microstructures. These tests are usually called micro- or even better nanoindentation. To carry out these tests, they usually use commercially available machines, called nanoindenters (displacement resolution nanometers) or picoindenters (displacement resolution picokilometers), which simultaneously measure force and displacement. Theoretically Young’s modulus and hardness of the films can be determined this way, but the interpretation of the experiments is not easy at all. A good description of the possible errors occurring during an indentation test is given by Mencik and Swain [41]. The advantage of this testing method is its access to very small structures and local areas, where conventional testing methods as described above, fail. By a careful analysis and procedure, trustworthy results can nevertheless be obtained, as shown by Pethica and Oliver [51], Pharr et al. [52], Field and Swain [16], Mencik et al. [42] and [43] and Knapp et al. [32].
Now one would assume that this really covers the field of possible mechanical testing on microstructures. Far from it, an important but hitherto neglected field is still missing, the field of torsional tests on microstructures. In fact, besides the work described in this thesis, there is only one more study, which has been published by Saif and Mac Donald [55]. A closer analysis of their paper will be provided in section 4.1.

1.2 Scope and outline of the present work

Trial and error prevails in the micro-electro-mechanical-systems (MEMS for insiders) world, at least as far as mechanical design and construction rules are concerned. MEMS devices are built in the secret hope they will not break the first time they are used. The responsible engineers probably pray after shipping that their device will survive a long enough time for people not to complain, because very often they cannot really tell you what their construction supports and what it does not. This lack of quantitative knowledge about mechanical reliability and the deficit in mechanical considerations is due to the fact that micromechanics, whose roots lie in an electrical field of research and applications, is a very young field.

This work takes a first step towards eliminating these obvious deficiencies. Before being able to optimally design a mechanical structure, it is of utmost importance to know the basic and fundamental mechanical parameters of the material considered. The aim of this work does however not consist of only providing these parameters. At least as much importance is accorded to the means and procedures necessary to determine them. The problems occurring during the experiments and their evaluation are discussed, and various solutions are presented.

This thesis contains four chapters. After the introduction, the second one deals with the microstructures used in the experiments, which were specially designed for mechanical tests. Two different kinds of specimens are used, one made of monocrystalline silicon, and one made of nickel and nickel-iron alloys. Their main features, i.e. their geometry and their fabrication process, are briefly presented. Problems occurring during the determination of characteristic dimensions are discussed.
Chapter 3 is dedicated to resonance tests. After a brief overview of the governing equations and of the numerical models used, the experimental setup and results are presented. For each sample several modes of vibration are excited and the resonance frequency, as well as the Q-factor are measured. The frequencies cover a range of 40 Hz to 150 kHz, which allow the excitation of beam modes, as well as plate modes. As the shapes and geometries involved are too complicated, the inverse problem (find the elastic constants when knowing the resonance frequencies) cannot be solved analytically. A new method in micromechanics involving mixed numerical experimental techniques, which use an iterative least square procedure, is developed. An error analysis shows the limitations and main error sources of this method. Higher harmonic oscillations are observed during the experiments, i.e. excitation with a fraction of the resonance frequency causes oscillation of the sample at resonance frequency. The causes of this phenomenon can be traced back to non-linearities in the mechanical system and to higher harmonics in the excitation signal.

In chapter 4 the static torsional test is described. A major part of the work was dedicated to the development of a torque sensor, able to do measurements in the μNm-range. First some basic measurement principles are introduced, then the finally used sensor is presented. The experimental setup allows to perform pure torsional tests, i.e. in the consideration of failure criteria stresses due to bending and tension of the sample can be neglected as compared to shear stresses. The shear-moduli determined in the torsional tests are in good agreement with those found during the dynamic experiments. For the determination of the shear-moduli, an analogous mixed numerical experimental technique as described in chapter 3 is used. One main aspect of static tests is of course the challenge to find failure criteria. Once again numerical simulations make life a little bit easier, and help formulate an energy failure criterion for silicon. For the metallic specimens, Tresca or von Mises criteria are found to be valid.
M.C. Escher
Convex and concave
lithograph 1955
In order to do micromechanical research, it is obvious that samples are needed which present micromechanical features, which means that the characteristic dimensions should be in the micro-meter range. A beam having a length of about 500 μm and cross-sectional dimensions of 50x50 μm² seems to qualify for the job. As a supplementary feature it would offer the possibility of producing large quantities on a single wafer. It has however a major drawback, it is impossible to perform any static or dynamic tests on these samples. To be able to do so, a link to the macroscopic world has to be established. This is done by two plates, having characteristic dimensions of a few mm, attached at each end of the microbeam. They offer the possibility of fixing the samples to the test-apparatus. As it turns out, they have furthermore the advantage of simplifying the measurement of displacement and rotation. By taking into account these two major features, microbeam and macroplates, one ends up with a structure resembling the one shown in Fig. 2.1. Two more features are visible in Fig. 2.1, the protective removable frame, which avoids damage of the testing region prior to the test and the microstructures for the detection of internal stresses of bending or tensile nature.

**Fig. 2.1** Schematic representation of the sample's design
2.1 Single crystal silicon sample

The silicon specimens are manufactured by an anisotropic wet etching process of (100) silicon wafers. A detailed review of the fabrication process is given by Mazza [39]. It should however be mentioned that at concave corners (111) planes are formed, due to their higher resistivity to the etching agent. This leads to notches at the meeting points of microbeam and transition regions, see Fig. 2.2. These notches are characteristic of single crystal silicon structures manufactured by anisotropic wet etching. At these notches stress concentrations occur which are ultimately responsible for the failure of the specimens.

The specimen itself consists of two plates which are connected to the microbeam by two transition regions, see Fig. 2.2. The plates' dimensions are roughly 6.3 x 4.2 x 0.38 mm$^3$. Due to the fabrication process they are surrounded by a thin membrane which has the same thickness as the microbeam and whose lateral dimension is 80 μm. The transition regions have the same thickness as the microbeam and their width is approximately 300 μm. The axis of the microbeam lies in the <100> direction of the crystal axis. The cross-sectional area and the length of the microbeam is largely determined by the time the wafers are immersed in the KOH etchant. In this work two different specimens have been produced. Their characteristic dimensions (length x width x height) are given by 230 x 90 x 50 μm$^3$, respectively 180 x 50 x 60 μm$^3$. Due to the (111) planes the length of the microbeam is not constant over its thickness, it gets larger towards the bottom. The lengths specified here are the lengths at the top of the microbeam. The surface roughness in the testing region is measured to be about $R_a = 20$ nm, as measured with the UBM profilometer, see Section 2.3.

In an optical microscope the deformations of the internal stress structures can be detected. As no visible deformation is observed, it is assumed that there are no internal stresses.
Fig. 2.2  CCD images showing the geometry of the silicon samples; all dimensions are given in µm
2.2 LIGA sample

The LIGA samples are metallic specimens, consisting of either pure Ni or various NiFe-alloys. They were produced at the IMM (Institut für Mikrotechnik in Mainz) using the LIGA (Lithographie, Galvanoformung, Abformung) technique. A good technical overview over the LIGA technique and its possible applications is given by Becker et al. [4], Ehrfeld et al. [11], [12] and Bacher et al. [3].

The layout of the samples is similar to the one used for the silicon specimens, see Fig. 2.3. The dimensions of the plates are 7 x 5 mm² with a varying height of 100...200 μm. Due to the LIGA technique the height remains more or less constant over the whole specimen. The transition region mechanically connects the plates to the microbeam. The possibility to design circles with the LIGA technique allows a smooth connection. The microbeam has a length of 300 or 1000 μm and a width of 20, 30 or 40 μm. These values are determined within certain error limits by the mask used. The height however varies from batch to batch in a range of 100 to 200 μm. The holes in the plates are necessary for the distribution of the etching solution, when a sacrificial layer technique is used for the separation of the sample from the substrate. Surface roughness is much higher than for silicon samples and varies from batch to batch. Typical values lie between $R_a = 200$ nm and $R_a = 1000$ nm. Five different materials are tested in this work, Ni, NiFe(13%Fe), NiFe(20%Fe), NiFe(27%Fe), NiFe(53%Fe).

A major problem with this fabrication technique is the detachment of the specimens from the substrate. More often than not the sample is plastically deformed. Analyses of intact structures for internal stress detection indicate that no internal stress is present in the specimens. To prevent experiments with predeformed specimens, each sample has to be controlled prior to testing, using an optical microscope.
2.3 **Determination of dimensions of microstructures**

Mechanical tests on microstructures reveal something about the characteristics of the actual specimen upon which the test is carried out. They do not explicitly contain information about the intrinsic properties of the material, i.e. elastic modulus or yield strength. This information is however implicitly present in the test results and can be extracted if the geometrical features of the test-specimen are known. This is not a trivial matter, as the determination of the characteristic dimensions is one of the major, if not the major, error source in micromechanical analysis. The exact influence of dimensional errors will be analysed and discussed in the
corresponding chapters. It is therefore of the utmost importance that the dimensions are measured with high accuracy.

The relative errors in dimensions are different for silicon and LIGA specimens. For the silicon samples all the dimensions have to be measured after the fabrication process, as they all depend on the concentration and temperature of the etching solution and on the time the samples are immersed. As for the LIGA specimens only the height depends on the fabrication process and has to be measured. The other dimensions are given with a resolution of ± 1 μm by the mask used.

For the measuring task, two regions have to be considered separately: on one hand the microbridge consisting of the microbeam and the transition regions, and on the other hand the plates. The lateral dimensions of the plate are measured with an optical microscope, which yields a relative error of 1%. All characteristic dimensions of the microbeam as well as the thickness of the plate are measured with a commercially available profilometer using a laser focusing principle, which minimizes the spot diameter of a laser light in a controlled closed loop (UBM measurement system). An out-of-plane resolution of 10 nm can thus be obtained at a working range of 100 μm. The in-plane resolution of 1 μm is given by the minimum spot-diameter and by the resolution of the translation stages which allow an in-plane motion. The measuring procedure to obtain height and width of the microbeam shall be outlined below:

• Put the microbeam on a sharp edge and make sure its axis is perpendicular to the edge.
• Make a surface scan of a region approximately 200 x 100 μm² in surface.
• Determine the line of contact between the specimen and edge, denoted as line A in Fig. 2.4.
• Determine the angle α between horizontal plane and specimen axis with help of line B.
• Viewing the two-dimensional profile of line A indicates the height/\cos(\alpha) of the sample.
• The profile is not suited to determine the width, as it does not present sharp edges. Fortunately the program does not only yield profile measurements, but it can display the quality of reflection as well. This allows an accurate determination of the width.
Fig. 2.4 Sample dimensioning using a laser focusing profilometer
a) surface scan of the microbeam over a region 200 x 120 μm²
b) angle \( \alpha \) between beam axis and horizontal plane
c) reflection along line A
d) profile along line A
The height of the plate and the length of the microbeam, as well as the dimensions of the transition regions can be found in an analogous way. The accuracy of the modelling, i.e. the determination of the dimensions, is not only given by the intrinsic accuracy of the profilometer. It is above all the out-of-plane component, i.e. the height, which may be inaccurate. For the silicon samples this is due to the fact that the original wafer’s height does not remain constant over its surface. To minimize errors, height measurements at three different locations on the beam are carried out and the absolute error is finally assumed to be 0.5 μm. As variations will be larger over a bigger surface, the height of the plate is measured at six different points and the absolute error is finally equalled to 3 μm. The same problems arise with LIGA specimens, only more so, as the variations in the height can be larger, due to the fabrication process. The accuracy of the other dimensions corresponds to the accuracy of the profilometer. The absolute errors for both silicon and LIGA samples are shown in Table 2.1. It should be mentioned that for the static experiments, only the dimensions of the beam are measured. During the resonance experiments, the dimensions of the plate also have to be determined. Needless to say that these measurements are performed for each specimen.

<table>
<thead>
<tr>
<th></th>
<th>$\Delta l_B$</th>
<th>$\Delta h_B$</th>
<th>$\Delta w_B$</th>
<th>$\Delta l_p$</th>
<th>$\Delta h_p$</th>
<th>$\Delta w_p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>LIGA</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>silicon</td>
<td>1</td>
<td>0.5</td>
<td>1</td>
<td>0.01 $l_p$</td>
<td>3</td>
<td>0.01 $w_p$</td>
</tr>
</tbody>
</table>

Table 2.1 Absolute errors in dimensions in μm
l, h and w stand for length, height and width respectively,
B and P stand for microbeam and plate respectively
Seite Leer / Blank leaf
M.C. Escher
Waterfall
lithograph 1961
3 Resonance experiments

3.1 Constitutive equations

Before trying to determine the governing equations of motion of the microstructure, the constitutive equation has to be established. A priori there is no reason to assume that the LIGA-structures are isotropic, apart from the fact that handling isotropic material makes life a lot easier than dealing with the more complex calculations of anisotropic material. Literature on the subject of electrodeposited Ni and NiFe mentions columnar as well as lamellar structures, see Landa [34], Brugger [5], Abel et al. [1] and Ilzhöfer and Tsakmakis [26]. During this work no metallographic, textural or structural studies of the specimens were performed. However, the studies mentioned before seem to indicate some kind of symmetry in the materials. This is the reason why the metallic samples are not modelled using the general anisotropic behaviour, featuring 21 independent elastic constants, but instead using orthotropic material characteristics, which are fully described by 9 independent elastic constants. The constitutive equation can be written in several forms. Which form is used depends on the preferences of the author, if he thinks of himself as a physicist he will either use stiffness coefficients, commonly denoted by $c_{ij}$, or compliance coefficients, represented by $s_{ij}$, to establish a relation between stresses and strains. Engineers, however, are used to working with Young’s modulus $E$, Poisson’s coefficient $\nu$ and shear modulus $G$. So the engineer tends to interpret the relation between stresses and strains in orthotropic material in the same way he does for isotropic material, by introducing generalized Young’s and shear moduli as well as Poisson’s coefficients. This is the approach which will be adopted in this work. The result of all this work and coefficient manipulation can be seen in Eq. (3.1), which represents the constitutive equation, or generalized Hooke’s law, for orthotropic material.
As previously mentioned only nine independent parameters are needed in the case of orthotropy, whereas Eq. (3.1) still has twelve. Due to symmetry considerations, the six Poisson coefficients are related by Eq. (3.2), which yields three more equations.

\[
\begin{pmatrix}
\varepsilon_{11} \\
\varepsilon_{22} \\
\varepsilon_{33} \\
2\varepsilon_{23} \\
2\varepsilon_{31} \\
2\varepsilon_{12}
\end{pmatrix} = 
\begin{pmatrix}
\frac{1}{E_{11}} & \frac{v_{21}}{E_{22}} & \frac{v_{31}}{E_{33}} & 0 & 0 & 0 \\
\frac{v_{12}}{E_{11}} & \frac{1}{E_{22}} & \frac{v_{32}}{E_{33}} & 0 & 0 & 0 \\
\frac{v_{13}}{E_{11}} & \frac{v_{23}}{E_{22}} & \frac{1}{E_{33}} & 0 & 0 & 0 \\
0 & 0 & 0 & \frac{1}{G_{23}} & 0 & 0 \\
0 & 0 & 0 & 0 & \frac{1}{G_{31}} & 0 \\
0 & 0 & 0 & 0 & 0 & \frac{1}{G_{12}}
\end{pmatrix}
\begin{pmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{33} \\
\sigma_{23} \\
\sigma_{31} \\
\sigma_{12}
\end{pmatrix}
\] (3.1)

To fully characterize orthotropic material, three Young’s moduli \(E_{11}, E_{22}\) and \(E_{33}\), three shear moduli \(G_{12}, G_{23}\) and \(G_{31}\) as well as three Poisson coefficients \(v_{12}, v_{23}\) and \(v_{31}\) are needed. To keep expectations at a reasonable level it should immediately be mentioned that not all nine constants can be determined. This non-determinability has nothing to do with Gödel’s incompleteness theorem [20]. For the specimens and loading conditions considered in this study, only five of these nine constants can be determined as will be shown in the next subsection. Due to experimental inaccuracies this number is even limited to four. The material behaviour described is applied both to the metallic and to the monocrystalline silicon specimens. The structure of Si is well known, it grows in a cubic face centered lattice, the same as diamond. Due to this particular symmetry, the number of nine constants can be further reduced to three, \(E, G\) and \(v\), which are sufficient to describe its elastic behaviour. Si is nevertheless modelled as orthotropic. This
more general approach gives a first indication of the validity and accuracy of the method used.

### 3.2 Governing differential equations

Two different categories of differential equations have to be considered. The first one concerns deformations of the microbeam, caused either by bending, torsion or tension. In this case the attached plate only performs a rigid body motion and acts like an attached mass which has supplementary rotational inertia. The second category deals with deformations of the free vibrating plate. It can be bent in two directions, permitting the determination of two Young's moduli and it can be twisted, a motion which is governed by shear-moduli. As will be seen mixed modes, where bending and twisting occur simultaneously are also possible. In all of the following analyses it is assumed that the displacements are small (as compared to characteristic dimensions of the sample) and that the material investigated is homogeneous, which means:

- Mass density remains constant over the sample.
- The elastic moduli remain constant over the sample.

It is further assumed that the coordinate axes correspond to the main axes of anisotropy. The coordinate system used is shown in Fig. 3.1. \( x_1 \) is parallel to the beam axis, \( x_2 \) is perpendicular to \( x_1 \) and is parallel to the mid-plane of the plate and \( x_3 \) is perpendicular to \( x_1 \) and \( x_2 \), so as to form a right-handed coordinate system. For the specimens considered here, it is also assumed that the principal axes of the cross sections are also principal material directions.

![Coordinate system used](image)

Fig. 3.1 Coordinate system used

The governing differential equations, which will be deduced in the following
subsections, do not claim to be a perfect analytical model of what is happening during vibrations in the specimens considered in this work. They are a more general approach to the resonance modes occurring, with the aim to show which elastic moduli are needed (and can therefore be determined by the subsequent analysis) to describe the different beam or plate modes. For this purpose it is fair enough to model the microstructure as a beam of constant rectangular cross-section, to which a thin plate of constant height but varying cross section is attached.

3.2.1 Beam bending

The vibrations can be described by the simple Euler-Bernoulli beam theory, which is valid under the following assumptions:

- Cross sections of the beam remain plane and perpendicular to the axis of the beam.
- Rotational inertia is negligible.
- The area moment of inertia I remains constant over the beam length.
- Cross sectional dimensions are small as compared to the length of the beam.
- Displacements and slopes are considered to be small.

The formulation of equilibrium of force and moment yields the Euler-Bernoulli differential equation:

\[
\begin{align*}
\rho A u_{2,tt} + E_{11} I_3 u_{2,1111} &= 0 \\
\rho A u_{3,tt} + E_{11} I_2 u_{3,1111} &= 0
\end{align*}
\]  

Eq. (3.3) is valid for bending vibrations with displacements in both \( x_2 \) and \( x_3 \) direction. The stresses in both cases are primarily in \( x_1 \) direction, and therefore these vibrations are solely governed by the Young’s modulus \( E_{11} \), besides of course the geometrical features and boundary conditions.

3.2.2 Beam torsion

The assumptions made to study torsional behaviour of an anisotropic beam with constant rectangular cross-section are explained in appendix A, and will be used in this dynamical analysis too. It is furthermore assumed that the acceleration in \( x_1 \)-direction can be neglected. The moment equilibrium in \( x_1 \)-direction then yields:
Resonance experiments

\[ T_{11} = \frac{1}{12}\rho w_B h_B (w_B^2 + h_B^2)\theta_{,tt} \]  \hspace{1cm} (3.4)

Replacing the torque \( T \) by Eq. (A.15), Eq. (3.4) yields:

\[ \frac{3^2 G_{12}}{\pi^6} \sum_{m=1,3}^{\infty} \sum_{n=1,3}^{\infty} \frac{1}{m^2 n^2} \left( \frac{4m^2}{w_B^2 G_{13}} + \frac{4n^2}{h_B^2 G_{12}} \right)^{-1} \beta \theta_{,11} = \rho (w_B^2 + h_B^2)\theta_{,tt} \]  \hspace{1cm} (3.5)

\( w_B \) and \( h_B \) are the width and height of the beam, respectively. Contrary to bending there are two moduli governing the torsional vibration, \( G_{12} \) and \( G_{13} \). This is due to the fact that there are, in a first approximation, only two non-vanishing components of the stress tensor, \( \sigma_{12} \) and \( \sigma_{13} \). It is important to see that \( G_{12} \) and \( G_{13} \) do not have the same influence on the torsional stiffness \( \beta \). Their influence is strongly dependent on the geometry, or to be more precise on the ratio \( w_B/h_B \). The larger this ratio is, the larger is the influence of \( G_{12} \) with respect to \( G_{13} \), and vice versa. Depending on the geometry, it might happen that one shear-modulus has practically no influence on the torsional resonance frequency.

### 3.2.3 Plate vibrations

The differential equation governing the vibration of thin plates, see Eq. (3.6), is deduced in Appendix G.

\[ D_{11} u_{3,1111} + 2(v_{21} D_{11} + 2D_{12})u_{3,1122} + D_{22} u_{3,2222} + \rho h u_{3,tt} = 0 \]  \hspace{1cm} (3.6)

The constant parameters in this equation are functions of the two Young's moduli \( E_{11} \) and \( E_{22} \) as well as of the shear-modulus \( G_{12} \) and the Poisson coefficient \( v_{12} \). This shows that they are the only four moduli which can be determined by measuring plate resonances.
3.3 Finite element simulations

It is obvious that in order to be able to calculate the elastic constants, the resonance frequencies have to be determined in another way than experimentally. A model has to be established, which describes the behaviour of the structure accurately and which can be handled easily. The model may either be analytical or numerical.

The governing differential equations presented in the previous section are only one part of the analytical model, linking the material parameters with the dynamic behaviour. Derive the resonance frequencies from these equations might well prove to be the harder task. In this case it turns out to be virtually impossible to calculate the eigenvalues of the problem by respecting the specific boundary conditions and geometries, especially for Eq. (3.6) and Eq. (3.5). This is due to several reasons, which will now be enumerated:

• The thickness of the plates is not constant.
• The plates do not have a simple shape, i.e. they are not rectangular.
• The holes in the plates of the LIGA-specimens present an irregularity.
• There are no standard boundary conditions.
• The microbeams do not have a constant cross-section.

As the analytical model does not present a viable solution, the only alternative is a numerical model, using the finite element method.

3.3.1 Theoretical background

In summary the finite element method is nothing else than a sophisticated implementation of Caesar’s maxim “Divide et impera!”. A continuous structure is divided into several small, but finite, elements, which are defined by their nodes. Instead of formulating a Ritz function describing the deformation of the whole body, a Ritz function is formulated for each element. This function is called shape function and can be linear, quadratic or of some higher order. By applying the mechanical principles and equations on these elements, respectively on the degrees of freedom of their nodes, the mechanical continuum problem can be reduced to an algebraic problem, which involves solving a large system of equations.

The two single most important mechanical quantities used by finite element
methods in this context are the potential energy $\Pi$ and the kinetic energy $T$, shown in Eq. (3.7) and Eq. (3.8) for the case of no damping. $K$ is the global stiffness matrix and $M$ the global consistent mass matrix. $F$ is the global load vector and $Q$ the global displacement vector, with the components $q_i$.

\[
\Pi = \frac{1}{2} Q^T K Q - Q^T F \quad (3.7)
\]

\[
T = \frac{1}{2} Q^T M Q \quad (3.8)
\]

\[
\frac{\partial}{\partial t} \left( \frac{\partial}{\partial q_i} (T - \Pi) \right) - \frac{\partial}{\partial q_i} (T - \Pi) = 0 \quad (3.9)
\]

Introducing Eq. (3.7) and Eq. (3.8) into Lagrange’s differential equations Eq. (3.9), the equations of motion are obtained:

\[
M \ddot{Q} + KQ = F \quad (3.10)
\]

Assuming free vibrations, i.e $F = 0$, and steady state conditions by setting

\[
Q = U e^{i\omega t} \quad (3.11)
\]

one obtains the generalized eigenvalue problem:

\[
KU = \lambda MU \quad (3.12)
\]

where $U$ is the vector of nodal amplitudes of vibration and $\lambda = \omega^2$. For the eigenvector $U$ to be non-trivial, the determinant of the system has to vanish. This yields a characteristic polynomial in $\lambda$, whose solutions are the eigenvalues of the system. Unfortunately solving this polynomial of degree $N$, where $N$ is the total number of degrees of freedom of the system, is a difficult task and is usually avoided in FE codes. The program MARC used in this thesis tackles the problem by an inverse vector iteration method, using the Rayleigh quotient $R$ and its property that it lies between the smallest and the largest eigenvalue, for an arbitrary displacement vector $u$. If the vector $u$ represents an eigenmode, $R$ is the corre-
sponding eigenvalue.

\[ R(u) = \frac{u^T Ku}{u^T Mu} \]  

(3.13)

The iterative solution procedure consists of the following steps (see [7]):

- Estimate an initial trial vector \( u^0 \). Set iteration index \( k = 0 \).
- Set \( k = k + 1 \)
- Solve \( K\hat{u}^k = M\hat{u}^{k-1} \)
- Estimate eigenvalue \( \lambda^k = \frac{(\hat{u}^k)^T K\hat{u}^k}{(\hat{u}^k)^T M\hat{u}^k} \)
- Normalize eigenvector \( u^k = \frac{\hat{u}^k}{((\hat{u}^k)^T \hat{u}^k)^{1/2}} \)

Provided \( u^0 \) is not an eigenvector, this iteration yields the lowest eigenvalue. The iteration is stopped when the difference between two subsequent estimated eigenvalues lies within a predefined tolerance. The value \( \lambda^k \) is then the first eigenvalue \( \lambda_1 \) and the vector \( u^k \) is the corresponding eigenvector \( U_1 \). Further eigenvalues can be calculated by using as initial trial vector a vector which is orthogonal to all the eigenvectors already found. This can be done by using the Gram-Schmidt process described in Meirovitch [40].

### 3.3.2 Numerical models used

As one might expect, there are several factors which influence the numerical results besides the accurate geometry of the model and the constitutive equations used. According to the different mechanical theories, which make assumptions on the stress and strain fields as explained in the previous section, there are special elements in FEM, which can be used to model the behaviour of beams, plates or shells. However, due to the complex geometry and the loading conditions, the behaviour of the structure investigated cannot be reduced to either beam or plate behaviour. Therefore a full three dimensional analysis is carried out. The elements used are three dimensional 20-node, isoparametric, arbitrary hexahedral brick elements. They use triquadratic shape functions to represent coordinates and displacements, hence the strains and stresses are linear since the analysis is linear elastic. The stiffness and mass matrices \( K \) and \( M \) are calculated numeri-
Resonance experiments

cally, using 27-point Gaussian integration.

Having chosen an element, it has to be decided how many elements are necessary for an accurate simulation, and how these elements will be distributed. The more elements are used the better is the approximation to the continuum behaviour and the better is the simulation. This seems quite natural but has a major drawback, which is the limited computational power. Another problem might be the fact that the numerical Gaussian integration can get inaccurate if the elements are too small. For the simulation of harmonic oscillations, this is of no practical consequences, as the elements remain rather large. The small elements used in the static tests cause no problem, as has been verified with convergence criteria. The number of elements should increase in regions where the stress gradients are highest. This is not carried out systematically due to problems in the modelling, and due to the fact that many modes are considered, modes in which the stress distribution is completely different. To solve the problem, the resonance frequencies of a trial model are calculated. In subsequent steps the trial model is refined and its frequencies are compared to those previously obtained. If the difference for each mode does not exceed a given tolerance, which was set to 0.5% of the frequency, the last model used is the definitive model in all further simulations. To do an intelligent refining, as compared to simply doubling the number of elements used, the number of elements has to be varied separately in the different directions. The procedure will be explained in detail for a silicon specimen but was the same for all LIGA-samples. The final silicon model is shown in Fig. 3.2.

![Fig. 3.2 FE model of silicon sample, dimensions in \( \mu m \)](image)

To show that the meshing of this model was fine enough, four more models were
tested, their respective element numbers are shown in Table 3.1. Taking into consideration all the elements of the beam, the transition region and the plate, about 1100 elements are used. They add up to about 6000 nodes or 18000 degrees of freedom. Due to the geometry and the loading conditions, no symmetry can be used.

<table>
<thead>
<tr>
<th></th>
<th>#elements in l_B</th>
<th>#elements in h_B</th>
<th>#elements in w_B</th>
<th>#elements in l_P</th>
<th>#elements in h_P</th>
<th>#elements in w_P</th>
</tr>
</thead>
<tbody>
<tr>
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<td>model 2</td>
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<td>9</td>
<td>8</td>
<td>6</td>
</tr>
<tr>
<td>model 3</td>
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<td>4</td>
<td>6</td>
<td>16</td>
<td>4</td>
<td>12</td>
</tr>
<tr>
<td>model 4</td>
<td>14</td>
<td>4</td>
<td>12</td>
<td>9</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>model 5</td>
<td>28</td>
<td>4</td>
<td>6</td>
<td>9</td>
<td>4</td>
<td>6</td>
</tr>
</tbody>
</table>

Table 3.1 # elements in various directions for different models

The corresponding resonance frequencies for several modes are shown in Table 3.2 (the modes themselves can be viewed in Fig. 3.5). For the simulation the elastic constants were assumed to be $E_{11} = E_{22} = 130$ GPa, $G_{12} = G_{13} = 80$ GPa, $\nu_{12} = 0.3$. With a single exception the resonance frequencies of all the models differ by less than 0.5% from the frequencies of model 1. The plate of model 3 was refined once more in its length and its width. The differences to the original model 3 drop well below 0.1%. For these reasons it was assumed that the mesh of model 1 was fine enough. All further simulations are done by using model 1.
Table 3.2  Circular resonance frequencies $\omega_R$ for several models
in parentheses difference in percent to reference model (Model 1)

For the simulations one plate is clamped and the beam as well as the second plate can freely vibrate. Variations of this boundary condition have no influence on the frequencies. No matter which part of the plate is clamped, the modes and frequencies remain the same.
3.4 Experiments

3.4.1 Experimental setup

1. Vacuum chamber with specimen
2. Laser sensor head: Polytec OFV 300
3. Pneumatically isolated research table: Newport
4. Stepping Motor controller of the Laser head positioning system
5. Excitational signal amplifier: Krohn-Hite KH 7500
6. PI controller: self built
7. Frequency counter: Keithley 7754
8. Digital storage oscilloscope: LeCroy 9304A 200 MHz
9. Digital function generator: Krohn-Hite KH 5920
10. Digital function generator: Stanford research DS 345
11. Band-pass filter: Krohn-Hite KH 3202
12. Laser demodulator: Polytec OFV 2100
14. PC
The samples are excited using a ceramic circular piezoelectric transducer with a radius of 5 mm and a thickness of 1 mm. Using a two-component glue (HBM X60) the piezo is attached to a metallic block. After detaching the specimen from its protective frame, one of its plates is fastened to the piezo, using either a two-component glue or a wax-like substance called Witepsol, which melts at about 35° C. By applying a sinusoidal voltage to the piezo via a function generator and an amplifier, the longitudinal motion induces vibrations of the sample. The vibrations are detected using a Laser-Doppler interferometer, whose beam is directed upon the lower free-vibrating plate. The direction of the beam has to be perpendicular to the plate. The signal of the laser-head, which is processed in the laser-demodulator, is displayed on a digital oscilloscope after going through a band-pass filter. The frequency is measured with a frequency counter put in series with the function generator. This is the open loop mode which is depicted in Fig. 3.4 and which was mainly used during this work. The other possibility is to work in closed loop mode by using a phase-locked loop (PLL). In PLL mode the difference of the phases of input and output signals are compared to a target phase difference, which corresponds to the phase difference at resonance, in a lock-in amplifier, whose output controls the frequency of the function generator via a PI controller.

![Diagram](image)

**Fig. 3.4** Experimental setup for resonance experiments (open loop)

To detect resonance frequencies, which are expected between 40 Hz and 150 kHz, the samples are excited by a linear sweep signal, going from 0 to 20 kHz, from 20 to 40 kHz and so on. Linear sweeps are signals whose amplitude is con-
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constant over a certain bandwidth, in this case 20 kHz. The measured vibrations are recorded on an oscilloscope and fourier transformed.

The amplitude spectrum gives a first approximation of the resonance frequencies. After focusing on the interesting frequency range and detecting several resonance peaks, each peak is analysed separately. This is done by simply changing the frequency of the function generator manually and by detecting the maximum amplitude $A_{\text{max}}$ of the response signal on the oscilloscope. To determine the quality factor $Q$, which is inversely proportional to the damping, the two frequencies $f_1$ and $f_2$ are determined for which the amplitude is $A_{\text{max}}/(\sqrt{2})$. The Q-factor can then be expressed as:

$$Q = \frac{f_R}{f_2 - f_1}$$  \hspace{1cm} (3.14)

Two observations should be made concerning this procedure to determine resonance frequency and Q-factor:

- Due to damping the measured resonance frequency $f_R$ differs from the actual resonance frequency $f_0$. $f_R$ is defined as the frequency at which the maximum amplitude occurs, whereas $f_0$ is defined as the frequency at which the phase shift between excitation signal and measured signal equals $\pi/2$. Eq. (3.15) shows that both are linked by the quality factor $Q$. In this work $Q$ always exceeds 100, which shows that the error committed by taking $f_R$ as resonance frequency is negligible.

$$f_0 = \frac{f_R}{\sqrt{1 - \left(\frac{1}{2Q^2}\right)}}$$  \hspace{1cm} (3.15)

- One could suggest that using a phase-locked loop in the experimental setup leads to better results. During such a procedure the phase $\phi$ is recorded as a function of angular frequency $\omega$. Both are related by Eq. (3.16), where $\omega_0$ is the angular resonance frequency. With the recorded couples $(\phi, \omega)$, Eq. (3.16) can be fitted for the values of $\omega_0$ and $Q$. The difference between this method and the one using the maximum amplitude is negligible as far as $f_0$ is con-
cerned (difference < 0.1%). Values of Q may differ by as much as 20%. For some modes measuring the amplitude in non-resonance regime is quite difficult and in these cases the phase locked loop has to be used.

\[
\varphi = \arctan \frac{\frac{\omega}{\omega_0}}{1 - \left(\frac{\omega}{\omega_0}\right)^2} \quad (3.16)
\]

After determining the resonance frequency it is important to check which mode belongs to which frequency. With the silicon specimens eight different modes are of interest. Five of those modes are 'beam-modes' (modes 1 to 5), which means that only the microbridge is deformed whereas the plate performs a rigid body motion. In this analysis the longitudinal mode has not been excited and measured. The other three modes concern deformations of the plates (modes 6 to 8). For the metallic specimens two more plate-modes can be measured (modes 9 to 10), see Fig. 3.5. The fact that for the LIGA specimens two more modes can be measured is due to their smaller plate thickness as compared to silicon specimens. In the frequency range considered, the modes 9 and 10 thus do not occur for silicon specimens due to their higher stiffness.

Fig. 3.5 shows the different modes as measured and calculated. For the calculated modes negative and positive amplitudes \( A \) are shown. The experimental modes only display absolute amplitude, i.e. no phase information is given by these pictures. The colour-coding is used to show the displacement perpendicular to the plane, with the exception of modes 2 and 5, where the displacement in direction of the beam axis is shown. The colour coding used is shown below.

<table>
<thead>
<tr>
<th>calculated modes:</th>
<th>experimental modes:</th>
</tr>
</thead>
<tbody>
<tr>
<td>3, 4, 6...10:</td>
<td></td>
</tr>
<tr>
<td>out of plane motion</td>
<td>out of plane motion</td>
</tr>
<tr>
<td>1:</td>
<td>0</td>
</tr>
<tr>
<td>2, 5: in plane motion</td>
<td>-A</td>
</tr>
<tr>
<td></td>
<td>A</td>
</tr>
</tbody>
</table>
Resonance experiments

Fig. 3.5  Resonance modes with dominating elastic constants
experiment and simulation
further explanations: please see preceding page
Using a scanning vibrometer the free vibrating plate is scanned for each resonance frequency and the displacement of about 250 points on the plate is recorded. By comparing these displacement profiles to the profiles known from finite element simulations, each mode with its corresponding frequency can be identified. Of course this procedure is only valid for modes where the displacement is perpendicular to the plates. Modes 2 and 5 don’t qualify, besides they cannot be excited by using the piezo element. Mode 5 was not excited during this work, which is of no further consequences. Mode 2 however can easily be excited by tapping slightly on the metal block. For this particular mode the laser beam does not hit the front of the plate, but its side. There are three methods to determine the corresponding frequency:

- Determine the frequency of the peak of the FFT of the output signal.
- Determine the frequency of the output signal with a frequency counter.
- A sinusoidal signal of known frequency is put on channel 1 of the oscilloscope. If the signal on channel 2 (output signal) is stable while triggering on channel 1, its frequency is the same as the frequency of the signal of channel 1.

All three methods have been tried and yield the same results within an error margin of 0.1%. The Q-factor for mode 2 is determined with the exponential decay of the amplitude over time.

### 3.4.2 Experimental results

To fix the minds first the influence of the experimental conditions, such as clamping and air pressure, shall be discussed.

The clamping realised with two different glues, even though it is not applied on exactly the same places, has no effect whatsoever on the resonance frequencies, a result which was expected after the FE simulations. This shows a general advantage of studying resonances of plates which are attached to a microbeam. The structure can be fixed outside the region which has to be studied, the microbeam acts like a filter as far as the influence of the clamping is concerned. The Q-factor shows no predictable behaviour, for some modes it increases, while it decreases for other ones.

The effect of the air pressure, as expected, is somewhat more spectacular. Concerning the silicon samples, the Q-factor increase between vacuum and normal pressure measurements lies between a factor 4 and 10 for the beam-modes. For
the plate-modes however no significant change can be detected. There are several main reasons for the damping and therefore the Q-factor of a system:

- Material related losses due to internal damping.
- Heat flow caused by expansion and compression.
- Damping due to clamping and boundary conditions.
- Viscous damping caused by air.
- Acoustic damping caused by air.

For the beam-modes the quality factor is strongly influenced by viscous and acoustic damping. As internal damping is supposed to be very small for single-crystal silicon, the plate-modes seem to be governed by damping due to clamping and to heat flow within the plates, see Table 3.3.

The LIGA specimens show no significant difference in Q-factor whether measured in air or in vacuum. It increases slightly for vacuum. As has already been shown before, there is no measurable significant difference between the resonance frequencies in air and in vacuum.

The measured frequencies for a silicon sample can be seen in Table 3.3, whereas the corresponding mode shapes are shown in Fig. 3.5.

<table>
<thead>
<tr>
<th></th>
<th>glue: HBM X60</th>
<th></th>
<th>glue: HBM X60</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>pressure: 1000 mbar</td>
<td></td>
<td>pressure: 0.3 mbar</td>
</tr>
<tr>
<td>mode 1</td>
<td>117.02 Hz</td>
<td>557</td>
<td>117.36 Hz</td>
</tr>
<tr>
<td>mode 2</td>
<td>214.34 Hz</td>
<td>3.564</td>
<td>213.37 Hz</td>
</tr>
<tr>
<td>mode 3</td>
<td>510.80 Hz</td>
<td>1702</td>
<td>511.58 Hz</td>
</tr>
<tr>
<td>mode 4</td>
<td>6.437.2 Hz</td>
<td>1.533</td>
<td>6.451.9 Hz</td>
</tr>
<tr>
<td>mode 6</td>
<td>80.750 Hz</td>
<td>3.230</td>
<td>80.766 Hz</td>
</tr>
<tr>
<td>mode 7</td>
<td>93.822 Hz</td>
<td>3.026</td>
<td>93.838 Hz</td>
</tr>
<tr>
<td>mode 8</td>
<td>166.360 Hz</td>
<td>978</td>
<td>164.710 Hz</td>
</tr>
</tbody>
</table>

Table 3.3  Resonance frequency and Q-factor for monocrystalline silicon samples

For the metallic samples, due to their different geometries, the height of the
microbridge is much bigger than its width and the height of the plate is the same as for the microbridge, the modes occur in a different order (with respect to frequency) as for silicon samples. The numbering of the modes is however related to the mode shapes and not to the frequencies of the different modes. Their frequencies are much lower and are usually in a bandwidth of 30 Hz - 40 kHz. Above 40 kHz finding or even measuring resonance frequencies gets more and more difficult. The signals get unstable, noisy and no clear peak in the amplitude can be detected. This phenomenon seems to slightly improve when working under vacuum conditions.

### 3.5 Mixed numerical experimental technique

#### 3.5.1 General information

From the experimental response of the system (resonance frequencies) its main characteristic parameters (elastic constants) have to be determined. This is a typical inverse problem which can be solved by mixed numerical experimental techniques (MNET). These techniques are increasingly popular with engineers and scientists, as the expanding computational power allows to tackle problems which were previously out of reach. The schematic working principle of all MNET is displayed in Fig. 3.6. An experiment is carried out under defined conditions (input) and some relevant response of the system is measured (output $\mu_E$). The experimental conditions are simulated by a numerical model which needs an initial trial set of parameters. The numerical and experimental output are compared and the parameter set is optimized by an algorithm which usually minimizes a cost function (also called error function). This new parameter set is then used in the simulation and this iterative process is repeated until some convergence criterion is met.
3.5.2 Theoretical background

The problem of determining the elastic constants when knowing the resonance frequencies is a non-trivial problem for several reasons:

- The geometries considered are not elementary (circular, rectangular, constant cross-section).
- One elastic constant may have an influence on the resonance frequencies of several modes, e.g., $E_{11}$.
- One mode may be influenced by several elastic constants, e.g., mode 10.

The problem can however be solved with MNET. For a given sample and a given set of parameters ($E_{11}$, $E_{22}$, $G_{12}$ and $G_{13}$) the corresponding resonance frequencies are numerically calculated by using finite elements. To understand why these four elastic constants are chosen, it is necessary to have a closer look at the relative sensitivity matrix in Appendix C. This matrix clearly shows that the constants $E_{33}$, $G_{23}$, $v_{23}$ and $v_{31}$ have no influence on any of the frequencies considered, as expected from theory. There are three constants, $E_{11}$, $E_{22}$ and $G_{12}$ which have a strong influence on at least one mode. Two more constants, $G_{13}$ and $v_{12}$ have only a weak influence on one mode. $G_{13}$ has an influence on mode 3, which is also influenced by $G_{12}$. There are however several modes where $G_{12}$ is
dominant. This allows an independent determination of $G_{12}$ and $G_{13}$. $v_{12}$ however cannot be determined independently from $E_{22}$, as both show their influence only on one and the same mode, mode 8. This means that the determination problem for $E_{22}$ and $v_{12}$ cannot be solved in a unique manner. One of the parameters has to be chosen a priori, in this case $v_{12}$, as its influence is weaker than the one of $E_{22}$.

After determining the resonance frequencies, the partial derivative of each frequency with respect to each parameter is numerically evaluated. The values of the frequencies and their derivatives allow the determination of the elastic constant in an iterative least-square procedure, which will be explained below:

- For a given sample the experimental resonance frequencies $\omega_{i,\text{Exp}}$ are measured ($1 \leq i \leq n$, where $n$ is the total number of different resonance modes).
- The sample is modeled using finite elements. Starting with trial values for the four prevailing parameters the $n$ resonance frequencies are numerically calculated. This yields the values $\omega_{i,\text{Num}}$.
- The difference between experimental and numerical values is denoted as $\Delta \omega_i = \omega_{i,\text{Exp}} - \omega_{i,\text{Num}}$. For each frequency this difference can be approximated using its total derivative, which yields:

$$
\begin{bmatrix}
\frac{\partial \omega_1}{\partial E_{11}} & \frac{\partial \omega_1}{\partial E_{22}} & \frac{\partial \omega_1}{\partial G_{12}} & \frac{\partial \omega_1}{\partial G_{13}} \\
\frac{\partial \omega_2}{\partial E_{11}} & \frac{\partial \omega_2}{\partial E_{22}} & \frac{\partial \omega_2}{\partial G_{12}} & \frac{\partial \omega_2}{\partial G_{13}} \\
\vdots & \vdots & \vdots & \vdots \\
\frac{\partial \omega_n}{\partial E_{11}} & \frac{\partial \omega_n}{\partial E_{22}} & \frac{\partial \omega_n}{\partial G_{12}} & \frac{\partial \omega_n}{\partial G_{13}}
\end{bmatrix}
\begin{bmatrix}
\Delta E_{11} \\
\Delta E_{22} \\
\Delta G_{12} \\
\Delta G_{13}
\end{bmatrix}
= 
\begin{bmatrix}
\Delta \omega_1 \\
\Delta \omega_2 \\
\vdots \\
\Delta \omega_n
\end{bmatrix}
$$

(3.17)

- As there are more equations (frequencies) than parameters, a cost function, which will be called $R$, has to be established. The minimizing of this cost function yields the values $\Delta E_{11}$, $\Delta E_{22}$, $\Delta G_{12}$ and $\Delta G_{13}$. $R$ is defined as the sum of the squares of the differences between $\Delta \omega_i$ and its total derivative, divided by $\omega_{i,\text{Exp}}$. 


40 Resonance experiments

\[ R = \sum_{i=1}^{n} \left( \frac{1}{\omega_i^{\text{Exp}}} \left( \Delta \omega_i - \sum_{j=1}^{4} \frac{\partial \omega_i}{\partial c_j} \Delta c_j \right) \right)^2 \]  \hspace{1cm} (3.18)

with \( c_j = E_{11} \ldots, G_{13} \) (for \( j = 1 \ldots, 4 \))

As the order of magnitudes of the frequencies differ quite much, a weight function has to be introduced, to guarantee that all frequencies have the same influence on the cost function, and by that means on the elastic constants. In Eq. (3.18) this weight function consists of \((1/\omega_i^{\text{Exp}})^2\). Without this cost function only the high frequencies, in this case the plate-modes, would have an influence on the determination of the constants.

The minimization of the cost function \( R \) with respect to \( \Delta E_{11}, \Delta E_{22}, \Delta G_{12} \) and \( \Delta G_{13} \), by putting

\[ \frac{\partial R}{\partial \Delta E_{11}} = 0 \quad \frac{\partial R}{\partial \Delta E_{22}} = 0 \quad \frac{\partial R}{\partial \Delta G_{12}} = 0 \quad \frac{\partial R}{\partial \Delta G_{13}} = 0 \]  \hspace{1cm} (3.19)

yields four equations for \( \Delta E_{11}, \Delta E_{22}, \Delta G_{12} \) and \( \Delta G_{13} \). To solve the equations the partial derivatives

\[ \frac{\partial \omega_i}{\partial E_{11}}, \frac{\partial \omega_i}{\partial E_{22}}, \frac{\partial \omega_i}{\partial G_{12}}, \frac{\partial \omega_i}{\partial G_{13}} \]  \hspace{1cm} (3.20)

have to be known. These derivatives are approximated by the finite differences

\[ \frac{\partial \omega_i}{\partial c_j}(..., c_j, ...) \equiv \frac{\omega_i(..., D + c_j, ...) - \omega_i(..., c_j, ...) }{D} \]  \hspace{1cm} (3.21)

In Eq. (3.21) the index \( i \) denotes the mode number, whereas the index \( l \) represents the iteration step. The resonance frequency is assumed to be a function of all four elastic constants, hence \( \omega_i = \omega_i(c_1, c_2, c_3, c_4) \).

The choice of \( D \) is precarious. By choosing too big a value, the derivatives are badly approximated and the iteration takes longer to converge. Too small a value can induce numerical problems. In the simulations considered in this
work, D was given a value of 3 GPa.

- Added to the starting values $E_{11}^1, E_{22}^1, G_{12}^1, G_{13}^1, \Delta E_{11}, \Delta E_{22}, \Delta G_{12}$ and $\Delta G_{13}$ yield the new improved set of parameters:

$$c_{j+1}^i = c_j^i + \Delta c_j$$ (3.22)

- The relative error in Eq. (3.23) between experimental and numerical frequencies gives a good survey and control of the iteration.

$$v_{rel} = \sum_{i=1}^{n} \left( \frac{\Delta \omega_{i}}{\omega_{i,Exp}} \right)^2$$ (3.23)

It should decay asymptotically with each step. With each step the cost function $R$ approaches the relative error, as $\Delta c_j$ gets smaller with each step. If the difference between the relative errors and the elastic constants in two consecutive iterative steps does not exceed a given value, convergence is assumed and the iteration is stopped.

### 3.5.3 Results

The iterative process described in the previous subsection is applied to three silicon samples and several metallic specimens. In a first subsection the detailed study of the first silicon sample will be reported. In a second subsection the general results for the remaining specimens and materials are presented.

#### Silicon sample s11

A first iteration is performed using all seven available frequencies. During the simulations, it is soon obvious that mode 4 leads to erroneous results, as the difference between the experimental and numerical resonance frequency is about 10%. As mode 4 is mainly influenced by $E_{11}$ (see Appendix C), which can be determined by other modes, its non-consideration in all further simulations is of no consequence. The fact that mode 4 leads to bad results (big difference between experimental and numerical resonance frequency, high relative error) has not yet been fully understood. There may be several reasons for this behaviour:
• The measured frequency is wrong. This can be excluded as measurements on a perfectly similar sample yielded the same frequency.
• The numerical simulation of this mode is not as good as it should be. It is however astonishing that this seems to be the only mode which is affected by this impediment.
• The mesh of the numerical model is not fine enough for this mode. Table 3.2 shows however that a further refinement has almost no consequences on the computed frequencies.
• The dimensions of the specimen are wrong. This is improbable, as the dimensions have been measured several times. Furthermore, if the dimensions were wrong, this should also have a strong effect on several other modes.
• The frequency has been associated to the wrong mode. As the frequency and the mode shape are measured at the same time and compared to the simulation, this cannot be the reason for the strange behaviour of mode 4.

Mode 4 will therefore be omitted in all future iterations for this specimen as well as for all the other ones.

The iteration is started with the following values:
\[ E_{11} = E_{22} = 150 \text{ GPa} \]
\[ G_{12} = G_{13} = 50 \text{ GPa} \]

After five iterations the procedure yields the following results:
\[ E_{11} = 131.4 \text{ GPa, } E_{22} = 117.4 \text{ GPa} \]
\[ G_{12} = 70.5 \text{ GPa, } G_{13} = 80.7 \text{ GPa} \]

The relative error at that moment amounts to \( 28 \times 10^{-3} \). \( E_{11} \) and \( G_{13} \) are in very good agreement with the expected values, given by Büttgenbach [6], the error is less than 1.5%. \( E_{22} \) and \( G_{12} \) have a bigger error of 10%, but they lie still within an acceptable error margin as will be shown by the error calculus in Section 3.6. It should be mentioned that the predicted value of these two constants is in both cases too low. Fig. 3.7 shows the evolution of the moduli, the resonance frequencies and the relative error in function of the iteration steps.
Fig. 3.7 Evolution during the iteration of
a) elastic constants
b) resonance frequencies of beam modes
c) resonance frequencies of plate modes
Table 3.4 lists the values of experimental and numerical resonance frequencies as well as the relative error between both. The differences are below 1%, only for mode 1 and mode 6 do they lie between 2 and 3%.

<table>
<thead>
<tr>
<th>Mode</th>
<th>Experimental $\omega_{res}$</th>
<th>Numerical $\omega_{res}$</th>
<th>$\Delta (%)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>mode 1</td>
<td>752.1</td>
<td>731</td>
<td>-2.8</td>
</tr>
<tr>
<td>mode 2</td>
<td>1360.3</td>
<td>1368</td>
<td>0.6</td>
</tr>
<tr>
<td>mode 3</td>
<td>3288.6</td>
<td>3279</td>
<td>-0.3</td>
</tr>
<tr>
<td>mode 6</td>
<td>502655</td>
<td>513320</td>
<td>2.1</td>
</tr>
<tr>
<td>mode 7</td>
<td>576231</td>
<td>577550</td>
<td>0.2</td>
</tr>
<tr>
<td>mode 8</td>
<td>1029751</td>
<td>1029100</td>
<td>-0.1</td>
</tr>
</tbody>
</table>

Table 3.4  Comparison between experimental and numerical frequencies for a silicon sample

The more parameters are introduced, the better the fit. This does not mean that the values thus obtained are more correct, it just says that the relative error gets smaller. To demonstrate the influence of the different number of constants a new iteration is launched for the same sample, this time including only two free parameters $E_{11}$ and $G_{12}$. $E_{22}$ and $G_{13}$ are set equal to $E_{11}$ and $G_{12}$, respectively. This yields the following results:

$E_{11} = E_{22} = 127.4$ GPa  
$G_{12} = G_{13} = 71.6$ GPa

A comparison between these values and the ones found previously shows the influence of each constant. For the determination of the shear-modulus modes 3 and 7 are dominant. Mode 7 is exclusively determined by $G_{12}$. Due to the geometry of the microbridge, its width being larger than its height, mode 3 too is mostly determined by $G_{12}$. That’s why the value of 71.6 GPa is much closer to $G_{12} = 70.5$ GPa than to $G_{13} = 80.7$ GPa. Things are similar when determining Young’s modulus. $E_{11}$ has an influence on three different modes, 1, 2 and 6 whereas $E_{22}$ just plays a role with mode 8. This ratio of 3:1 explains why the value of 127.4 is closer to $E_{11} = 131.4$ GPa than to $E_{22} = 117.4$ GPa.
Silicon

Besides the sample discussed in the above section, the experiment was carried out on two more silicon samples. Averaging yields the following elastic constants:

\[ E_{11} = 132.9 \text{ GPa}, \ E_{22} = 117.4 \text{ GPa} \]
\[ G_{12} = 71.8 \text{ GPa}, \ G_{13} = 81.2 \text{ GPa} \]

Three samples are of course not enough to carry out a relevant statistical analysis, it is nevertheless interesting to compare the individual values with the averaged ones. The deviation from average lies below 4% for \( E_{11}, \ E_{22} \) and \( G_{12} \), whereas it may get as high as 25% for \( G_{13} \). This shows that the accuracy for \( G_{13} \) is much worse than for the other constants. It has to do with the fact that \( G_{13} \) only partially influences one mode, mode 3, which is mainly determined by \( G_{12} \). The fact that \( G_{13} \) can only be determined inaccurately can be shown by creating a finite element model, whose height exceeds the measured height by 1 \( \mu \text{m} \). The difference between the original model and the altered one lies at about 30% for \( G_{13} \), whereas it remains below 2% for all the other constants.

LIGA specimens

The experiments and the numerical procedure are carried out for eight metallic samples made of five different materials. A comparison between experimental and numerical resonance frequencies is shown in Table 3.5.

<table>
<thead>
<tr>
<th>mode</th>
<th>exp. ( \omega_{\text{res}} )</th>
<th>num. ( \omega_{\text{res}} )</th>
<th>( \Delta ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1190.7</td>
<td>1203.7</td>
<td>1.1</td>
</tr>
<tr>
<td>2</td>
<td>382.0</td>
<td>384.3</td>
<td>0.6</td>
</tr>
<tr>
<td>3</td>
<td>1550.1</td>
<td>1545.8</td>
<td>-0.3</td>
</tr>
<tr>
<td>6</td>
<td>94763</td>
<td>94349</td>
<td>-0.4</td>
</tr>
<tr>
<td>7</td>
<td>106205</td>
<td>103620</td>
<td>-2.4</td>
</tr>
<tr>
<td>8</td>
<td>149653</td>
<td>150050</td>
<td>0.3</td>
</tr>
</tbody>
</table>

Table 3.5 Comparison between experimental and numerical frequencies for a metallic sample made of NiFe20%
The general results for all the materials can be viewed in Table 3.6. The elastic constants as obtained with resonance experiments are compared with values from static tests, both torsional (see chapter 4) and tensile, Mazza (internal report 1997). For the dynamic calculations the densities assumed are also listed in Table 3.6. Some general comments have to be made concerning Table 3.6. The values of $G_{12}$ of the metallic specimens as determined with static torsional tests have an accuracy of about only 50% (see Table 4.5). This has to do with the geometry of the samples and the resulting poor sensitivity of the identification method to $G_{12}$. The values of $E_{11}$ of dynamic and static tests are in good agreement, the difference in each case being less than 5%. No comparison can be made for $E_{22}$ as it can only be determined by dynamic measurements. Torsional and dynamic tests concerning $G_{13}$ are in good agreement for Si (2% difference) and Ni (7% difference) as well as for NiFe(53%Fe), but in poor agreement for NiFe(20%Fe). The only valid explanation for this phenomenon is that the specimens used in both tests may have been manufactured in different processes, resulting in different mechanical properties. Fig. 3.8 shows the evolution of the elastic moduli of NiFe alloys in function of the content of iron. The Young’s moduli decrease with increasing iron percentage. No clear trend is found for the shear moduli.
<table>
<thead>
<tr>
<th>material</th>
<th>method</th>
<th>$E_{11}$</th>
<th>$E_{22}$</th>
<th>$G_{12}$</th>
<th>$G_{13}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si $\rho = 2329$</td>
<td>res. tensile torsion</td>
<td>132.9</td>
<td>117.4</td>
<td>71.8</td>
<td>81.2</td>
</tr>
<tr>
<td>Ni $\rho = 8900$</td>
<td>res. tensile torsion</td>
<td>209.3</td>
<td>177.6</td>
<td>68.5</td>
<td>54.9</td>
</tr>
<tr>
<td>NiFe(13%) $\rho = 8750$</td>
<td>res. tensile torsion</td>
<td>179</td>
<td>165.3</td>
<td>65.4</td>
<td>67.2</td>
</tr>
<tr>
<td>NiFe(20%) $\rho = 8600$</td>
<td>res. torsion</td>
<td>163.5</td>
<td>172.1</td>
<td>59.4</td>
<td>62.0</td>
</tr>
<tr>
<td>NiFe(27%) $\rho = 8500$</td>
<td>res. torsion</td>
<td>174</td>
<td></td>
<td>41</td>
<td>65</td>
</tr>
<tr>
<td>NiFe(53%) $\rho = 8200$</td>
<td>res. tensile torsion</td>
<td>135</td>
<td>114</td>
<td>40</td>
<td>56</td>
</tr>
</tbody>
</table>

Table 3.6 Elastic constants [GPa] of monocrystalline silicon and metallic microstructures, as determined with various tests, $\rho$ [kg/m³]

![Graph showing elastic moduli vs iron content](image)

Fig. 3.8 Elastic moduli of NiFe-microstructures in function of iron content
3.6 Error calculus

These results seem to be quite reasonable and trustworthy, but before jumping to a premature conclusion, the errors accumulated during the process should be carefully analysed. This will be done in this section.

Under the assumption that the finite element modelling bears no inherent errors and is accurate, the error in the determination of elastic moduli is given by the errors committed while measuring the frequency and the errors committed while measuring the dimensions of each sample. It can be said that each elastic constant is a function of the measured frequencies, as well as of the geometries, a fact which is expressed in Eq. (3.24).

\[
E_i = E_i(\omega_1, ..., \omega_8, l_B, w_B, h_B, l_P, w_P, h_P, w_M) \quad (3.24)
\]

\(w_M\) is the width of the membrane, the other constants have been explained in section 2.

The procedure to determine the actual relative error of each elastic modulus of each sample consists of several steps:

- The sensitivity of the resonance frequencies with respect to the geometrical features is determined numerically. To do so several models of the same sample are generated from an original model. The geometrical features are changed one by one and the corresponding resonance frequencies are calculated. As there are seven geometrical characteristics and six frequencies (in the case of silicon), this yields a 7x6 matrix \(D\), where \(\omega_j\) represents a resonance frequency and \(l_i\) a characteristic dimension.

\[
D = \begin{bmatrix}
\cdots & \cdots & \cdots \\
\frac{l_i \cdot \partial \omega_j}{\omega_j \cdot \partial l_i} & \cdots \\
\cdots & \cdots & \cdots 
\end{bmatrix}
\quad (3.25)
\]

- The relative errors in the dimensions have to be estimated, yielding a seven-dimensional vector \(d\):
Applying the matrix $D$ on vector $d$ gives rise to a vector $m$, whose components characterize the relative error in the resonance frequencies due to inaccurate dimensions for a given mode.

In a next step the sensitivity of the elastic moduli with respect to the resonance frequencies is determined numerically. This yields a $6 \times 4$ matrix $R$, where $E_j$ represents an elastic modulus.

Besides errors due to dimensions the error due to inaccurate measuring of the resonance frequency has to be quantified for each mode. This is done by the vector $w$, which has six components as $m$.

The total relative error for each mode is given by $r = m + w$.

The final step consists in establishing the relative error for each constant, by applying the matrix $R$ on the vector $r$. This is a delicate operation, because it has to take into account that for the determination of an elastic modulus not all the modes are of the same importance, e.g. $E_{22}$ is almost solely determined by mode 8. If all the modes were respected in the same manner this would lead to wrong error estimations. To take into account this fact a weight function has to be introduced, which is given in (3.28) by $1/R_{ij}^2$. 

\[
\mathbf{d}^T = \begin{bmatrix} \Delta l_B / l_B, & \Delta h_B / h_B, & \ldots, & \Delta h_p / h_p, & \Delta w_M / w_M \end{bmatrix}
\]

\[
R = \begin{bmatrix} \cdots & \cdots & \cdots \\ \cdots & \frac{\omega_1 \cdot \partial E_j}{E_j \cdot \partial \omega_i} & \cdots \\ \cdots & \cdots & \cdots \end{bmatrix} \quad (3.26)
\]

\[
\mathbf{w}^T = \begin{bmatrix} \Delta \omega_1 / \omega_1, & \Delta \omega_2 / \omega_2, & \Delta \omega_3 / \omega_3, & \Delta \omega_6 / \omega_6, & \Delta \omega_7 / \omega_7, & \Delta \omega_8 / \omega_8 \end{bmatrix} \quad (3.27)
\]
This systematic approach of the error estimation has been applied for both silicon and metallic specimens.

The relative errors of the frequency measurement are given by Eq. (3.29) and are more or less the same for silicon and metallic samples. The relative dimensional errors are listed in Table 3.7. The total relative error for each modulus is given by Table 3.8.

\[
\frac{\Delta E_i}{E_i} = \frac{\sum_j \frac{1}{R_{ij}^2} \cdot R_{ij} \cdot r_j}{\sum_j \frac{1}{R_{ij}^2}} \quad (3.28)
\]

\[
\left| \frac{\Delta \omega_i}{\omega_i} \right| \leq \begin{cases} 
0.003 (i \to 1, 2, 3, 6) \\
0.03 (i \to 7, 8, 9, 10)
\end{cases} \quad (3.29)
\]

<table>
<thead>
<tr>
<th></th>
<th>$\Delta l_B/l_B$</th>
<th>$\Delta w_B/w_B$</th>
<th>$\Delta h_B/h_B$</th>
<th>$\Delta l_p/l_p$</th>
<th>$\Delta w_p/w_p$</th>
<th>$\Delta h_p/h_p$</th>
<th>$\Delta \rho/\rho$</th>
</tr>
</thead>
<tbody>
<tr>
<td>silicon</td>
<td>1/220</td>
<td>1/80</td>
<td>1/100</td>
<td>1/100</td>
<td>1/100</td>
<td>3/380</td>
<td>0</td>
</tr>
<tr>
<td>LIGA</td>
<td>1/1000</td>
<td>1/40</td>
<td>1/100</td>
<td>1/6000</td>
<td>1/5000</td>
<td>3/100</td>
<td>1/80</td>
</tr>
</tbody>
</table>

Table 3.7 Relative dimensional errors for silicon and LIGA samples

<table>
<thead>
<tr>
<th></th>
<th>$\Delta E_{11}/E_{11}$</th>
<th>$\Delta E_{22}/E_{22}$</th>
<th>$\Delta G_{12}/G_{12}$</th>
<th>$\Delta G_{13}/G_{13}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>silicon</td>
<td>0.10</td>
<td>0.16</td>
<td>0.15</td>
<td>0.75</td>
</tr>
<tr>
<td>LIGA</td>
<td>0.10</td>
<td>0.18</td>
<td>0.18</td>
<td>0.15</td>
</tr>
</tbody>
</table>

Table 3.8 Total relative error for the elastic constants of microstructures

In the case of silicon, the theoretical relative error for $E_{11}$, $E_{22}$ and $G_{12}$ lies between 10% and 16%. $G_{13}$ has a relative error of 75%, due to the fact that $G_{13}$
influences only one mode, whose frequency is mainly dominated by $G_{12}$. The errors in Table 3.8 should be interpreted as upper limits of the errors. As the results for the silicon specimens show, the errors are usually far less important. Due to their geometry the LIGA specimens are better suited to determine the four constants than their silicon counterparts. Their relative errors are smaller and always below 18%.

### 3.7 Higher harmonic oscillations

#### 3.7.1 General observations

While trying to detect the resonance frequencies of the microstructures, superharmonic oscillations were detected during the experiment. Superharmonic oscillations are oscillations of the following kind: while exciting at a frequency of $f_1/2$ the system oscillates at the frequency $f_1$, which is a main resonance frequency. This special case can be called a double harmonic excitation. With the structures analysed not only double harmonic excitation (DHE) occurs but multiple harmonic excitation (MHE), up to ten-fold. A four-fold harmonic excitation measured with a silicon sample at its first bending mode is shown in Fig. 3.9.

![Excitation and Oscillation](image)

**Fig. 3.9** Four-fold-HIE of a Si-sample with a main resonance frequency of 117 Hz.

The MHE is not an isolated phenomenon, which was accidentally observed one time:
• MHE occurs with each specimen, LIGA and silicon.
• MHE occurs when the samples are excited using a piezoceramic element or an electromagnetic coil (as in the case of NiFe alloys).
• MHE were found as well in vacuum as at normal pressure.
• Several types of glue were used to fix the sample on the piezo element. MHE could be detected independent of the glue used.
• MHE occur in a frequency range of 100 Hz - 100 kHz.
• For a given mode, MHE of any order occur, two-fold, three-fold, etc. The intensity of the MHE diminishes with increasing order.

These observations cannot be explained by a linear elastic model of the specimen excited by a purely sinusoidal excitation. There are two possible explanations:
• Some part of the mechanical system has a non-linear behaviour.
• The actuation signal coming from the function generator itself has higher harmonics.

3.7.2 Experiments

A quantitative analysis of the phenomenon was made on two specimens, a silicon and a LIGA sample. For each sample one mode and its multiple HE were analysed. The experimental setup is shown in Fig. 3.10. The general idea of these measurements is to analyse the amplitude spectrum of each signal, the signal from the function generator, the signal after the amplifier and the signal from the laser demodulator. By comparing these signals to one another, deductions can be made concerning the linearity of the system and the possible sources of non-linearity. To do so a very high resolution is needed to detect even weak signals (μV range). The 8-bit resolution of the oscilloscope used was insufficient for these purposes. Therefore a digital lock-in amplifier (Stanford Research Systems SR 850) was used for the analysis of the signals. The digital lock-in amplifier also allows the precise measurement of higher harmonics. The TTL signal from the function generator was used as reference signal for the lock-in.
Before going into the details of the experiments, the indices convention of this section should be explained. A subscript numerical index indicates the order of the harmonic. The first harmonic is defined to be the main frequency of the output of the function generator. The subscript letter R means resonance. For example $A_{R2}$ indicates the amplitude at the second harmonic, which is at the same time the resonance frequency. The voltage $U$ is always the voltage at the output of the amplifier, thus $U_3$ means the voltage of the third harmonic after the amplifier.

**Experiments on LIGA sample**

The experiments were performed on the first flexural beam mode of a NiFe sample. The resonance frequency is $f_R = 239.78$ Hz with a Q-factor of $Q = 100$. The displacements were measured at the lower left corner of the oscillating plate. Only two-fold HE could be measured, the higher order MHE were too weak to be detected. Fig. 3.11 shows the amplitude at main and at DHE in function of the output signal of the amplifier at $f_R = 239.78$ Hz. The linear behaviour at main resonance shows that the piezo behaves linearly at this frequency and these voltages.
A comparison between the voltage $U_2$ of the second harmonic and the amplitude $A_{R2}$, see Fig. 3.12, clearly shows that no relation can be established between both variables. This means that higher harmonics are not responsible for the non-linear behaviour at DHE.

**Experiments on silicon sample**

The third flexural beam mode of a silicon sample is studied. Its resonance frequency $f_R = 6614.4$ Hz with $Q = 2500$. The displacements were measured on the
plate glued to the piezo element (point \( P' \)) and on the free vibrating plate (point \( P \)), see Fig. 3.13.

![Fig. 3.13 Points at which the amplitude is measured for silicon specimens](image)

It was also possible to measure triple HE. HE of different order are shown in Fig. 3.14. From the behaviour at main resonance it can again be seen that the piezo element has a linear characteristic in these particular conditions. Furthermore it can be seen that in all three cases the relation between the displacements of \( P \) and \( P' \) is linear. This means that no non-linear effects take place in the specimen itself.

![Fig. 3.14 Resonance amplitude at main, double and triple HE at two points P and P’](image)

By comparing the higher harmonics of the amplifier output to the amplitudes at the MHE (Fig. 3.15), a linear relation can be found. This indicates that in this case the MHE are due to higher harmonics in the excitation signal.
Resonance experiments

3.7.3 Theoretical interpretation

To have access to an analytical and theoretical analysis of the phenomenon, some simplifications have to be made. Under the assumption that the bandwidth $\Delta f$ is small as compared to the gap between two resonance frequencies, Eq. (3.30), the oscillating continuum can be described by a damped mass spring system.

$$\Delta f = \frac{f_R^n}{Q} \ll \left| f_R^n - f_R^{n-1} \right| \wedge \left| f_R^n - f_R^{n+1} \right|$$

Eq. (3.30) is fulfilled in both considered cases, as well for the LIGA as for the silicon sample.

LIGA sample

As has been seen in the experiments, some non-linear effect takes place in this mode. The corresponding dimensionless differential equation which describes the behaviour of the system, can be written in the form:

$$b \cdot \ddot{x}_\Theta + c \cdot \dot{x}_\Theta + x + \varepsilon \cdot h(x_\Theta, \dot{x}) = a \cdot \sin \Theta$$

(3.31)
where $h$ is a nonlinear function of $\bar{x}$ and $\bar{x}_0$, $\bar{x}$ and $\Theta$ are dimensionless parameters defined in Eq. (3.32). $x_{\text{unit}}$ can be arbitrarily chosen, but should have the same order of magnitude as the amplitude. $\omega$ represents the circular frequency.

$$\bar{x} = \frac{x}{x_{\text{unit}}}, \quad \Theta = \omega \cdot t \quad (3.32)$$

Further explanations of the different coefficients as well as the solving method for these non-linear differential equations are given in appendices $E$ and $F$. In this special case it makes sense to assume the function $h$ to be quadratic, as the DHE is very strong as compared to triple HE, and as the amplitude curve shows a quadratic behaviour. This yields the following differential equation:

$$b \cdot \bar{x}_\Theta + c \cdot \bar{x}_0 + \bar{x} + \varepsilon \cdot \bar{x}^2 = a \cdot \sin \Theta \quad (3.33)$$

Solving Eq. (3.33) to the order $\varepsilon^2$ for $\bar{x}$ yields:

$$\bar{x} = \left[ A_{11} \sin \Theta + A_{12} \cos \Theta \right] + \varepsilon \left[ A_{21} + A_{22} \sin (2 \Theta) + A_{23} \cos (2 \Theta) \right] + \varepsilon^2 \left[ A_{31} \sin \Theta + A_{32} \cos \Theta + A_{33} \sin (3 \Theta) + A_{34} \cos (3 \Theta) \right] + O(\varepsilon^3) \quad (3.34)$$

The coefficients $A_{ij}$ can be expressed in function of $a$, $b$ and $c$, see appendix D. The amplitude of the $\Theta$-terms and $2\Theta$-terms, neglecting terms with $\varepsilon^2$, can be written as:

$$A_\Theta = \sqrt{A_{11}^2 + A_{12}^2} = \frac{a}{\sqrt{(1-b)^2 + c^2}} \quad (3.35)$$

$$A_{2\Theta} = \sqrt{A_{22}^2 + A_{23}^2} = \varepsilon \frac{a^2}{2[(1-b)^2 + c^2] \sqrt{(1-4b)^2 + (2c)^2}} \quad (3.36)$$

The coefficients $a$, $b$ and $c$ can be determined by considering main resonance, which yields $b = 1$ and $c = 1/Q = 0.01$. These values of $b$ and $c$ are only valid at resonance frequency. Their values at arbitrary frequencies can now however be calculated by keeping in mind the fact that $b$ is a quadratic function of $\omega$ and $c$ a
linear one. Choosing $x_{\text{unit}} = 1 \mu m$ and fitting the experimental curve at main resonance leads to the relation between dimensionless excitation amplitude $a$ and the applied voltage (given in Volt) $U_1$ given by Eq. (3.37)

$$a = \frac{A_{R1}}{Q \cdot x_{\text{unit}}} = 4.41 \times 10^{-3} \frac{U_1}{x_{\text{unit}}}$$

Fitting the experimental curve at DHE, $\varepsilon$ can be found to be $\varepsilon = 0.04$, which is small compared to the factors of the linear terms. Introducing the values of $a$, $b$, $c$ and $\varepsilon$ in Eq. (3.34) and solving of the system yields the equations for the amplitudes shown in Fig. 3.16.

![Figure 3.16](image.png)

Fig. 3.16 Experimentally determined and calculated values of the amplitudes at main, double and triple HE

The good agreement between experimental and theoretical values at main and DHE is not astonishing, as the governing equation parameters were chosen so
that the computed curves would fit the experimental ones. It is however important to see that it is possible to describe the behaviour of the system by choosing a non-linear approach. This approach yields very low amplitudes at triple HE, which explains why they could not be measured in the experiment. The dotted line in Fig. 3.16 shows the theoretical amplitude at DHE if a linear assumption were made, as discussed in the following subsection for the silicon samples.

The non-linear behaviour of the system cannot be explained by geometric or material non-linearities, as the deformations remain small and the static stress-strain curve was determined to be linear. One possible explanation might be non-linear effects of the clamping, as the glue used may have non-linear effects. A more detailed analysis is however needed to be able to draw conclusions. This analysis will not be provided in this work.

**Silicon samples**

According to the experiments, a linear model is used to describe the behaviour of the sample at resonance. The corresponding differential equation can be written in the form of:

\[
b \cdot \ddot{x} + c \cdot \dot{x} + x = \sum_{n=0}^{\infty} a_n \cdot \cos(n\Theta)
\]  

A linear regression at main resonance yields the relation between amplitude (in nm) and input voltage (in V):

\[A_{R1} = 73.5 \cdot U_1 \]  

As the system is assumed to be linear this relationship holds also at MHE, see appendix D:

\[A_{Rn} = 73.5 \cdot U_n \]  

The experimental and theoretically expected values, as determined by Eq. (3.40) are shown in Fig. 3.17. It can be seen that the experimental values at MHE are higher than expected. This can be explained by the fact, that the values of the
input signal and the amplitudes were not measured at the same time. Besides there may still be some weak non-linear effect causing the difference between experimental and theoretical value.

![Graph showing comparison between experimental and theoretical values at main, double and triple HE](image)

By comparing the values of the amplitudes at the points P and P', it can be shown that in any case the non-linear effects are not caused by the specimen itself, as there is a constant linear relation between the amplitudes $A_{Rn}(P)$ and $A_{Rn}(P')$:

$$A_{Rn}(P) = \kappa \cdot A_{Rn}(P')$$  \hspace{1cm} (3.41)
3.8 Conclusion and outlook

The inverse problem of determining material constants from resonance frequencies is solved with the help of a mixed numerical experimental technique. The procedure makes use of an iterative least squares procedure, minimizing the error between measured and simulated resonance frequencies. To be sure to make the right mapping of experiment to simulation, not only the frequencies but their corresponding modes as well are compared.

The materials used, silicon and metallic samples, are modelled as orthotropic. Theoretical considerations show that the geometries, boundary and loading conditions applied in this work, allow the determination of five constants, namely, $E_{11}$, $E_{22}$, $G_{12}$, $G_{13}$, $\nu_{12}$. Due to a problem of uniqueness, which originates in the geometries used and the modes considered, in fact only four constants can be computed, while an assumption has to be made for the remaining constant, in this case Poisson's ratio $\nu_{12}$. The values found for silicon specimens are in good agreement with values known from bulk material or from static tests on microstructures. It could be shown that metallic samples have an anisotropic behaviour.

The inaccurate geometry measurement is identified as the main error source. The relative errors can be drastically reduced by an optimized geometry. The geometry should be chosen so that there is at least one mode which depends strongly on one of the parameters. As the final error is mostly influenced by the dimension measurements, some more effort has to be made in this direction. Image processing algorithms are a promising approach. The image processing software, able to automatically recognize geometrical features, uses a calibrated standard to determine the geometrical dimensions.

As has been mentioned, the frequencies can be determined with high accuracy, whereas the dimensions of the samples are more erroneous. In a further step it is imaginable to use resonance frequency measurements to determine the dimensions of a microstructure when the elastic constants are known.

It was shown that the multiple harmonic excitations occurring with microstructures are due to different causes, once to higher harmonics in the excitation signals, and once to non-linearities in the mechanical setup. Further studies have to be made to determine the causes of the non-linearity.
M.C. Escher
Ascending and Descending
lithograph 1960
4 Torsion of microstructures

4.1 Previous work

Except for the work of Saif and Mac Donald [55], previously little work has been done on torsional tests with microstructures.

In their work, the test sample consists of a pillar of varying rectangular cross section of about $1 \times 1 \, \text{µm}^2$. It has a length of 10 µm and is made of single crystal silicon. During the fabrication process a lever is fixed at the upper end of the pillar. Using a micromechanical electrostatic comb drive, a loading device is actuated, which deflects the lever, supposed to be rigid. Due to the asymmetric setup, torsion as well as bending is induced in the sample. It is not possible to perform purely torsional tests. Besides this mixed-mode loading condition there are two main points of criticism of their work:

- As the test sample and the loading device are fabricated on the same substrate in the same step, and due to the narrow margin between both entities, the loading device cannot be calibrated. Only a similar loading device without the test sample can be calibrated by the deflection of two AFM cantilever beams. So the torque acting in the specimen is not measured directly, whereas the total angle of twist is recorded from the rotation of the lever arm.
- Though the cross section of the specimen varies over its length, classical theory for constant cross sections is used to calculate the resulting maximum shear stress at the side of the pillar. By a superposition of stresses due to bending and torsion, fracture stresses of about 20 GPa are found. Stress concentrations occurring at the notches and causing the failure of the specimen are not taken into account.
4.2 Torque measurement principles

4.2.1 General remarks

For the torsional tests it is essential to measure not only the angle of twist but also the corresponding torque, to be able to make assertions about the material behaviour. Based on the sample geometry, the torque sensor to be used has to fulfil several criteria, which will now be enumerated below:

- Resolution: 0.5 μNm
- Accuracy: 5% of measured value
- Working range: -100...+100 μNm
- Other loading modes, like tension and bending, have no influence on the measured torque
- Small temperature changes (≤ 2° C) have no influence on the measurement
- Easy fixation of the test specimens without damaging the sensor
- Easy removal of the destroyed sample
- Possibility of either dynamic or static calibration

There are several possibilities to measure the torque acting in an axis. Three main groups can be distinguished.

In the first group, the torque acts on a rigid but movable structure. The structure is held in place by some kind of bearings and is in contact with force sensors. Knowing the distance between the force sensors, the torque is readily calculated from differential force measurement. An example can be seen in Fig. 4.1.

The second group consists of sensors made up by an elastic body, which is deformed by the acting torque, and of some kind of measurement apparatus, able to detect and quantify the deformation. It usually yields an electric signal proportional to the torque. The elastic body exists in many shapes and geometries and is either deformed by bending or by torsion. Now either the strain or the displacement at some point of the structure can be measured, using for example strain gauges, inductive or capacitive sensors or some optical means. A sensor belonging to this category can be seen in Fig. 4.2.

A control loop is the main feature of the third group. Again an elastic body is subjected to a torque $T_1$, and the resulting deformation generates an electric signal in
a displacement or strain sensor, usually a capacitance. This signal $s$ is amplified and after some more processing used as input $i$ for a construction which generates a torque $T_2$ compensating $T_1$. The input $i$ is at the same time the output of the sensor and should be proportional to the torque. The control loop ensures that the signal $s$ remains at a minimum. This approach has now been used for some years by physicists to study the magnetization of high-$T_c$ superconductivity materials, see Condon and Marcus [8] as well as Farrell [15] and Qvarford et al. [54]. They are able to measure torques as low as 10 pNm.

Sensors belonging to the first and second category have been studied and partially used in this work. Their mechanical design as well as their characteristics will be presented.

### 4.2.2 Sensor based on differential force measurement

As the initial goal of this work was the study of the mechanical properties of microstructures under torsional load and not the design of a micro-torque sensor, the first sensor used was a commercially available one, designed and described by Gass et al. [18]. A schematic representation is shown in Fig. 4.1. Via a spring blade and an axis the torque is converted to two forces which are measured piezoresistively. The axis itself is mounted into two ruby bearings which are attached to a brass frame.

Fig. 4.1 Working principle of differential force measurement sensor
The force sensors are preloaded, which means that an applied torque generates a decrease of force in sensor 1 and an increase in sensor 2. The differential force measurement has the advantage to eliminate forces due to bending of the axis. Several experiments on microstructures were run with this sensor, yielding only partially satisfactory results. The main drawbacks and points of criticism will now briefly be listed.

- Though the sensor has a specified resolution of 0.3 μNm at a working range of ±280 μNm, the torques measured experimentally lie systematically 10% below the torques expected.
- A drift of about 0.2 μNm/s can be observed, due to thermal effects.
- Changes of the longitudinal force acting on the sensor axis have a detrimental influence on the torque measurements. The torques measured at different loads differ and the sensor behaves nonlinearly. Besides the nonlinearity a strong hysteresis can always be seen in these experiments.

These anomalies are all due to the fact that friction occurs in the bearings, where tensile and shear forces are generated if the torque axis is under tensile or bending loads. This shows that in order to measure small forces or small torques, it is of the utmost importance to have a frictionless system.

### 4.2.3 Differential inductive sensor

The differential measuring principle is a good, if not the only, way to make torque measurement independent of bending, and will therefore be applied in the other sensors studied.

This sensor consists of a cylinder attached to a ring by four membranes, see Fig. 4.2. The cylinder as well as the ring are rigid as compared to the steel membranes (spring blades), which have a thickness of 50 μm. The torque acting on the cylinder is converted to bending in the membranes. The resulting displacements at two different points are measured by inductive resonant sensors. Each displacement sensor is made of a coil (inductivity L), a capacitance C and a resistance R in series, thus defining an RLC-circuit. L is dependent on the gap between coil and membrane, thus due to the displacement δ of the membrane the inductivity of the coil changes, whereas C and R remain constant. The resonance frequency $f_R$ of the circuit is a square root function of the displacement δ, which is directly proportional to the torque T. $f_R$ can be measured with high accuracy in a phase locked loop. The inductive resonant sensor developed in this work, used in
combination with a membrane of 50 μm thickness, and integrated in a differential setup, has the following characteristics:

- Resolution: 100 nm
- Repeatability: 200 nm
- Strong temperature drift: 1μm/°C

Fig. 4.2 Setup for inductive resonant torque-sensor

If two inductive sensors are used in the torsional setup, as shown in Fig. 4.2, a displacement of 100 nm corresponds approximately to an applied torque of 0.1 μNm. Unfortunately these encouraging results for the displacement sensor could not be transposed to the torque-sensor. The mechanical setup with the thin membranes is much too sensitive to temperature variations. In the first prototype the membranes were glued to the cylinder and the outer ring. With this fabrication procedure it was inevitable to have predeformations in the membranes, which sharply increased the instability of the system, i.e. its deformation behaviour due to small loads. It was impossible to make any useful measurements. In the second prototype, the membranes were glued to the cylinder and using a special mechanism, they were preloaded against the ring. In order to obtain reasonable temperature dependencies however, the preload has to be pretty high. Due to the preload the deflections of the membranes due to an applied torque diminish dramatically. The resulting displacements are too small to be measured with the inductive sensor. That is the reason why this concept was abandoned, as the effects of temperature outran the effects due to torque.
4.3 Torque-sensor

The experience gained during the tests with the sensors described in the previous section was very helpful in the design and development of the final sensor. Having pitifully failed to buy something worthwhile from the industry, further industrial adventures were excluded. The second, self-developed sensor had a fancy design and apparently all other features to become a success, but again a severe disillusion could not be avoided. Two important points were learnt during these lessons:

- Do not trust the industry, but try to design your own torque-sensor.
- Do not try to make a spectacular construction, but take the easiest design which might possibly work and see what you get.

It should perhaps be mentioned that a good starting point was given by the already mentioned works of Condon and Marcus [8] and Farrell [15]. In fact they provided the idea for the mechanical part of the setup. All the characteristics of the sensor, as its geometry, its actual sensor part and its further specifications will now be explained.

4.3.1 Working principle and general setup

The sensor developed belongs to group II as exposed in Section 4.2.1. Its deformable part consists of a thin steel wire, which has a length of 10 mm and a diameter of 0.8 mm. One end of the wire is welded to a rigid metal plate, whereas the other end is attached to a rigid steel bar. The rotation of the wire induced by an applied torque generates displacements at both ends of the bar (see Fig. 4.3). The displacements $u_1$ and $u_2$ can be measured using a two-point heterodyne laser interferometer together with a Polytec OFV 3001 laser demodulator. In these interferometers, the reference beam is not reflected by an internal, fixed mirror in the laser head, but, guided by an optical fibre, it also hits an external object which moves. Using a fringe-counter and interpolating between the fringes allows static measurements with a resolution of about 10 nm to be performed. In this case both laser heads, separated by a distance $d$, are placed so that their beams hit the bar at right angles. This differential measurement allows to eliminate displacements due to bending. The relative displacement measured, $u_1 - u_2$, depends only on the applied torque $T$ and is proportional to $T$. For small angles, the rotation angle $\alpha$ of the wire is given by:
\[ \alpha = \frac{u_1 - u_2}{d} \]  

(4.1)

If the torsional stiffness of the wire, \( k_T \), is known, the torque \( T \) acting on the wire, and thereby on the microsample, can directly be deduced from \( \alpha \):

\[ T = k_T \alpha \]  

(4.2)

Fig. 4.3 Working principle of torsional sensor

### 4.3.2 Calibration

One of the positive aspects of this sensor is the easy calibration procedure, which allows its recalibration after several measurements. The sensor is calibrated dynamically by measuring the torsional resonance frequency of the system rigid bar-wire. This is done by applying a sinusoidal voltage to a coil which electromagnetically excites the system wire-bar. The amplitude of the resulting motion is measured with the same laser interferometer as previously described. The frequency generating the maximum amplitude is taken to be the resonance frequency.

The resonance frequency of this system can be calculated analytically as a function of the torsional stiffness of the wire, if the geometry of the bar is known. The bar has a constant cross-section, whose dimensions are shown in Fig. 4.5. The other characteristics of the bar are its length \( l \) and mass \( m \) and its density \( \rho \). This
specific geometry was chosen for the following reasons:

- Good coincidence of wire and specimen axes.
- Easy fixation of the specimen on the bar. The access for applying the glue has to be guaranteed.
- Easy removal of the destroyed sample.

This geometry implies that the centre of gravity of the bar does not lie on the wire axis and that the main axes of inertia are not parallel or perpendicular to the wire axis. These two conditions necessitate a full-blown analysis of the system, which is modelled as a rigid body with six degrees of freedom connected to springs, three rotational and three translational ones. Force and moment equilibrium in all three directions yield six equations. The six resonance frequencies of the model are computed by assuming a harmonic response and setting the determinant of the resulting equation system to zero, to obtain non-trivial solutions. The final calibration is done with the plot of the resonance frequency $f_R$ versus the torsional stiffness of the system $k_T$, as shown in Fig. 4.4. As it is difficult to analytically solve the inverse problem, the solution is found graphically. The difference between this computation and a computation neglecting these two facts and considering only the rotation around the wire axis are however negligible ($< 0.1\%$).

![Calibration diagram](image)

The torsional resonance frequency was experimentally determined to be $f_R = 25.42$ Hz with a Q-factor of about 220. The characteristics of the bar (Fig. 4.5) yield the calibration diagram of Fig. 4.4. The torsional stiffness of the system, $k_T$, amounts to:

$$k_T = 0.497 \text{ Nm/rad} \quad (4.3)$$
Calculating the torsional stiffness for a steel wire as described previously yields a value of 0.33 Nm/rad. The difference between this value and the one found in Eq. (4.3) can be explained by the fact that the welding changes the material properties and the geometry of the wire. The wire gets thicker at its ends, which induces an increase in stiffness.

\[ \begin{align*}
  h_1 &= 10 \text{ mm} \\
  h_2 &= 2.90 \text{ mm} \\
  b_1 &= 4.92 \text{ mm} \\
  b_2 &= 2.41 \text{ mm} \\
  l &= 98.85 \text{ mm} \\
  m &= 23.90 \text{ g}
\end{align*} \]

Fig. 4.5  Geometry of the sensor-bar

The next section deals with the resolution, accuracy and thermal drift of the sensor.

### 4.3.3 Sensor specifications

#### Accuracy

According to Eq. (4.2) there are two main error sources, the determination of the torsional stiffness \( k_T \) and the determination of the angle of twist \( \alpha \). The errors in the stiffness are due to geometrical inaccuracies (determination of the polar moment of inertia \( I_p \) of the bar) and to errors in the resonance frequencies \( f_R \). The latter ones are negligible as compared to the former ones. The relative error in the stiffness can be written as:
\[ \frac{\Delta k_T}{k_T} = 2 \frac{\Delta f_R}{f_R} + \frac{\Delta \rho}{\rho} = 0.017 \] (4.4)

The relative error in the angle is determined by the accuracy of the distance between the two laser spots \(d\) and by the accuracy of the displacement measurement \(u\). The fact that both beams are neither strictly parallel to each other nor perpendicular to the bar, can be neglected, as it is only a second order effect. The total relative error amounts to:

\[ \frac{\Delta \alpha}{\alpha} = \frac{\Delta u}{u} + \frac{\Delta d}{d} = 0.013 \] (4.5)

The total relative error of the torque measurement, and thus the accuracy, amounts to 3.0% of the measured value. To check this value, one of the experiments on silicon microstructures was simulated by FEM. Silicon was chosen, as its elastic constants are well known from macroscopic and microscopic investigations. The numerical stiffness of the specimen is 5.94 \(\mu\)Nm/\(\theta\), as compared to the experimental stiffness of 5.92 \(\mu\)Nm/\(\theta\). This yields the amazingly low difference of 0.3%.

**Resolution**

The resolution of the sensor is given by the amount of white noise of the laser interferometer system. It amounts to 10 mV, which corresponds to a resolution of 0.05 \(\mu\)Nm.

**Thermal drift**

The thermal drift of the system, which is mainly due to the electronic components of the demodulator, is characteristically about 1 mV/s, or 0.005 \(\mu\)Nm/s. To get reasonable results, the actual experiment, from beginning of rotation to failure of the sample, should not take longer than 30 seconds.

The issues of repeatability, hysteresis or influence of speed and acceleration will be addressed in the next section, as they are factors that are best determined experimentally.
4.4 Experiments

4.4.1 Experimental setup

Fig. 4.6  Experimental setup

1. Rigid bar with wire
2. Glued silicon specimen
3. Two-component glue: HBM X60
4. Steel support block
5. Optical fibre with focussing lens
6. Vertical translation stage: Aerava-9
7. Translation stages: 2 x Newport M-426A with 2 SM-13 Vernier micrometers
8. Rotation stage: Newport M-URM100ACC
9. Laser sensor head: Polytec OFV 512
10. Power Macintosh 7100
11. Digital multimeter: Fluke 8842A
12. Digital storage oscilloscope: LeCroy 9304A 200 Mhz
13. Motor controller: Newport MM1000 DC
14. Laser demodulator: Polytec OFV 3001
15. Pneumatically isolated research table: Newport

In this section the setup using the self-built sensor, denoted by TS, will be described. At the exception of small details, the setup with the commercial sensor, denoted by TMX, was similar and will not be described at this place. For more information about the setup using TMX, see Schiltges et al. [58]. While explaining the setup used during this work, see Fig. 4.6, the experimental procedure and test preparations will also be exposed.

After detaching the specimen from its protective frame, it is placed horizontally with both plates on two different support blocks. Under a microscope the edge of the lower plate is placed parallel to an alignment edge on the support. After the alignment, only the lower plate is glued to its support using a two component glue for strain gauges (HBM X60), whereas the other plate remains free.

When the glue has hardened, which takes approximately 15 minutes, the support is placed in the centre of a translation stage, which is mounted on a rotation stage, with a resolution of 1/1000° and a maximum wobble of 70 μrad. The wobble is the maximum angle between the ideal vertical axis, which would always remain perpendicular to the rotation stage, and the actual rotation axis. In order to have pure torsion, other stresses, in particular stresses due to bending need to be minimized. Therefore a minimal wobble is important in order to have nearly parallel axes of the rotation stage and the specimen. Parallel axes are a necessary, but not a sufficient condition to minimize stresses due to bending. Even if both axes are parallel, they may not coincide. Their alignment is obtained by means of the translation stage. For this purpose a CCD camera is needed which records the position of the specimen axis during rotation. This camera is not shown in Fig. 4.6, but is placed at the position of the optical fibre-lens system, so as to be
able to view the specimen axis. The initial position of the specimen axis is recorded with the CCD-camera, which captures a magnified image through a monozoom microscope (LEICA Monozoom 7, numerical aperture 0.2 at maximum magnification).

In a second step, the specimen, with its upper end still free, is rotated by 180° and an image of this second position is taken. By comparing both images with an image processing software, see Fig. 4.7, the translational error can be reduced to about 2 μm. This procedure has to be repeated twice, once while the optical line and the plate of the microstructure are perpendicular to each other and once while they are parallel.

![Fig. 4.7 Two superimposed images in the process of alignment of rotation and specimen axes](image)

After both axes are in good coincidence, the interferometer system with its optical fibres is installed. Using the vertical translation stage the mechanical part of the sensor is lowered to the sample, till the lower end of the rigid bar is at half height of the upper plate of the specimen. In the next step the sensor axis and the specimen axis are aligned. As the sensor is fixed, the specimen is moved by means of the second translation stage, mounted between optical table and rotation stage.

After this operation the upper plate is glued to the rigid bar. During the hardening of the glue tensile forces act on the specimen. These forces can be measured with a balance, placed between the translation stage and the support block. This has been done for some experiments, but most tests were done without a balance, as the stability and the signal to noise ratio of the system improved drastically without a balance. After hardening of the glue, the experiment itself may start. Two
further comments have to be made concerning the start of the experiment.

- Before beginning, the electronic equipment, especially the dual interferometer, must have been switched on for about an hour, in order to avoid a too strong thermal drift.
- If the optical table or the mechanical part of the setup is touched, the rigid bar of the sensor begins to vibrate. At a resonance frequency of 25 Hz and a Q-factor of 200, it takes some time before the system is at rest again. By the way, avoiding vibrations of the system is the reason why the experiment takes place on an optical damping table.

The experiment is computer controlled, both the actuation and the sensor part. Via a motor controller, linked to a Mac through GPIB interface, the rotation stage can be driven at different speeds and accelerations. The resulting torque is measured with the sensor. The signal of the laser head is processed in the laser demodulator, whose output goes to a digital multimeter and a digital oscilloscope. The signal of the multimeter, directly proportional to the torque, is fed to the computer via GPIB. The oscilloscope has only a control function, as the progress of the experiment can be monitored on the display.

Besides the torque it is necessary to measure the corresponding angle of twist. Theoretically the torsional stiffness of the specimen is so small, that the compliance of the rest of the system can be neglected. This has been verified experimentally. The actual rotation angle is measured with a HeNe-laser and a linear diode detector. The laser beam is adjusted so that its reflection on the specimen surface can be captured with the linear detector. Depending on the rotation of the specimen the beam hits the diode at a different position. The detector has a resolution of 50 µm at a total range of 70 mm. The distance between specimen and detector being about 100 mm, the angular resolution is about 0.03°. It can now be shown that the rotation of the lower plate corresponds exactly, within the resolution limits of the diode detector, to the angle given by the rotation stage. The rotation of the rigid bar is given by the measurements with the interferometer. At a torque of 50 µNm its angle amounts to about 0.006°, which is negligible. Thus the angular detector is not implemented and the angle of twist is taken to be the angle of the rotation stage.

The experiment lasts about 30 seconds, after which failure of the specimen occurs. The data, which consists of time, torque and corresponding angle is written in a file on the computer and can be processed afterwards.
After failure, the rigid bar-wire system is detached from the vertical stage and the support block is removed from the translation stage. The remaining parts of the specimen are viewed under a microscope, see Fig. 4.17. Both parts, the support as well as the rigid bar are submerged in acetone, which dissolves the upper layer of the glue and thus facilitates the removal of the lower and upper plate of the microstructure. The torque sensor part can now again be fixed to the vertical stage.

4.4.2 First tests

To test the experimental setup, four experiments have been carried out, two on silicon structures, named si1 and si2, and two on NiFe specimens of different geometries, named li1 and li2. The aim of these tests was twofold:
• Check the repeatability of the measurements.
• Investigate the influence of speed, acceleration and total angle.

For this purpose four series à 8 experiments are carried out for each specimen. After each experiment the torsional stiffness of the specimen is determined. In the first series the specimens are rotated by an angle of 3°, with a speed of 0.2°/s and an initial acceleration of 1°/s². These parameters are held constant throughout the first series. During the second series the speed is raised by 0.05°/s from one experiment to the next, with an initial value of 0.05°/s, the other parameters remaining constant. The third series is characterized by an augmentation of the acceleration of 0.2°/s², beginning with 0.4°/s². In the fourth series the rotation is increased by 1° in each experiment, until failure occurs. The resulting curves can be seen in Fig. 4.8.
Fig. 4.8  Initial tests of torsional sensor on two silicon samples, si1 and si2, and on two LIGA samples, li1 and li2. The torsional stiffness of the specimen for each experiment is shown. Further explanations are given in Section 4.4.2.

With the exception of the two erroneous measurements of series 4 for si1, it can be seen that all the measured values for a given sample lie in a narrow band. Table 4.1 lists the mean values and standard deviations for each series of each experiment.
Table 4.1  Mean value and standard deviation of torsional stiffness of each specimen for each experimental series
*: without the two erroneous measurements

In the worst case of all these constellations, the standard deviation amounts to 2.1% of the mean value. With these evaluations it can be said that the repeatability of the experiments is guaranteed within a margin of 2%. Neither speed, nor acceleration, nor total rotation have a clearly decipherable influence on the measurements, within the limits examined. All further experiments will be carried out with a speed of 0.3°/s and an acceleration of 0.5°/s². An example of the diagrams obtained can be seen in Fig. 4.9.
4.4.3 Experiments on silicon

Several experiments were carried out on silicon microstructures. Four out of eight were performed using a balance to measure the normal force occurring during gluing. During these experiments the noise and stability of the system were much worse than without the balance. This means that the signal to noise ratio was about a factor 10 higher without a balance and that the drift of the signal was lower without a balance. This is the reason why the other four experiments on silicon and all the experiments on metallic microstructures were carried out without a balance.

Tensile forces

The forces due to glue-hardening are almost the same for the four samples. During the hardening process, the tensile force increases, to reach a plateau and stay constant at about 100 mN after the glue has hardened (see Fig. 4.10 a). This tensile force, which is small as compared to a critical force of about 2.5 N found by Mazza [39], stretches the specimens during the torsional test. The forces measured while rotating the specimens are not very conclusive. At the beginning of the test, the tensile force seems to increase, gets to an absolute maximum and decreases afterwards. This general behaviour is the same for all the specimens, but there are strong differences in the maximum force attained, and in the amount by which the force decreases afterwards. An example of the tensile force in function of the rotation angle is given in Fig. 4.10 b.

![Fig. 4.10 Tensile forces occurring](image-url)
Torsional stiffness

As the specimens used have slightly different geometries, the experimental results are compared to numerical simulations (see Table 4.2). For these simulations the following constants were assumed for silicon: $E = 130$ GPa, $G = 79.6$ GPa and $\nu = 0.28$. The discrepancy between measured and calculated stiffnesses can be explained by errors occurring as well in the experiment as in the simulation. As was shown in the previous section, the accuracy of the measurement amounts to 3%. The accuracy of the simulation is given by errors in the model, due to inaccurate geometries, and to errors due to the refinement of the mesh. The latter lies at about 1% and the former yields about 4.5%. With the exception of one measurement, experiments and simulations are in good agreement within the error margins.

<table>
<thead>
<tr>
<th>specimen</th>
<th>experimental stiffness [$\mu$Nm/°]</th>
<th>numerical stiffness [$\mu$Nm/°]</th>
<th>$\Delta$ [%]</th>
<th>fracture angle [°]</th>
</tr>
</thead>
<tbody>
<tr>
<td>97si8 (78x49x215)</td>
<td>5.99</td>
<td>6.27 ± 0.34</td>
<td>-4.5</td>
<td>5.3</td>
</tr>
<tr>
<td>97si16 (78x49x216)</td>
<td>6.96</td>
<td>6.25 ± 0.34</td>
<td>+11.3</td>
<td>5.7</td>
</tr>
<tr>
<td>97si15 (65x49x200)</td>
<td>5.46</td>
<td>5.27 ± 0.29</td>
<td>+3.6</td>
<td>5</td>
</tr>
<tr>
<td>98si2 (78x50x210)</td>
<td>6.28</td>
<td>6.64 ± 0.35</td>
<td>-5.4</td>
<td>6.2</td>
</tr>
<tr>
<td>98si1 (79x48x213)</td>
<td>5.87</td>
<td>6.06 ± 0.34</td>
<td>-3.1</td>
<td>6.5</td>
</tr>
<tr>
<td>97si18 (79x47x210)</td>
<td>6.03</td>
<td>5.79 ± 0.31</td>
<td>+4.1</td>
<td>5</td>
</tr>
<tr>
<td>98si6 (79x49x212)</td>
<td>6.55</td>
<td>6.39 ± 0.34</td>
<td>+2.5</td>
<td>5.2</td>
</tr>
<tr>
<td>98si7 (79x49x211)</td>
<td>6.31</td>
<td>6.39 ± 0.34</td>
<td>-1.3</td>
<td>5.9</td>
</tr>
</tbody>
</table>

Table 4.2 Comparison between experimentally determined and numerically expected torsional stiffnesses of various specimens. The value behind the numerical stiffness indicates the error margin of the simulation, which lies at about 5.5%.

*: experiments with a balance

Hysteresis and repeatability

The sensor has no hysteresis, as can be seen in Fig. 4.11 a. The curves of loading and unloading show no difference and follow the same path. Within the error
margin due to the resolution of the sensor, both curves yield the same torsional stiffness, varying between 5.89 μNm/° versus 5.92 μNm/°.

To show the repeatability of the measurements, Fig. 4.11 b shows the curves obtained by repeating the same experiment, all factors held constant. The specimen is twisted by 3° and then twisted back to 0°. This procedure is repeated three times. The six torsional stiffnesses thus determined vary between 5.83 and 5.92 μNm/°.

\[ a) \]
\[ \text{angle [°]} \]
\[ \text{torque [μNm]} \]

\[ b) \]
\[ \text{angle [°]} \]
\[ \text{torque [μNm]} \]

Fig. 4.11  Torque/rotation diagrams of specimen 98si1

a: loading and unloading step 0-3° and 3-0°
b: three consecutive loading and unloading steps

4.4.4 Experiments on LIGA specimens

A series of experiments was carried out on six metallic specimens with different geometries, but of the same material NiFe(53%Fe). The shear-moduli of this material were known from previous dynamic tests. It is an orthotropic material, whose shear-moduli are: \( G_{12} = 40 \) GPa, \( G_{13} = 56 \) GPa.

Torsional tests

The tests were carried out in the same way as for the silicon specimens, no balance was used.

For each specimen several loading and unloading cycles were carried out in the elastic regime, usually below a rotation of 5°. The resulting curves yield the torsional stiffness of the specimen. After eliminating the two extreme values, the
final torsional stiffness is taken to be the mean value of the other experiments, see Table 4.3.

The type of curves obtained during these experiments can be seen in Fig. 4.12.

<table>
<thead>
<tr>
<th>specimen</th>
<th>dimensions [μm³] [length x width x height]</th>
<th>torsional stiffness [μNm/°]</th>
</tr>
</thead>
<tbody>
<tr>
<td>662c</td>
<td>300 x 40 x 120</td>
<td>4.45</td>
</tr>
<tr>
<td>662d</td>
<td>300 x 40 x 122</td>
<td>4.74</td>
</tr>
<tr>
<td>662a</td>
<td>300 x 30 x 119</td>
<td>2.07</td>
</tr>
<tr>
<td>662f</td>
<td>300 x 20 x 101</td>
<td>0.65</td>
</tr>
<tr>
<td>96</td>
<td>300 x 30 x 129</td>
<td>2.41</td>
</tr>
<tr>
<td>156</td>
<td>1000 x 40 x 118</td>
<td>1.61</td>
</tr>
</tbody>
</table>

Table 4.3 Torsional stiffness and dimensions of metallic specimens

Fig. 4.12 Torque-rotation diagrams

1: line characteristic of torsional stiffness
2: torque at beginning of yielding
3: rotation at beginning of yielding
MNET

MNET was used to determine the shear-moduli of the material. This technique has been explained in chapter 3, where elastic constants are obtained from the measurement of resonance frequencies. Some modifications have to be made in order to use this technique also in the case of static experiments.

The experimentally determined torsional stiffnesses of all six specimens were used. Seven iterative steps were made. The evolution of the various numerical stiffnesses, the shear-moduli and the relative error is shown in Fig. 4.13. Both final numerical and experimental stiffnesses are shown in Table 4.4.

<table>
<thead>
<tr>
<th>specimen</th>
<th>experimental stiffness [μNm/°]</th>
<th>numerical stiffness [μNm/°]</th>
<th>Δ [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>662c</td>
<td>4.45</td>
<td>4.55</td>
<td>-2.2</td>
</tr>
<tr>
<td>662d</td>
<td>4.74</td>
<td>4.67</td>
<td>+1.5</td>
</tr>
<tr>
<td>662a</td>
<td>2.07</td>
<td>2.24</td>
<td>-7.6</td>
</tr>
<tr>
<td>662f</td>
<td>0.65</td>
<td>0.63</td>
<td>+3.1</td>
</tr>
<tr>
<td>96</td>
<td>2.41</td>
<td>2.53</td>
<td>-4.7</td>
</tr>
<tr>
<td>156</td>
<td>1.61</td>
<td>1.57</td>
<td>+2.5</td>
</tr>
</tbody>
</table>

Table 4.4  Comparison between experimental and final numerical stiffness

$G_{13}$ almost remains constant after two iterative steps, its final value is 51.9 GPa. The variation of $G_{12}$ is large even after six or seven steps, the final value is found to be 35.4 GPa. This is due to the fact that the influence of $G_{12}$ on the torsional stiffness is much smaller than for $G_{13}$. The height, length and width of the specimens can be determined with 1 μm accuracy. The relative error of the torsional stiffness is given by the accuracy of the sensor, which amounts to 3%. These various error sources yield a relative error for both shear-moduli of:
\[
\frac{\Delta G_{12}}{G_{12}} = 0.48 \\
\frac{\Delta G_{13}}{G_{13}} = 0.13
\]  

(4.6)

The relative error decreases continuously, with the exception of the sixth iterative step. The values for the shear moduli found in dynamic, see Section 3.5.3, and in static tests are in good agreement. A difference of 11\% for \( G_{12} \) and of 7\% for \( G_{13} \) can therefore be accepted.

Fig. 4.13 Variation during iteration of
a) stiffnesses
b) shear-moduli
c) relative error
4.5 Mixed numerical experimental technique

In this section the results of static torsional experiments with MNET are presented. For the method to work at least two experiments on specimens with different cross-sections have to be carried out. Besides the MNET another method, using analytical results as well as numerical and experimental insights will also be presented. This method was used prior to MNET and indirectly led to MNET.

4.5.1 Determination of shear moduli with MNET

The method used to determine shear-moduli is the same as the one that was applied for the resonance frequencies. The difference consists in the fact that the resonance frequencies are replaced by torques and the different modes by torque measurements on different samples.

This procedure was used to evaluate the shear moduli of three different materials, tested with the industrial sensor TMX. For silicon and pure Ni, three experiments were analysed whereas only two experiments were used for NiFe(20%). The results obtained, as well with TMX as with the self-built sensor TS, are shown in Table 4.5.

<table>
<thead>
<tr>
<th>material</th>
<th>sensor used</th>
<th>$G_{12}$</th>
<th>$G_{13}$</th>
<th>$\frac{\Delta G_{12}}{G_{12}}$</th>
<th>$\frac{\Delta G_{13}}{G_{13}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>TMX</td>
<td>80</td>
<td>73</td>
<td>0.09</td>
<td>0.26</td>
</tr>
<tr>
<td>Ni</td>
<td>TMX</td>
<td>86</td>
<td>59</td>
<td>0.46</td>
<td>0.12</td>
</tr>
<tr>
<td>NiFe(20%)</td>
<td>TMX</td>
<td>83</td>
<td>41</td>
<td>0.46</td>
<td>0.15</td>
</tr>
<tr>
<td>NiFe(53%)</td>
<td>TS</td>
<td>35</td>
<td>52</td>
<td>0.48</td>
<td>0.13</td>
</tr>
</tbody>
</table>

Table 4.5 Shear moduli of different materials as determined in microtorsional tests and corresponding relative errors

As for each procedure to determine material parameters, the accumulation of errors is inevitable. The calculation of the relative errors is similar as in Section 3.6 and will not be described at this place. The relative errors for silicon
are in acceptable limits, whereas the relative errors for $G_{12}$ for the metallic samples are way beyond good and evil. This is due to the fact that the height of all the LIGA specimens is much larger than their width. The specimens can therefore almost be considered as thin-walled cross sections, where the shear-stresses in one direction almost vanish. This means that the influence of $G_{12}$ on torsional stiffnesses is much smaller than the influence of $G_{13}$. This phenomenon is not present with silicon microstructures, where there are two specimens whose width exceeds their height and one specimen whose height exceeds its width.

4.5.2 Determination of shear moduli with a mixed analytical, numerical and experimental technique

For those who do not trust the purely numerical approach of the previous chapter, and who want to see some formulas to understand what happens, there fortunately exists another approach involving and necessitating analytic comprehension of the torsion phenomenon. A direct analytic approach to the problem is not possible, as the cross sections of the specimens are not constant. Using finite elements, the change of twist per unit length in the actual microbeam, where the cross section does remain constant, can be found. This can be used in the governing equation of torsion to determine the shear-moduli involved.

Torsion of an anisotropic rod with constant rectangular cross section

The torsional problem of an anisotropic rod with constant rectangular cross section is described in Voigt [69] by the generalized Poisson-equation (the derivation of this equation is given in Appendix A):

$$\frac{\Phi_{22}}{G_{13}} + \frac{\Phi_{33}}{G_{12}} = -2 \quad (4.7)$$

$\Phi$ is the stress function and $G_{12}$ and $G_{13}$ are the governing shear moduli. For a given rotation angle $\theta$, the corresponding torque $T$ is a function of $\theta$ and of $\Phi$:

$$T = 2 \cdot \theta \cdot \int \int \Phi(G_{12}, G_{13}) dx_2 dx_3 \quad (4.8)$$

For a rectangular cross-section, the stress function can be computed from (4.7) by
minimizing the potential energy of the elastic body (the potential energy is
defined to be the difference between deformation energy and work potential of
the acting forces), and can then be introduced in (4.8). The torque can then be
expressed as an infinite sum (4.9), which converges rapidly. In (4.9) a and b are
the half-width and half-height of the rod respectively.

\[
T = \frac{2^{12}}{\pi^6} \cdot \theta \cdot a \cdot b \cdot \sum_{m=1,3} \sum_{n=1,3} \frac{1}{m^2 \cdot n^2} \left( \frac{m^2}{a^2 \cdot G_{13}} + \frac{n^2}{b^2 \cdot G_{12}} \right)^{-1} \tag{4.9}
\]

\(G_{12}\) and \(G_{13}\) are the two unknown values to be determined. The torque and the
rotation can directly be deduced from the experiment. At least two experiments
have to be carried out to obtain two equations. If the ratio width/height of the two
specimens differs, a system of two linear independent equations results (4.10),
which can then be solved numerically for \(G_{12}\) and \(G_{13}\).

\[
T_1 = \frac{2^{12}}{\pi^6} \cdot \theta_{1,1} \cdot a_1 \cdot b_1 \cdot \sum_{m=1,3} \sum_{n=1,3} \frac{1}{m^2 \cdot n^2} \left( \frac{m^2}{a_1^2 \cdot G_{13}} + \frac{n^2}{b_1^2 \cdot G_{12}} \right)^{-1} \tag{4.10}
\]

\[
T_2 = \frac{2^{12}}{\pi^6} \cdot \theta_{2,1} \cdot a_2 \cdot b_2 \cdot \sum_{m=1,3} \sum_{n=1,3} \frac{1}{m^2 \cdot n^2} \left( \frac{m^2}{a_2^2 \cdot G_{13}} + \frac{n^2}{b_2^2 \cdot G_{12}} \right)^{-1}
\]

**Torsion of an anisotropic rod with changing rectangular cross section**

The system of equations in (4.10) is valid only if the cross section of the beam is
of constant shape and area. This is not true for the samples used in this study, nei¬
ther for the silicon nor for the metallic specimens. Thus (4.10) cannot be directly
used to obtain the shear moduli. The problem can however be solved in a mixed
analytical, numerical and experimental approach, whose individual steps will
now be explained.

- Two experiments on specimens with a different width/height ratio are carried
  out and the ensuing torque-rotation relation is recorded.
- A three dimensional finite element model of both specimens is generated. In a
  first step, as the elastic parameters are unknown, an initial guess of their val¬
  ues has to be made. The lower plate is clamped and fixed, and the upper plate
is rotated by an angle $\theta_u$. The value of $\theta_1$ changes over the transition region but remains constant, $\theta_{r,1}$, in the testing region. Its value can be determined in the finite element model:

- The values of $\theta_{1,1}$ and $\theta_{2,1}$, determined by the FE simulation, can be introduced in Eq. (4.10). The torques $T_1$ and $T_2$ can be extracted from the experimental data. They correspond to the angles $\theta_{u1}$ and $\theta_{u2}$ by which the upper plates of the FE models are rotated. By solving the equation system (4.10) with these values, a first approximation of the shear moduli is obtained.
- The calculated shear moduli are introduced in the FE simulation, assuming homogeneous properties for the whole specimen, and a new value of $\theta_{r,1}$ is computed for both specimens.
- These steps are repeated till the difference between the shear moduli of two consecutive iterative processes is small enough.

### 4.6 Failure criteria

#### 4.6.1 Failure criterion for silicon

Due to the stress concentrations in the vicinity of the sharp notch, silicon specimens always fail at the notch. It is no longer useful to determine critical stresses. A possible approach might be to establish critical stress intensity factors, using analytic solutions of the near-field behaviour and determining the stress intensity factors at the critical load. There are three reasons why this approach was not chosen in this work.

- To be able to establish an analytic solution, by using for example Stroh’s formalism (see Stroh [64]), simplifying assumptions have to be made concerning either plane stress or plane strain conditions in the plane of the macroscopic plate. Whereas this might be an option in the case of tension or bending, it is not a viable solution for torsional problems.
- Even if the dependency of the stresses on the coordinates $r$ and $\varphi$ from the notch apex could be found by analytic considerations, it is not possible to determine by a purely analytical calculation the stress intensity factors. To do so the analytic solution has to be compared to numerical results, i.e. finite element programs have to be used.
- The stress intensity factors are dependent on the geometry and on the loadcase considered.
To circumvent the problem of the determination of stresses at the notch apex, which, with the present numerical techniques, presents major difficulties, and to be independent on the geometry and loadcase, it is useful to make energy considerations. The strain energy in any volume will remain finite, though the stresses may be unbounded. To determine the strain energies, numerical simulations will be used. The elements used are three dimensional 20-node, isoparametric, arbitrary hexahedral brick elements, using 27-point Gaussian integration. The strain energies in the immediate vicinity of the notch tip will also be inaccurate, due to large strains, numerical problems and the fact that continuum mechanics is no longer valid at that scale. The strain energies of the near-field solution however are accurate. They can be used to determine the strain energy in function of the radius from the notch centre. By comparing the strain energy in a characteristic volume to the energy necessary to create two free (111) surfaces, it might be possible to establish a failure criterion. This criterion has furthermore the advantage that it is independent on the state of stress acting on the specimen. It is valid for torsional, tensile and bending modes, and of course for a combination of these different loading modes. The criterion has first been formulated by Mazza [39].

The procedure to obtain the energy failure criterion will now be explained in detail.

- The rotation angle at failure $\theta_f$, resp. the critical load in tensile tests is determined from the experiment.
- A finite element model is generated using the geometries of the specimen. The mesh used at one of the four notches is refined in the $x_1$-$x_2$ plane (see Fig. 4.14), so as to obtain a good approximation of the behaviour of strain energy density (SED). It does not matter which notch is chosen, as all four are equivalent concerning SED. The minimal distance between two nodal points amounts to about 1 nm. Due to numerical problems it was not possible to do simulations using an even finer mesh.
- A first numerical simulation is computed to determine the node along the apex with the highest SED. Though the numerical value of SED at this point is wrong, it is nevertheless at this point that a crack will originate.
- In several steps the model is refined in $x_3$-direction around the point with maximum SED. The refinement in $x_3$-direction is stopped when the SED for three consecutive nodes along the notch line remains the same (difference < 3%). Typically the final distance between two nodal points along the notch line is smaller than 1 $\mu$m.
• In the finite element model obtained this way, several sets of elements are defined and the strain energy as well as the volume of these sets are calculated. The first set is defined by the two elements in the $x_1-x_2$ plane adjacent to the node with maximum SED. The second set is defined by the elements of the first set plus the elements adjacent to them, etc. This way each set defines a region around the critical node. The largest region has a radius of about 5 µm.

![Image](https://via.placeholder.com/150)

**Fig. 4.14** FE model for calculation of strain energy

• As the dimension in $x_3$-direction remains constant for each set, a strain energy per unit thickness can be generated: $U^* = U/h$, where $U$ is the strain energy for each set and $h$ is the constant thickness of the elements. The corresponding radius can be computed from the calculated volume and so a function $U^*(r)$ can be established, whose graph is shown in Fig. 4.15. This function will be of the form:

$$U^*(r) = B \cdot r^\alpha$$

(4.11)

The factor $\alpha$ is dependent on the geometry of the notch and may vary between 1 and 2, whereas $B$ varies depending on the applied loads.
The surface energy $S^*$ necessary to create two new (111) surfaces in the considered plane and region, see Eq. (4.12), is calculated and compared to $U^*$. The factor $\sqrt{3/2}$ is due to the fact that the (111) plane is inclined with respect to the vertical plane

$$S^*(r) = \sqrt{3/2} \cdot 2 \cdot \gamma_S \cdot r$$

(4.12)

$\gamma_S$ is the surface energy of (111) surfaces. Gilman [19] and Messmer and Bilello [45] give a value for $\gamma_S$ of 1.25 J/m$^2$ and 1.14 J/m$^2$ respectively. In this work an average value of 1.20 J/m$^2$ will be taken. The radius $r_c$ for which $U^* = S^*$ under critical loading conditions can be seen as a material parameter. If the strain energy in the region defined by $r_c$ exceeds the energy necessary to form two new (111) surfaces, failure occurs.

The results of the aforementioned procedure will now be shown in the case of tension and torsion, marked by the upper indices “ten” and “tor”, respectively. The tensional experiments have been performed by Edoardo Mazza. More details can be found in his thesis (see Mazza [39]). For the purposes of this work a representative test on a silicon microstructure has been taken and simulated numerically. The cross section of the specimen was 88x49.1 µm$^2$. For the determination of the SED in the torsional tests, the experiment on the specimen 98si1 was simulated. Fitting the curve of the strain energy per unit thickness found in the numerical simulation at critical load yields:
In the case of tension the analytic solution for $U^*$ would lead to

$$U_{\text{ten}}^* = B \cdot r^{1.348}$$  \hspace{1cm} (4.14)$$

Setting the expressions in Eq. (4.13) equal to the surface energy as given in Eq. (4.12) yields the critical and characteristic radiuses:

$$r_{c,\text{tor}} = 4.5 \text{ nm}$$

$$r_{c,\text{ten}} = 2.2 \text{ nm}$$  \hspace{1cm} (4.15)$$

Theoretically both radiuses should be the same, but they differ by as much as 100% even if the strain energies $U^*$ differ by only 30%. This suggests that it might be wiser to choose a different criterion, namely one involving directly $U^*$. One has however to be aware of the fact that such a criterion is valid only for the geometry considered in this study, which is a major drawback for a criterion which longs to be as general as possible. From this point of view, the radius criterion seems much more appropriate, and as has been shown by Mazza [39], though there might be big differences in the radiuses, the critical loads differ only by a small amount. Coming back to the energy criterion, $U^*$ can be written as $U^* = U_0 \cdot r^{1.34}$. If the value of $U_0$ exceeds a critical value $U_c$, which would be about 2000 N/m$^{1.34}$, failure occurs. The difference of 30% between both critical values will be explained qualitatively by examining possible error sources.

- The rotation at failure, respectively the critical force in a tensile test can only be determined within an error margin of about 2%.
- Predeformations of the specimens are present in both cases. Due to the hardening of the glue, tension and bending occurs in the sample. These supplementary loads are not considered in the analysis presented on the preceding pages. It can however be seen that they might perfectly explain the differences found. The tensile forces due to gluing are taken into account in the tensile tests, but not in torsion, which explains why $U_{c,\text{ten}}$ is higher than $U_{c,\text{tor}}$. The tensile stresses due to bending of the specimen during the glue hardening are
superimposed on the tensile stresses due to pure tension. In particular they may decrease the total tensile stresses at the critical point. This again shows why possibly $U_{c}^{\text{ten}} > U_{c}^{\text{tor}}$.

- In the torsional test, due to non-coincidence of the axes, bending occurs, which is not taken into account in the simulations. The value of $U_{c}^{\text{tor}}$ determined thus is too low, as it does not take into account the bending moments.

A slightly modified failure criterion will now be proposed. It postulates that failure occurs if the strain energy $U$ in a sphere with critical radius $r_{c1}$ and whose centre is the point of maximum SED exceeds the surface energy $S$ in this sphere necessary to create two new (111) surfaces. In a first step the SED are calculated. Under the assumption that SED are only a function of the radius $r$ and are independent of the angle $\phi$, which is a valid approximation for the considerations made in this work, the SED $U'(r)$ can be derived from Eq. (4.11):

$$U'(r) = \frac{\alpha B}{\phi_0} r^{\alpha - 2}$$

(4.16)

$\phi_0$ is the total angle which corresponds to 225°, see Fig. 4.16.

Fig. 4.16 : Top and side view of specimen

To be able to calculate the total strain energy, the SED from Eq. (4.16) has to be expressed in function of the coordinates $r_2$ and $\theta$. This yields:

$$U'(r_2, \theta) = \frac{\alpha B}{\phi_0} \sqrt{2}^{\alpha - 2} r_2^{\alpha - 2} (\cos \theta)^{\alpha - 2}$$

(4.17)

The total strain energy is then given by Eq. (4.18), under the assumption that
SED does not vary from one point to another if the line between both points is parallel to the notch. This is a very good approximation if the considered distances are small (<1 \( \mu \text{m} \)), which is the case.

\[
U = \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \int_{0}^{\theta_r} \int_{0}^{r_2} \bar{U}(r_2, \theta) r_2^2 \cos \theta \, dr_2 \, d\theta \, d\phi \tag{4.18}
\]

\( \eta \) equals \( \pi/2 \) in the case of torsion, as the critical point lies between the top and the bottom sides of the specimen, whereas it equals \( \pi/4 \) in the case of tension, as the point with maximum SED lies at the top side. The strain energy for the critical loads in torsion and tension is given in Eq. (4.19).

\[
U_{01}^{\text{tor}} = 2368 \cdot r^{2.340} \\
U_{01}^{\text{ten}} = 2424 \cdot r^{2.342} \tag{4.19}
\]

The surface energy is given by:

\[
S(r)^{\text{tor}} = \pi \cdot \gamma_s \cdot r^2 \\
S(r)^{\text{ten}} = \frac{\pi}{2} \cdot \gamma_s \cdot r^2 \tag{4.20}
\]

Setting the strain energy equal to the surface energy yields the critical radiuses:

\[
r_{c1}^{\text{tor}} = 5.9 \text{ nm} \\
r_{c1}^{\text{ten}} = 0.8 \text{ nm} \tag{4.21}
\]

From Eq. (4.15) and Eq. (4.21), it can be said that the critical radius is in the low nanometer-range.

Some comments have to be made about the assumption that the crack growth initiates at the notch and that it propagates along (111) planes. The first remark concerns the fact that the assumption really is an assumption and has not been
verified experimentally. It nevertheless makes sense to assume that failure starts at the point with the highest SED. It also seems adequate to suppose that at least at the very beginning the crack grows so as to create planes which have lowest surface energy, i.e (111) planes. These two conditions have of course to be fulfilled for the failure criterion to be valid. No assumptions have to be made about the further behaviour of the crack after this initial phase. As can be seen in Fig. 4.17, this behaviour may be quite varied. There usually is not only one crack which propagates, but a whole piece of the specimen is ejected, which shows that at least two cracks are formed. Sometimes only a part of the microbridge is broken free, as in Fig. 4.17 in b) and d), whereas in other experiments parts of the transition region are also damaged. There seems to be a wide variety of fracture behaviour under the loads investigated. Two constant features can however be seen in Fig. 4.17. There is always a crack going to or coming from the notch and there is always a (111) plane. These two features indicate that the two initial assumptions may be well-founded.

Fig. 4.17 Cracks in silicon specimens after torsional tests
4.6.2 Plastic regime of metallic microstructures

The metallic specimens show an accentuated elastic-plastic behaviour, see Fig. 4.12. As the material considered is orthotropic, an appropriate yield criterion has to be formulated. A possible criterion in the case of orthotropy has been proposed by Hill [21]:

\[
F(\sigma_{22} - \sigma_{33})^2 + G(\sigma_{33} - \sigma_{11})^2 + H(\sigma_{22} - \sigma_{11})^2 \\
+ 2L\tau_{23}^2 + 2M\tau_{31}^2 + 2N\tau_{12}^2 = 1
\]  
(4.22)

To determine the six constants of this criterion, six independent experiments leading to a yield condition have to be performed. At least a seventh independent experiment is necessary to validate the criterion. For the samples discussed in this work, only two independent tests are available, tension and torsion. These two tests yield the values of \(G+H\) and of \(M\):

\[
G + H = \sigma_{1y}^2
\]  
(4.23)

\[
M = 1.78\sigma_{1y}^2
\]

\(\sigma_{1y}\) is the yield stress in \(x_1\)-direction as determined from the tensile test by Mazza [39], its value is 1200 MPa. The value of \(M\) is obtained by taking the mean maximum shear stress \(\tau_{13\text{max}}\) from the six torsional experiments, see Table 4.6, which amounts to 637 MPa. Even though the material is orthotropic, it is interesting to make a comparison to isotropic yield criteria. In the case of isotropy the von Mises hypothesis would yield a value \(M = 1.5 \sigma_{1y}^2\), whereas the Tresca assumption leads to \(M = 2 \sigma_{1y}^2\). It can be seen that the value of \(M\) as given by Eq. (4.23) lies between those two values. For the microstructures considered, only \(\sigma_{11}, \tau_{12}\) and \(\tau_{13}\) are non-vanishing stresses. If the deformation due to bending loads is not in \(x_3\)-direction, the critical point where yielding occurs does not involve \(\tau_{12}\). In this case an empirical yield criterion as in Eq. (4.24) can be proposed.

\[
\sigma_{11}^2 + 3.56\tau_{13}^2 = \sigma_{1y}^2
\]  
(4.24)

In further studies this criterion has to be verified by performing combined tensile
and torsional tests.

<table>
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<td>mean value</td>
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Table 4.6  Beginning of yielding and equivalent stresses

In Fig. 4.18 an example of a metallic specimen in plastic regime is shown.

![Deformed metallic specimen after a rotation of 475°](image)

Two facts are neglected in this plastic analysis. Due to the gluing of the specimen and due to the fact that the three axes involved do not coincide, tensile stresses may occur in the samples. It will now be shown that these effects are negligible and that the ensuing errors lie well below 1%.
Gluing of the specimen

Due to the gluing of the specimen, tensile, bending and torsional forces act on the specimen. The corresponding stresses can partially be quantified, either by measurement of the forces or by measurement of the displacements:

- The tensile force acting on a specimen with cross-sectional dimensions of 120 x 30 µm² was measured to be about 100 mN. This corresponds to a stress σ₁₁ over the whole cross-section of approximately σ₁₁ = 30 MPa.
- Bending can occur either in the x₂ or the x₃ axis. The maximum stress is then either on the outer small or on the outer large side of the rectangular cross-section. Due to the symmetry of the gluing it is assumed that no bending occurs along the x₁-axis. The bending along the other axis can be quantified by measuring the displacement perpendicular to the plate of two points during the gluing process. This is done by placing the specimen under the UBM focusing laser and measuring the distance before and after gluing. This is done at four different points on the plate, see Fig. 4.19.

![Procedure to determine stresses due to gluing](image)

The displacement field of the plate and the beam is characterized by the three translations and three rotations at one point of the plate and by the elastic properties of the structure. The displacement u₁ in x₁ direction is characterized by the tensile force acting on the specimen. The rotation α₁ amounts to virtually 0, as the displacements of points 3 and 4 (see Fig. 4.19) are the same. This shows that the assumption of symmetric gluing is valid and that the displacement u₂, as well as the rotation α₃ can be set to zero. By comparing the measured displacements of points 1 and 2 with numerical models (one
model with a unitary displacement at point 1 and one model with a unitary rotation at point 1), the displacement \( u_1 \) and rotation \( \alpha_2 \) can be deduced. Thus the displacement field and the stress field in both the plate and the beam are known. The maximum tensile stress \( \sigma_{11} \) due to bending is about 25 MPa. The total tensile stress thus yields 55 MPa. This value is negligible for calculating equivalent stresses, if the maximum shear stress is 600 MPa. The error committed is below 0.2%.

### Non-coincidence of rotation, specimen and sensor axes

By taking the rotation axis as reference, several errors may occur, see Fig. 4.20. The sensor axis is not parallel to the rotation axis, error called rotational mismatch and designated by \( \alpha \). This error may be superposed to a translational mismatch between both axes, designated by \( \delta \). The same kind of errors can happen with the specimen and rotation axis. All these errors can be quantified, which is done in Table 4.7. Mismatches between sensor and rotation axes are designated by the index \( s \), whereas the index \( p \) denotes mismatches between specimen and rotation axes.

<table>
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<tr>
<th>errors</th>
<th>( \delta_{px2} )</th>
<th>( \delta_{px3} )</th>
<th>( \alpha_{px2} )</th>
<th>( \alpha_{px3} )</th>
<th>( \delta_{sx2} )</th>
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<th>( \alpha_{sx2} )</th>
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<tbody>
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<td>1</td>
<td>200</td>
<td>200</td>
<td>2</td>
<td>2</td>
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</table>

Table 4.7 Possible errors and their magnitude

The non-coincidence of these three axes may have various effects:
- In the specimen tensile stresses due to bending occur besides the shear stresses due to pure torsion. In the worst case scenario the tensile stresses \( \sigma_{11} \) do however not exceed 20 MPa, in the region where the maximum shear stresses occur. Added to the tensile stresses due to gluing this yields a total tensile stress of \( \sigma_{11} = 75 \) MPa. This value of 75 MPa is of course only valid in the region of maximum shear stress. Outside of this region, the total tensile stress may get as large as 500 MPa, but the yielding will not occur in these zones, as the equivalent stress is lower as in the zones of interest.

The difference between the equivalent stresses calculated by assuming once only shear stresses of 600 MPa and the other time shear stresses of 600 MPa and tensile stresses of 75 MPa is below 0.3%. Thus the error committed by not
taking into consideration the tensile stresses is negligible.

- The measurement used to determine the torque acting on the specimen may be negatively influenced. This is mainly due to mismatches between sensor and rotation axes. In fact the displacements of both points on the beam of the sensor may differ quite much as compared to the case of perfect axes coincidence, due to bending of the sensor wire. The difference between both displacements however remains constant, and thus only torque is directly measured in the setup used in this study.

Fig. 4.20  Rotational and translational mismatches between the three axes
4.7 Conclusion and outlook

A torsional sensor with high resolution and accuracy has been developed to be able to measure torques acting on microstructures. The sensor has an accuracy of 3% of the measured value with a resolution of 0.05 μNm and a working range of ± 200 μNm. This sensor is used in a torsional setup, in which the samples are rotated until failure occurs. During the rotation, both the angle and the acting torque are measured. By analysing the structural response of the system with finite elements, the mechanical properties of the material can be studied. This is mainly done by mixed numerical experimental techniques. This procedure allows the determination of the governing shear-moduli, if torsional experiments are carried out on specimens with different cross-sections.

Failure criteria are established for both silicon and LIGA microstructures. For the metallic specimens, Tresca and von Mises yield criteria are compared, and it is found that a criterion with a yield surface between those surfaces fits the experimental data best. For the silicon specimens, due to their sharp notches, a failure criterion based on energy considerations is used. This criterion states that failure occurs, if the energy per unit thickness around any point along the notch grows faster in function of the radius than a critical rate. An alternative, more general formulation, valid for any geometry of the notch, states that failure occurs if the strain energy per unit thickness exceeds the surface energy in a critical region around the notch defined by a radius $r_c$. These failure criteria are validated by applying them not only to torsional, but also to tensile tests.

In a future step, further tests should be performed to see whether these criteria are well-founded. These tests should include mixed-mode testing, where torsion and tension are applied simultaneously. The torque-sensor developed in this work is insensitive to tensile load and can therefore be used in these combined tests. The tensile force can be measured with a balance, and the resulting elongation of the specimen could be recorded with a microscope-CCD camera system, as done by Mazza et al. [37].

It would also be interesting to get a better understanding of the fracture mechanism at the notch of the silicon sample. This could be done by introducing a piezoresistive element at the critical point, whose fracture would trigger a high-speed camera, recording the propagation of the crack.
M.C. Escher
House of stairs
lithograph 1951
Appendix A  Static torsion of an anisotropic rod with constant rectangular cross-section

All three axes are considered to be main axes of anisotropy.

The rotation $\theta$ is assumed to be small and proportional to $x_1$. The displacements can then be written as:

$$\begin{align*}
    u_2 &= -\theta(x_1) \cdot x_3 \\
    u_3 &= \theta(x_1) \cdot x_2 \\
    u_1 &= \theta_{,1} \cdot f(x_2, x_3)
\end{align*}$$  \hspace{1cm} (A.1)

As St. Venant [56] showed in 1856 the displacement $u_1$ must not be neglected, if the boundary conditions Eq. (A.6) are to be respected. Introducing these displacements in the kinematic relations yields the following equations:

$$\begin{align*}
    \varepsilon_{12} &= \frac{1}{2} (u_{1,2} - x_3 \theta_{,1}) \\
    \varepsilon_{13} &= \frac{1}{2} (u_{1,3} + x_2 \theta_{,1})
\end{align*}$$  \hspace{1cm} (A.2)

The strains are related to the stresses by the constitutive law of the material, which is assumed to have an orthotropic behaviour. In this case the shear-stresses are given by:

$$\begin{align*}
    \sigma_{12} &= 2G_{12}\varepsilon_{12} = G_{12}(u_{1,2} - x_3 \theta_{,1}) \\
    \sigma_{13} &= 2G_{13}\varepsilon_{13} = G_{13}(u_{1,3} + x_2 \theta_{,1})
\end{align*}$$  \hspace{1cm} (A.3)

As the angular change $\theta_{,1}$ is constant, $\sigma_{11} = 0$. Without body forces, the equilibrium condition in $x_1$-direction reduces to:
The equilibrium conditions in the other two directions are automatically satisfied. To determine the shear-stresses two differential equations are needed. One is provided by Eq. (A.4), while the second one is provided by the compatibility equation Eq. (A.5), which is obtained with the aid of Eq. (A.3) by taking derivatives and subtracting both stresses:

\[ G_{13}\sigma_{12,3} - G_{12}\sigma_{13,2} = -2\theta \frac{1}{G_{12}G_{13}} \quad (A.5) \]

In combination with the boundary condition Eq. (A.6) that there are no stresses on the outer surface of the beam, these equations allow the determination of both shear-stresses.

\[ \sigma_{12}n_2 + \sigma_{13}n_3 = 0 \quad (A.6) \]

where the normal vector to the surface \( \mathbf{n} \) is given by \( \mathbf{n} = (0, n_2, n_3) \).

To solve this system a stress function solution method is usually applied. The stress function, called \( \Phi(x_2, x_3) \), is chosen to automatically fulfil Eq. (A.4). Eq. (A.5) then yields a differential equation for \( \Phi \). The stress function is defined by Eq. (A.7).

\[
\begin{align*}
\sigma_{12} &= \theta x_2 \Phi_3(x_2, x_3) \\
\sigma_{13} &= -\theta x_2 \Phi_2(x_2, x_3)
\end{align*}
\quad (A.7)
\]

By choosing \( \Phi \) as in Eq. (A.7) the equilibrium equation Eq. (A.4) is satisfied. The compatibility equation yields:

\[ \frac{\Phi_{22}}{G_{13}} + \frac{\Phi_{33}}{G_{12}} = -2 \quad (A.8) \]

Eq. (A.8) is called the generalized Poisson-equation. With Eq. (A.6) and Eq. (A.7) the corresponding boundary condition for \( \Phi \) is described by Eq. (A.9), which means that the gradient of the stress function \( \nabla \Phi \) is parallel to the normal vector \( \mathbf{n} \) to the surface. This means that \( \Phi \) is constant on the surface, it can there-
fore be set to 0, in the case of rectangular cross-sections.

\[ \Phi_{x_n x_3} - \Phi_{x_3 x_2} = 0 \]  

(A.9)

The stress function \( \Phi \) is developed in a Fourier series, which automatically satisfies the boundary conditions. \( a \) and \( b \) are the half-width and half-height of the cross-section, respectively:

\[ \Phi(x_2, x_3) = \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \alpha_{mn} \cos \frac{m \pi x_2}{2a} \cos \frac{n \pi x_3}{2b} \]  

(A.10)

To determine the coefficients \( \alpha_{mn} \), the difference between strain energy \( U \), Eq. (A.11), and work potential \( W \), Eq. (A.12), has to be minimized with respect to \( \alpha_{mn} \), Eq. (A.13), see Sechler [61].

\[ U = \frac{\pi^2 \theta^2_{1} abL}{8} \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \alpha_{mn}^2 \left( \frac{m^2}{a^2 G_{13}} + \frac{n^2}{b^2 G_{12}} \right) \]  

(A.11)

\[ W = \frac{32 \theta^2_{1} abL}{\pi^2} \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \alpha_{mn} \frac{1}{mn} (-1)^{m+n} \frac{(m+n)^2}{2} \]  

(A.12)

\[ \frac{\partial}{\partial \alpha_{mn}} (U - W) = 0 \]  

(A.13)

The derivation of both \( U \) and \( W \) is explained in Appendix B. The equations Eq. (A.11), Eq. (A.12), Eq. (A.13) yield for the coefficients \( \alpha_{mn} \):

\[ \alpha_{mn} = \frac{128 (-1)^{m+n-1}}{\pi^4 mn} \left( \frac{m^2}{a^2 G_{13}} + \frac{n^2}{b^2 G_{12}} \right)^{-1} \]  

(A.14)

Now the torque \( T \) can be expressed as a function of \( G_{12} \) and \( G_{13} \).
\[ T = \frac{2^{12}}{\pi^{6}} \theta_{1,ab} \sum_{m = 1, 3} \sum_{n = 1, 3} \frac{1}{m^{2} n^{2}} \left( \frac{m^{2}}{a^{2} G_{13}} + \frac{n^{2}}{b^{2} G_{12}} \right)^{-1} \]  

(A.15)

It should be noted that convergence of the infinite double-sum occurs after about 20 terms.
Appendix B  Strain energy and work potential of a rectangular beam subjected to torsion

Strain energy

The strain energy can be written as:

\[ U = \frac{L}{2} \int_{-a-b}^{a+b} \int_{-a-b}^{a+b} (2\sigma_{12}\varepsilon_{12} + 2\sigma_{13}\varepsilon_{13}) \, dx_1 \, dx_2 \]  
(A.16)

By using the constitutive equations Eq. (A.3) and by expressing the shear-stresses as functions of \( \Phi \), Eq. (A.16) yields:

\[ U = \frac{L^2}{2} \left( \int_{-a-b}^{a+b} \int_{-a-b}^{a+b} \frac{1}{G_{12}} \Phi_{12}^2 \, dx_1 \, dx_2 \right) + \int_{-a-b}^{a+b} \int_{-a-b}^{a+b} \frac{1}{G_{13}} \Phi_{13}^2 \, dx_1 \, dx_2 \]  
(A.17)

The two parts of the deformation energy thus obtained are very similar, therefore only \( C_1 \) will be further investigated. By using the Fourierseries of \( \Phi \), \( C_1 \) can be described by:

\[ C_1 = \frac{\pi^2}{4b^2G_{12}} \int_{-a-b}^{a+b} \left( \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} a_{mn} \cos \frac{m\pi x_2}{2a} \sin \frac{n\pi x_3}{2b} \right)^2 \, dx_3 \, dx_2 \]  
(A.18)

The square of the double sum yields a four-fold sum, which is shown in Eq. (A.19). Two different cases have to be considered:

- \( m=m' \) and \( n=n' \): in this case the integration yields \( ab \)
• \( m \neq m' \) or \( n \neq n' \): in all these cases the integrated terms vanish, due to the orthogonality of cosine and sine functions

\[
C_1 = \frac{\pi^2}{4b^2G_{12}} \int_{-a}^{b} \sum_{m=1,3} \sum_{n=1,3} a_{mn} a_{m'n'n'} \left( \cos \frac{m\pi x_2}{2a} \right) \left( \cos \frac{m'\pi x_2}{2a} \right) \left( \sin \frac{n\pi x_3}{2b} \right) \left( \sin \frac{n'\pi x_3}{2b} \right) dx_3 dx_2
\]

(A.19)

\[
C_1 = \frac{\pi^2 ab}{4} \sum_{m=1,3} \sum_{n=1,3} \frac{a_{mn}^2}{b^2G_{12}} \frac{n^2}{m^2}
\]

(A.20)

Following the same procedure, \( C_2 \) can be calculated. Eq. (A.17) and Eq. (A.20) yield:

\[
U = \frac{\pi^2 \theta_{11}^2 abL}{8} \sum_{m=1,3} \sum_{n=1,3} a_{mn}^2 \left( \frac{m^2}{a^2G_{13}} + \frac{n^2}{b^2G_{12}} \right)
\]

(A.21)

**Work potential**

The work potential is a function of the applied torque \( T \) and of the rotation per unit length: \( W = L T \theta_{11} \). The torque \( T \) is expressed as a function of the shear stresses:

\[
T = \int_{-a}^{b} \int_{-b}^{a} (- \sigma_{12} x_3 + \sigma_{13} x_2) dx_3 dx_2
\]

(A.22)
By using Eq. (A.7) and the theorem of Gauss, \( T \) can be expressed as a function of \( \Phi \):

\[
T = 2\theta_{1} \int_{-a-b}^{a+b} \int \Phi(x_2, x_3) \, dx_3 \, dx_2 \quad (A.23)
\]

After replacing \( \Phi \) by its Fourier series and integrating, this yields for the potential energy:

\[
W = \frac{32\theta_{1}^{2} a b L}{\pi^{2}} \sum_{m=1, \, 3}^{\infty} \sum_{n=1, \, 3}^{\infty} a_{mn} \frac{1}{mn} (-1)^{\frac{m+n}{2} - 1} \quad (A.24)
\]
Appendix C  Relative sensitivity matrix

The relative sensitivity matrix $S$ is defined by its components given in Eq. (A.25):

$$s_{ij} = \frac{c_j \partial \omega_i}{\omega_i \partial c_j} \quad (A.25)$$

where $c_j$ are the elastic constants and $\omega_i$ are the circular resonance frequencies. The index $i$ goes from 1 to the number of modes (see Fig. 3.5) considered, whereas the index $j$ goes from 1 to 9 in the case of orthotropic material. Eq. (A.26) shows the relative sensitivity matrix of the silicon sample si1. The elastic moduli take consecutively the values of $E_{11}$, $E_{22}$, $E_{33}$, $G_{12}$, $G_{23}$, $G_{13}$, $v_{12}$, $v_{23}$, $v_{31}$.

$$S = \begin{bmatrix}
0.45 & 0.00 & 0.00 & 0.02 & 0.00 & 0.00 & 0.02 & 0.00 & 0.00 \\
0.45 & 0.01 & 0.00 & 0.03 & 0.00 & 0.00 & 0.01 & 0.00 & 0.00 \\
0.02 & 0.00 & 0.00 & 0.33 & 0.00 & 0.10 & 0.00 & 0.00 & 0.00 \\
0.41 & 0.02 & 0.00 & 0.05 & 0.00 & 0.02 & 0.04 & 0.00 & 0.01 \\
0.34 & 0.03 & 0.00 & 0.12 & 0.00 & 0.00 & 0.04 & 0.00 & 0.00 \\
0.47 & 0.02 & 0.00 & 0.00 & 0.00 & 0.00 & 0.00 & 0.00 & 0.00 \\
0.02 & 0.01 & 0.00 & 0.44 & 0.01 & 0.01 & 0.01 & 0.00 & 0.00 \\
0.02 & 0.49 & 0.00 & 0.02 & 0.01 & 0.00 & 0.10 & 0.00 & 0.00
\end{bmatrix} \quad (A.26)
Appendix D  Dimensionless form of the differential equation of motion of a damped spring-mass system and its solution

The equation of motion of a damped spring-mass system can be written as:

\[ m \cdot x_{tt} + \mu \cdot x_t + k \cdot x = \sum_{n=0}^{\infty} K_n \cdot \cos(n\omega t) \quad (A.27) \]

By setting \( \bar{x} = x/x_{\text{unit}} \) (\( x_{\text{unit}} \) can be arbitrarily chosen, but should have the same order of magnitude as the amplitude) and \( \Theta = \omega t \), Eq. (A.27) yields after some transformations:

\[ b \cdot \bar{x}_{\Theta\Theta} + c \cdot \bar{x}_\Theta + \bar{x} = \sum_{n=0}^{\infty} a_n \cdot \cos(n\Theta) \quad (A.28) \]

The dimensionless coefficients are given by:

\[ a_n = \frac{K_n}{kx_{\text{unit}}} \quad b = \frac{m\omega^2}{k} \quad c = \frac{\mu\omega}{k} \quad (A.29) \]

The solution of the homogeneous part of Eq. (A.28) is of no interest, as it vanishes for \( t \to \infty \). Only the particular solution will be discussed. It is given by:

\[ \bar{x}(\Theta) = \sum_{n=0}^{\infty} \bar{x}_n(\Theta) \quad (A.30) \]

where
\( \chi_n(\Theta) = a_n \left[ \frac{1 - n^2 b}{(1 - n^2 b)^2 + (nc)^2} \cdot \sin(n \Theta) + \frac{-nc}{(1 - n^2 b)^2 + (nc)^2} \cdot \cos(n \Theta) \right] \) \( A.31 \)

The amplitude of this harmonic function is given by Eq. (A.32):

\[ A_n = \frac{a_n}{\sqrt{(1 - n^2 b)^2 + (nc)^2}} \] \( A.32 \)

The amplitude reaches a maximum \( A_{Rn} \) at the resonance frequency \( \omega_R \):

\[ \omega_R = \sqrt{\frac{k}{m} - \frac{\mu^2}{2m^2}} \] \( A.33 \)

\[ A_{Rn} = \frac{a_n}{\sqrt{\left( \frac{c^2}{b} \right) - \frac{1}{4} \left( \frac{c^2}{b} \right)^2}} \] \( A.34 \)

If the damping of the system is weak (\( c^2/b \ll 1 \)), the following approximations can be made at resonance:

\[ b \approx 1 \]
\[ c \approx \frac{1}{Q} \]
\[ A_{Rn} = Q \cdot a_n \] \( A.35 \)
Appendix E Solving non-linear differential equations

The differential equations considered are dimensionless non-linear differential equations of the second order, as shown in Eq. (A.36)

\[ b \cdot \ddot{x} + c \cdot \dot{x} + \ddot{x} + e \cdot h(\dot{x}, \ddot{x}) = \sum_{n=0}^{\infty} a_n \cdot \cos(n\Theta) \quad (A.36) \]

Assuming that \( h(\dot{x}, \ddot{x}) \) is a polynomial function and that \( e << 1 \), the solution \( \ddot{x} \) can be written as a Taylor-series:

\[ \ddot{x} = \sum_{i=0}^{\infty} e^i \cdot \ddot{x}_i \quad (A.37) \]

\( \ddot{x}_i \) are unknown functions of \( \Theta \). The first and second derivative can now be written as:

\[ \dot{x},_\Theta = \sum_{i=0}^{\infty} e^i \cdot \dot{x}_i, \Theta \quad (A.38) \]

\[ \ddot{x},_\Theta = \sum_{i=0}^{\infty} e^i \cdot \ddot{x}_i, \Theta \quad (A.39) \]

Inserting Eq. (A.37) and Eq. (A.38) in Eq. (A.36) and by setting terms with the same \( e^i \) coefficient equal to 0 yields the system of equations shown in Eq. (A.39). The functions \( h_i \) of the system can be computed from the non-linear function \( h \), and their Fourier series contains higher frequent components, which explains the multiple resonance phenomenon. Each equation has the form of the differential equation of a damped linear mass-spring system and can be solved with conventional methods. Thus \( x_i \) can be calculated if \( x_{i-1} \) is known. The initial function \( x_0 \)
can be computed from the first equation of Eq. (A.39).

\[ \begin{align*}
    b \cdot \ddot{x}_0,\Theta + c \cdot \dot{x}_0,\Theta + \dot{x}_0 &= \sum_{n=0}^{\infty} a_n \cdot \cos(n\Theta) \\
    b \cdot \ddot{x}_1,\Theta + c \cdot \dot{x}_1,\Theta + \dot{x}_1 &= h_1(\ddot{x}_0) \\
    b \cdot \ddot{x}_2,\Theta + c \cdot \dot{x}_2,\Theta + \dot{x}_2 &= h_2(\ddot{x}_0, \dot{x}_1)
\end{align*} \]  

(A.39)

The solution of the non-linear differential equation can now be approximated by introducing the solutions of Eq. (A.39) in Eq. (A.37).
Appendix F Quadratic differential equation of the second order

The differential equation considered is of the form:

\[ b \cdot \ddot{x} + c \cdot \dot{x} + \ddot{x} + \varepsilon \cdot \dddot{x} = a \cdot \sin \Theta \]  \hspace{2cm} (A.40)

By taking into consideration terms of the order \( \varepsilon^2 \), the solution can be approximated by:

\[
\ddot{x} = [A_{11} \sin \Theta + A_{12} \cos \Theta] + \varepsilon [A_{21} + A_{22} \sin (2 \Theta) + A_{23} \cos (2 \Theta)] + \varepsilon^2 [A_{31} \sin \Theta + A_{32} \cos \Theta + A_{33} \sin (3 \Theta) + A_{34} \cos (3 \Theta)] + O(\varepsilon^3) \hspace{2cm} (A.41)
\]

The coefficients are given by:

- \( A_{11} = \frac{a(1 - b)}{(1 - b)^2 + c^2} \)
- \( A_{12} = \frac{a(-c)}{(1 - b)^2 + c^2} \)
- \( A_{21} = \frac{-1}{2} (A_{11}^2 + A_{12}^2) \)
- \( A_{22} = \frac{(1 - 4b)A_{11}A_{12} + c(A_{11}^2 + A_{12}^2)}{(1 - 4b)^2 + (2c)^2} \)
- \( A_{23} = \frac{-1}{2} (1 - 4b)(A_{11}^2 + A_{12}^2) + 2cA_{11}A_{12} \)
  \( (1 - 4b)^2 + (2c)^2 \)
\[
A_{31} = \frac{(1 - b)(-2A_{11}A_{20} + A_{11}A_{22} - A_{12}A_{21})}{(1 - b)^2 + c^2} + \frac{c(-2A_{12}A_{20} + A_{11}A_{21} - A_{12}A_{22})}{(1 - b)^2 + c^2}
\]

\[
A_{32} = \frac{(1 - b)(-2A_{12}A_{20} + A_{11}A_{21} - A_{12}A_{22})}{(1 - b)^2 + c^2} - \frac{c(-2A_{11}A_{20} + A_{11}A_{22} - A_{12}A_{21})}{(1 - b)^2 + c^2}
\]

\[
A_{33} = \frac{(1 - 9b)(-A_{11}A_{22} - A_{12}A_{21}) + 3c(A_{11}A_{21} - A_{12}A_{22})}{(1 - 9b)^2 + (3c)^2}
\]

\[
A_{34} = \frac{(1 - 9b)(A_{11}A_{21} - A_{12}A_{22}) - 3c(-A_{11}A_{22} - A_{12}A_{21})}{(1 - 9b)^2 + (3c)^2}
\]
Appendix G  Differential equation for vibration of an orthotropic plate

The differential equation governing the vibration of thin plates, see Leissa [35], will be deduced using the following assumptions:

- The thickness of the plate is small as compared to its lateral dimensions. Due to this fact stresses with a component in $x_3$ direction can be neglected.
- Normals to the midplane of the undeformed plate remain straight and normal to the midplane during deformation, which means that shear effects are not taken into consideration.
- In-plane inertia forces are neglected.
- The height of the plate remains constant.
- Displacements and slopes are considered to be small. This means that linear elastic theory can be used and that the equilibrium equations are not written in the deformed but in the original state.

To derive the governing equation, the procedure is the same as usual, equilibrium on a differential element has to be formulated. This element as well as the forces and moments per unit length acting on it are shown in Fig. A.1.

---

**Fig. A.1** Forces and moments per unit length acting on a plate element
forces shown only on the faces $x_1 = dx_1$ and $x_2 = dx_2$
moments shown only on the faces $x_1 = 0$ and $x_2 = 0$
The equilibrium of the plate element in Fig. A.1 yields the equations:

\[ Q_{1,1} + Q_{2,2} = \rho h u_{3,tt} \]  
\[ (A.42) \]

\[ Q_{1} - M_{1,1} - M_{12,2} = \frac{\rho h^3}{12} u_{3,1tt} \]  
\[ (A.43) \]

\[ Q_{2} - M_{2,2} - M_{12,1} = \frac{\rho h^3}{12} u_{3,2tt} \]  
\[ (A.44) \]

The displacements in \( x_1 \) and \( x_2 \) direction of a general point \( P \) can be expressed by the motion of a point \( O \) of the midplane lying on the same normal line, and by a term due to the slope at this point, see Eq. (A.45). The kinematic relations are given by Eq. (A.46):

\[ u_1 = u_{10} - x_3 u_{3,1} \]  
\[ (A.45) \]

\[ u_2 = u_{20} - x_3 u_{3,2} \]

\[ \varepsilon_{ij} = \frac{1}{2} (u_{i,j} + u_{j,i}) \]  
\[ (A.46) \]

Due to these assumptions, there remain only three non-vanishing strains and stresses. Eq. (3.1) simplifies to Eq. (A.47).

\[
\begin{pmatrix}
\varepsilon_{11} \\
\varepsilon_{22} \\
2\varepsilon_{12}
\end{pmatrix} =
\begin{pmatrix}
\frac{1}{E_{11}} & \frac{v_{12}}{E_{22}} & 0 \\
\frac{v_{12}}{E_{11}} & \frac{1}{E_{22}} & 0 \\
0 & 0 & \frac{1}{G_{12}}
\end{pmatrix}
\begin{pmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{12}
\end{pmatrix}
\]  
\[ (A.47) \]

Integrating the stresses over the height of the plate leads to the bending and twisting moments. By inverting Eq. (A.47) the stresses can be expressed as functions
of strains. Using Eq. (A.45) and Eq. (A.46) and introducing them into the inverted equation of Eq. (A.47), the stresses, and thereby the moments can be expressed in terms of $u_3$ and its derivatives, which is done in Eq. (A.48) to Eq. (A.50).

\[
M_1 = \int_{\frac{h}{2}}^{\frac{h}{2}} \sigma_{11}x_3 \, dx_3 = -D_{11}(u_{3,11} + \nu_{21}u_{3,22}) \tag{A.48}
\]

\[
M_2 = \int_{\frac{h}{2}}^{\frac{h}{2}} \sigma_{22}x_3 \, dx_3 = -D_{22}(\nu_{12}u_{3,11} + u_{3,22}) \tag{A.49}
\]

\[
M_{12} = \int_{\frac{h}{2}}^{\frac{h}{2}} \sigma_{12}x_3 \, dx_3 = -2D_{12}u_{3,12} \tag{A.50}
\]

$D_{11}$ and $D_{22}$ are the bending plate stiffnesses in $x_1$ and $x_2$ direction respectively, $D_{12}$ is the torsional stiffness. Their values are shown in Eq. (A.51).

\[
D_{11} = \frac{E_{11}h^3}{12(1 - \nu_{12}\nu_{21})}
\]

\[
D_{22} = \frac{E_{22}h^3}{12(1 - \nu_{12}\nu_{21})}
\]

\[
D_{12} = \frac{G_{12}h^3}{12}
\]

(A.51)

Substituting the expressions of Eq. (A.48) to Eq. (A.50) for the moments $M_1$, $M_2$
and $M_{12}$ into Eq. (A.43) and Eq. (A.44) allows to express the shear forces $Q_1$ and $Q_2$ as functions of the plate stiffnesses and the out-of-plane displacement $u_3$. The expressions thus found, in combination with Eq. (A.42), lead to the differential equation for plate vibration Eq. (A.52). The constant parameters in this equation are functions of the two Young’s moduli $E_{11}$ and $E_{22}$ as well as of the shear-modulus $G_{12}$ and the Poisson coefficient $v_{12}$.

$$D_{11}u_{3,1111} + 2(v_{21}D_{11} + 2D_{12})u_{3,1122} + D_{22}u_{3,2222} + \rho \phi u_{3,tt} = 0 \quad (A.52)$$
M.C. Escher
Drawing hands
lithograph 1948
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