Dynamics of Shape Memory Alloy coated micro cantilevers
- theory and experiments -

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Preface

This project is the result of a collaboration between the groups Dynamics & Control and Micro & NanoScale Engineering. The experience of both groups about dynamics, modeling, materials science and experimental techniques is combined, in order to investigate the thermomechanical and dynamical behavior of Shape Memory Alloy coated micro cantilevers. The results give way to research on micro vibration control and future joint projects of these groups.

During this research, the help of experts from various fields of work was of great value. I want to thank Marc van Maris of the Mechanics of Materials group for the opportunity to use the equipment in the Multi-scale laboratory. From the Micro & NanoScale Engineering group, I thank Willie ter Elst for helping with the realization of the experimental setup, as well as Eric Homburg, for giving advice about the optics and electronics.

I greatly appreciate the effort of dr. Xi Wang of Harvard University for sputter coating and annealing the NiTi thin films on our micro cantilever samples. With these excellent samples, we were able to do several unique and interesting thermomechanical and dynamical experiments. I am also very thankful of Michael Hopper (formerly working at Quintenz Hybridtechnik) who designed in his spare time the piezoelectric amplifier electronic circuit.

I thank prof. dr. Henk Nijmeijer for our inspiring discussions during the project. Last, but not least, I thank dr. Yves Bellouard for his personal coaching on every aspect of the project. I could walk by almost anytime and I could always expect an enthusiastic reaction on my results.
Abstract

With the increasing attention for the use of Micro Electro Mechanical Systems (MEMS) in various fields of work, gaining knowledge about the dynamics of these devices is an interesting new research topic. In this project, a structural solution for actively influencing the dynamics of a micro cantilever, using a Shape Memory Alloy (SMA) coating, is investigated. An SMA shows temperature- and stress-induced martensitic phase transformations, drastically changing the material’s mechanical properties and adding damping. The influence of this change on the thermomechanical and dynamical behavior of the cantilever is experimentally and theoretically analyzed.

Samples of commercially available Silicon micro cantilevers are sputter-coated with equiatomic Nickel-Titanium (NiTi). To investigate the thermomechanical behavior of the coated cantilevers, temperature-stress curves are experimentally obtained. The curves clearly show two-step hysteretic phase transformations between Martensite, R-Phase and Austenite. Several material properties can be deduced from the slope of the curves. A thermomechanical model is formulated, describing the stress evolution in the interface layer between the film and substrate as function of temperature. The stress is decomposed into bimorphic stress and phase transformation stress. For modeling the phase transformation stress, several mathematical models are discussed. Eventually the Krasnosel’skii-Pokrovskii hysteron is chosen because of its appealing simplicity, while still capable of capturing most of the geometrical properties of the hysteresis curve.

An experimental setup is designed, capable of exciting the micro cantilevers to high frequencies, simultaneously heating them and measuring their dynamic response. The results show that the flexural resonance frequency of the beam for increasing temperature first decreases due to a change of the geometry of the cross-section. Upon further increasing the temperature, the phase transition results in an increase of the resonance frequency up to 10%. Near the flexural resonance, a second resonance frequency is seen, only slightly changing as function of temperature. The two modes separate or merge for increasing temperature. These observations make it difficult to assess damping, but pose an interesting new research topic. A model of the structural dynamic of the cantilevers is made, including the temperature dependence of the resonance frequencies. The numerical results partially correspond with with the measurements.

The results show that with an SMA coating it is possible to actively influence the dynamics of the cantilevers. Further research has to show whether the influence is sufficient for useful applications in MEMS.
Samenvatting

In de afgelopen decennia is de interesse in het gebruik van Micro Electro-Mechanische Systemen (MEMS) toegenomen. Het analyseren van het dynamisch gedrag van deze systemen is daarbij een nieuw onderzoeksgebied. In dit project is een structurele oplossing voor het actief aanpassen van het dynamisch gedrag van een eenzijdig ingeklemde micro balk onderzocht, door middel van het aanbrengen van een film van geheugenmetaal. Onder invloed van temperatuur en spanning vertoont geheugenmetaal drastische veranderingen in materiaaleigenschappen. De invloed hiervan op het thermomechanische en dynamische gedrag van de balk is experimenteel en theoretisch onderzocht.

Commercieel verkrijgbare Silicium micro balken zijn met een dunne film Nikkel-Titanium (NiTi) gecoat door sputterdepositie. Het thermomechanische gedrag van deze balken is beoordeeld door de temperatuur-spanningscurves te meten. Er is een tweetraps facettransformatie zichtbaar tussen de Martensiet fase, R-fase en Austeniet fase. Verschillende materiaalparameters kunnen vervolgens bepaald worden met behulp van deze grafieken. Een thermomechanisch model is geformuleerd, dat het spanningsverloop in de film bij de aanhechting met de balk beschrijft. De spanning is daartoe opgedeeld in facettransformatie spanning en in spanning die veroorzaakt is door een verschil in de thermische expansiecoëfficiënten. Voor het beschrijven van de facettransformatie spanning wordt een aantal modellen besproken. Het Krasnosel’skii-Pokrovskii hysteron wordt uiteindelijk gekozen vanwege haar eenvoud, terwijl het model tevens de belangrijkste eigenschappen van de hystereselus beschrijft.

Er is een experimentele opstelling ontworpen, waarmee het mogelijk is de balken te exciteren, op te warmen en de dynamische responsie te meten. De resultaten laten zien dat de eigenfrequentie voor buiging bij stijgende temperatuur lichtelijk neemt, om na facettransformatie tot 10% toe te nemen. Rond deze buigresonantie is een tweede resonantie te zien, welke licht verandert als functie van de temperatuur. Deze resonanties vallen samen of scheiden zich bij stijgende temperatuur. Dit gegeven bemoeilijkt het onderzoeken van de dempingseigenschappen van het materiaal, maar levert daarentegen nieuwe onderzoeksvragen op. Een model van de structurele dynamica van de balken is geformuleerd, waarin de resonantiefrequenties afhankelijk zijn van de temperatuur. Deze numerieke simulaties worden vergeleken met de gemeten responsies.

De resultaten laten zien dat het mogelijk is met een SMA coating het dynamisch gedrag van de balken te beïnvloeden. Aanvullend onderzoek is nodig om te onderzoeken of de invloed van dit de facettransformaties voldoende is voor concrete toepassingen in MEMS.
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Chapter 1

Introduction

In the last decades, engineering on micro scale gained increasingly more attention. Integrating and embedding micro components in devices reduces production costs and power consumption, increases efficiency, flexibility and functionality of the application. Micro scale engineering research gives great opportunities in improving the quality and costs of micro and macro devices. An important class of micro scale applications are the micro electro mechanical systems (MEMS). These devices combine electronics and mechanics in a system, operating on micro scale. They are used as sensors and actuators in automotive industry, medical applications and many other fields of work. In Figure 1.1 some MEMS products are shown.

(a) Large force electrostatic MEMS comb drive. A potential difference over the two combs results in an electrostatic force, resulting in a lateral displacement of the intermediate body. The device is used to actuate small structures, such as micro mirrors [MEM07].

(b) MEMS electrostatically driven gear. At every actuation step, triggers rotate the gear with one increment [San08].

Figure 1.1: Scanning Electron Microscope pictures of applications of MEMS.

With the use of MEMS as sensors and actuators, analysis and control of their dynamics becomes an important field of research. Investigation and improvement of the dynamic behavior of MEMS can lead to better performance. Because of the small size, innovative solutions are required to change the dynamic behavior of a micro device, to meet the desired
performance. Furthermore, measurements of the mechanical and dynamical behavior of MEMS are not straightforward, because standard (macro) experimental techniques are not always applicable at smaller scale.

In this project, a structural solution for actively influencing the dynamics of a micro scale structural element is investigated. A micro cantilever beam is coated with a Shape Memory Alloy (SMA) thin film. The properties of this material change drastically when varying the temperature and stress. The mechanics and dynamics of the coated cantilever are investigated both analytically and experimentally, providing better knowledge about the properties of SMA thin films and the applicability of the material for structural vibration control on micro scale.

In this chapter, an introduction on applications of micro cantilevers and Shape Memory Alloys is given. We conclude with the problem formulation of this project.

1.1 Applications of micro cantilevers

In micro technology cantilevers are used as sensors, measuring the interaction with a substrate or surroundings, or a change in their own properties as a result of this interaction. Cantilevers are also used as actuators or switches, with various underlying physical principles of actuation. In this section, some interesting examples are discussed.

A Scanning Electron Microscope (SEM) image of a micro cantilever is shown in Figure 1.2(a). This cantilever is used in Atomic Force Microscopy as a sensor. The working principle of an Atomic Force Microscope (AFM) is to measure the interaction of a probe -the cantilever- with a substrate. The deflection of the cantilever gives information about the material surface topography, but also on other surface and material properties. In Appendix A, more information about the working principle of an AFM is given.

![A SEM picture of a Silicon cantilever, used in Atomic Force Microscopy [McG07].](image1)

![An artistic impression of biomechanical micro cantilevers [NYU07].](image2)

Figure 1.2: Examples of the application of micro cantilevers as sensors.

Dimensions of these types of micro cantilevers differ a lot, depending on the application. In
1.1 Applications of micro cantilevers

In general, the length can range from 60 µm to 600 µm, the width from 20 µm to 60 µm and the thickness from 0.5 µm to 10 µm. The most commonly used material is single-crystal Silicon (Si), because its properties are well known and because the material is suitable for micro machining (e.g. photo-lithography and chemical etching or Focused Ion Beam milling). This design is required to use the cantilever when detecting the very small interaction forces with the substrate and subsequent displacements. The cantilever has a low stiffness. This is an advantage, because a low stiffness results in large displacements when the forces are small \( s = \frac{F}{k} \). Being such a sensitive sensor, the cantilever is also sensitive for disturbances. Several measures are taken to reduce the influence of disturbances. Still, surrounding noise and noise from other components in the AFM have significant influence on the measurements.

Another type of cantilever is shown in Figure 1.2(b). This is a bio-mechanical sensor. The cantilever has a reactive surface, selective for a substance of which the concentration is to be measured. When the substance reacts with the surface, the mass and stiffness change, which can be measured as a deflection or a change in resonance frequency. These cantilevers are generally very thin (< 1µm), but have a large surface area. The reactive surface of these cantilevers is as large as possible, while the cantilever is sensitive to small changes in the mass or stiffness.

Micro cantilevers are also used as actuators. If the cantilever consists of two materials with different thermal expansion coefficient, a change of temperature results in stress variations and subsequently a deflection of the cantilever tip, called the bimorph effect. Such a design is shown in Figure 1.3(a). The cantilever is heated resistively by passing a current through the circuitry, fabricated on the cantilever and substrate. As in macro scale, these bimorph structures can be used as switches or levers. Another actuator design is shown in Figure 1.3(b). This is an electrostatic switch. A potential difference over the source and the gate results in an electrostatic force, attracting the cantilever tip to the drain. Another actuation principle is to use magnetic force to deflect the beam or to laminate the cantilever with piezoelectric material.

![Figure 1.3: SEM pictures of the application of micro cantilevers as actuators.](image-url)


1.2 Shape Memory Alloys

Shape Memory Alloys are materials, showing reversible martensitic phase transformations, induced by temperature or stress [OR05, Wan07]. These phase transformations can result in the so-called Shape Memory Effect and the Superelasticity Effect.

The Shape Memory Effect is a process, where a seemingly plastic deformation in Martensite phase is undone by heating the material to Austenite phase, called self-accommodation of the crystals. The recovery of the original shape in Austenite can be seen as a ’memorized’ shape. The Superelasticity effect is the process of recovery of large strains (up to 10\%) at certain temperatures, during a load cycle. The reason for this recovery is the stress-induced phase transformation. In Figure 1.4 both processes are shown in the temperature-stress plane. It can be seen in the figure, that the phase transition temperatures depend on the stress levels and that transformation can occur both temperature-induced and stress-induced. This dependence is described by Clausius-Clapeyron equation

\[ \frac{d\sigma}{dT} = c, \tag{1.1} \]

where \( c \) is a constant. The temperature dependence of the transformation temperatures is visualized as the solid lines.

![Figure 1.4](image-url)

Figure 1.4: Critical temperature and stress for phase transformation in an SMA. The Clausius-Clapeyron relation is drawn as the solid lines. The dashed line indicate the R-phase transition temperatures. Temperature-induced and stress-induced transformation is indicated with the arrows. The temperatures below indicate the zero stress transformation temperatures, further explained below. A graphical representation of the crystal structure in various phases is also shown.
For temperature induced transformation the stress level remains constant, while the transformation takes place. When cooling from high temperature at constant stress no macroscopic change takes place, but the crystals reorient from the Austenite crystal structure to Martensite, which can have several crystal structures. If no mechanical load is applied, heating again does not change the macroscopic shape of the material. When the material is deformed in Martensite shape, a seemingly plastic deformation on macroscopic scale is obtained. This appears like plastic deformation, because the strain-stress curve is similar. However, when the heating the material again, the Austenite phase is formed and the original shape of the material is recovered.

The phase transitions are reversible, but show a large difference in back and forward transformation temperatures; a hysteresis phenomenon. This is shown in Figure 1.5. Here the fraction of the Austenite (high temperature) phase is plotted against temperature. At $T = T_0$, the material is in full Martensite phase. When heating, the Austenite phase starts forming from $T = A_s$. Further heating increases the Austenite fraction, until the material has fully transformed at $T = A_f$. Upon cooling, Martensite phase starts forming at $T = M_s$, a lower temperature than $A_s$. At $T = M_f$ the material is in full Martensite phase again.

![Figure 1.5: Hysteresis in an SMA. Direction of the temperature profile is indicated with the arrows. The transformation temperatures are also indicated. The dashed lines indicate the minor loops.](image)

The phase transitions result in hysteresis in the temperature-stress plane, which is also shown in Figure 1.4 as four solid lines. On the zero stress axis the zero stress transformation temperatures are shown. These are Martensite finish $M_f$, Martensite start $M_s$, Austenite start $A_s$, and Austenite finish $A_f$ temperatures. Combining Figure 1.4 and 1.5, we conclude that the shape and location of the hysteresis curve in the temperature-stress plane are depending on the initial stress levels and the type of transformation mechanism, i.e. temperature-induced or stress-induced. If the temperature cycle is smaller than $M_f$ to $A_f$, the so-called minor loops appear. These are shown as the dashed lines in Figure 1.5. The existence of the minor loops inside the big major loop, implies that the transformation hysteresis is
strongly dependent on the history of the temperature cycles.

**Nickel-Titanium**

One of the most commonly used SMA materials is Nickel-Titanium (NiTi). The transition temperatures of NiTi greatly depend on the composition and on heat treatments. It is reported that for equiatomic NiTi (∼ 50 wt% Ni), when stress levels are low, an intermediate phase transition takes place, the R-phase transition [OR05]. The appearance of this R-phase only at low stress levels is also explained by the Clausius-Clapeyron equation from Figure 1.4. The R-phase transition temperature evolves along the dashed line. We can see that at high temperatures the transition temperature equals the Austenite transition temperature, so the material transforms instantly from Martensite to Austenite.

The phase transformation in equiatomic NiTi is accompanied with large changes in the material properties of the alloy. A change in Young’s modulus and thermal expansion of up to 100% is observed. These properties influence the mechanics and dynamics of the material significantly. Furthermore, the sliding of crystal planes, especially during R-phase transformation gives friction, which results in high damping [Wut03]. The change of these properties during phase transition will have influence on the mechanical and dynamical behavior of materials, containing NiTi.

1.3 Problem definition

Micro cantilevers, used as sensors and actuators, can benefit in several ways from an SMA coating. The basis for this benefit is that the properties of the film can be changed, having influence on the behavior of the cantilever. This may increase the performance of the cantilever in a situation where the operation requirements change.

For the AFM cantilever and the bio-mechanical sensor (Figure 1.2), small disturbances have large influence on the measurement, because the cantilevers are sensitive. Since the SMA film in some cases shows large damping during phase transformation, phase transformations can be seen as a means of active damping. The temperature of the film then determines whether the damping is ‘switched on’ or not. Another benefit is that the cantilever has a tunable stiffness. This means that for specific cases one could choose to do the measurement with a stiffer cantilever. It is to be investigated if the influence of the phase transformations on the stiffness and damping is significant for this application. The bimorph cantilever (Figure 1.3(a)) is a system that uses the deposited material to deform, as a result of a thermal mismatch. As discussed above, an SMA film can show large strain recovery for temperature- and stress-induced transformation, which can greatly increase the stroke of the actuator. The electrostatic switch (Figure 1.3(b)) can benefit from an SMA film, because the switch has a tunable switching threshold. If the stiffness can be tuned, the applied voltage for attracting the cantilever to the drain varies. This makes the switch more versatile, though still relatively simple to manufacture.

In a larger scope, an SMA thin film can be deposited on other micro or macro structures. If we are able to change the temperature of the film, damping or stiffness can be added if desired. Depositing the film on a micro cantilever gives the opportunities of investigating the
suitability of using SMA on micro scale. It also gives more insights in the thermomechanical behavior of the material, which still is a relevant research topic.

The problem definition is formulated as follows:

"Investigate the suitability of using a Shape Memory Alloy coating on micro cantilevers to actively influence its dynamic behavior."

The first step is to analyze the requirements for measuring and analyzing the behavior of the coated cantilever. An experimental setup is designed, meeting these requirements. Samples of NiTi coated micro cantilevers are prepared for thermomechanical and dynamical measurements. Secondly, the thermomechanical behavior of the cantilever is analyzed. With these results a prediction can be made about the dynamical behavior. Characterization of the thermomechanical and dynamical behavior of the cantilever is done using the experimental setup and is compared to analytical results. A semi-empirical model, based on theoretical analysis and experimental results is formulated, which is useful for further assessment of the possibilities and limitations of the proposed design.

Outline of the report

The report is built up as follows. Chapter 2 deals with the design and realization of an experimental setup for dynamic and thermal analysis of NiTi coated micro cantilevers. In Chapter 3, modeling approaches for the electrothermal, thermomechanical and dynamical behavior are presented, useful for understanding the behavior of the system. Measurement results are discussed in Chapter 4. We also propose a semi-empirical model to describe the behavior of the coated cantilever. Finally, conclusions and recommendations are stated in Chapter 5.
Chapter 2

Experimental setup

For thermomechanical experiments, measurement equipment is available at the Multi-Scale Laboratory at the department of Mechanical Engineering at the TU/e. To investigate the dynamical response of the micro cantilevers, a specialized experimental setup is designed. An AFM would also do, but availability issues, software limitations and lack of specifications limit its usefulness for this project. Furthermore, the objective is to investigate the dynamic properties of NiTi coated micro cantilevers for varying temperature. An experimental setup is designed, capable of exciting the cantilevers, varying their temperature and measuring the vibrations. This gives insight in the possibilities, limitations and applicability of the design and provides a realistic view on this approach on dynamic control at micro scale, using a NiTi coating. The specifications of the setup, the functions of its components, calibration and preliminary measurements are discussed in the following sections.

2.1 Specifications

Schematically the measurement approach is shown in Figure 2.1. Specifications are indicated in the figure. We will discuss the requirements on each component.

Figure 2.1: Dynamic measurement approach. The sample, a chip with a NiTi coating, is placed on an excitation stage. The sample is positioned under a sensor, which measures the tip vibrations as a result of excitation of the chip.
Cantilevers

Uncoated micro cantilevers with known properties are coated with an equiatomic NiTi thin film by means of sputter deposition. As deposited, the material is amorphous. The film is annealed in order to crystallize it, giving the material its phase transformation properties. The cantilever chips have to be properly handled and suspended for assembly into the excitation stage and for heating the film.

Excitation and measurement

Depending on its application, the resonance frequency of an AFM cantilever can range from 5 up to 500 kHz. In contact mode, the probes are required to be sensitive to small force fluctuations, so low stiffness is desired, giving a low resonance frequency (recall that $\omega_0 = \sqrt{k/m}$). For tapping mode AFM, stiff cantilevers (thus having a high resonance frequency) are used. The cantilever is operated near or at its resonance frequency. Interaction with the substrate results in shifts of the resonance frequency, the amplitude and the phase, which gives information about the sample. Introducing damping or additional shifts in the resonance frequency is not desired. A NiTi coated cantilever will not improve the measurement in tapping mode, we therefore concentrate on low stiffness cantilevers.

In contact mode Atomic Force Microscopy, the cantilever moves across the surface of the sample at low speed, typically at 0.1 Hz - 1 Hz over a stroke of several microns. A shock or other disturbance can excite the cantilever, causing it to vibrate, which in this case is not desirable. It is therefore useful to investigate the possibility of changing the dynamics in such a case. These cantilevers generally have a resonance frequency between 10 kHz and 100 kHz. Excitation to and beyond the resonance frequency is necessary. To measure the response to this excitation, a measurement system capable of high frequency dynamic measurements and data acquisition hardware and software with high sampling rates is required.

In typical AFM measurements, the amplitude of excitation of a cantilever in tapping mode is in the order of tenths of nanometers. Larger amplitudes can cause breaking, delamination or unstable behavior. Small amplitudes ($\pm 50$ nm) at high frequencies (10-100 kHz) are required for analyzing the cantilevers. Measuring these vibrations requires a high sample rate and a resolution in the order of nanometers.

Heating

Changing the temperature of the NiTi film is expected to change its mechanical and dynamical properties significantly, as a result of phase transformations. For an equiatomic NiTi film on a glass or Silicon substrate, the transition temperatures are reported in the range of $-10^\circ$C to $100^\circ$C [Wan07]. Relevant temperatures for dynamic operation may differ. A way of heating and cooling the NiTi film without perturbing the excitation or obstructing the sensor is required. Moreover, the temperature needs to be dynamically changed, so fast heating and cooling is preferred.
2.2 Experimental setup design

The proposed setup is shown in Figure 2.2.

Here we distinguish

- a NiTi coated cantilever chip, supported by a steel mounting plate for easy handling and positioning;
- a piezo element with amplifier and high voltage power supply, to excite the cantilever chip to high frequencies;
- an optical measurement head with amplifier, supported by a stiff measurement frame;
- a five degree of freedom manual positioning stage, to position the cantilevers in the focal point of the optical measurement head;
- a current source and wire contacts to the NiTi film, to resistively heat the film (Joule heating);
- data acquisition hardware and software.

These components and their assembly are discussed in the following sections.
2.2.1 Sample preparation

The micro cantilevers, used in these experiments, are supplied by µMasch [Mik07]. The cantilevers are tipless, rectangular and made of single crystal Silicon. A chip contains six cantilevers of different length and thus have a different resonance frequency. Typical dimensions are \(lxwxh = 300 \times 35 \times 2 \ \mu m\). The chips have the dimensions \(lxwxh = 3.4 \times 1.6 \times 0.4 \ mm\). Other specifications are found in Appendix C.

The cantilever chips are coated at Harvard University by Dr. Xi Wang of Prof. Vlassak’s Group. A thin film of equiatomic NiTi approximately 1µm thick is co-sputtered under high vacuum \((1 \cdot 10^{-4} \ mTorr)\) at room temperature. After sputtering, the film is annealed in the same vacuum at 500°C for approximately 30 minutes, to fully crystalize the film. A SEM picture of the coated cantilever chip is shown in Figure 2.3.

![Figure 2.3: SEM image of NiTi coated Silicon cantilevers on chip NSC12. The glue, used for attaching the chip on the supporting plate, is seen, as well as a wire, glued to the NiTi film. The three cantilevers of different length are distinguished on the right hand side of the chip.](image)

The chips are mounted on a supporting steel plate with an epoxy (EPO-TEK 353ND). In Figure 2.4 it can be seen that the film contains some inhomogeneous parts, the 'white' spots. It is not clear what causes this contamination, but it is relatively small and it is expected to have no influence on the behavior of the material. In this figure is also seen that the film is deposited over the sides of the cantilever and that the cantilever sides are tapered, due to the anisotropic etching process. The sputtering process and subsequently annealing of the film introduces residual stresses in the film. The cantilevers have an initial curvature as a result of the residual stress. More on this matter is discussed in Chapter 4.
2.2 Experimental setup design

(a) Tip of the coated cantilever. The sides of the cantilever are tapered, due to the anisotropic etching process. The white spots are also shown.

(b) Cantilever connection to chip. The NiTi film is covering the chip well.

Figure 2.4: SEM pictures of the deposited film on the Silicon cantilever.

2.2.2 Excitation stage

As noted in Section 2.1, the excitation stage has to be capable of exciting the cantilever to and beyond its resonance frequency. For high frequency excitation at small amplitudes, piezoelectric elements are a suitable choice. When a sinusoidal voltage profile is supplied, the piezoelectric element will expand and contract. The direction of largest expansion depends on the way the element is prepared; the poling process. Suppose the poling direction is the '33' direction as shown in Figure 2.5. If the element is operated to expand along the '33' axis, the voltage has to be applied over the thickness of the element. Relevant material properties in this direction are listed in Table 2.1. Applying a positive voltage will result in an expansion in the '33' direction and a - much smaller - contraction in the other two dimensions. The amount of displacement relative to its length depends on the electric field applied and a material property, the piezoelectric constant in the relevant direction $d_{33}$, which is depending on the crystal orientation.

Figure 2.5: Schematic overview of the working principle of a piezoelectric element. At $V = 0$, the piezo has the dashed geometry. At $V = +V_0$, the piezo expands $\Delta L$ in the '33' direction and contracts in the '22' (and '11') direction, drawn as the solid geometry.
Choice of a piezo oscillator

In [Phi01] it is thoroughly explained what properties have to be considered when selecting a piezo element. Important design considerations are the application, the size, the material and the possible need for prestraining.

There are two types of piezo actuators: single crystal and stacked actuators. Single crystal actuators (thickness from 150\(\mu\)m up to several centimeters) require a high voltage to fully expand, while stacked actuators (generally with <100\(\mu\)m layers) require much lower voltages and generate larger displacements. Stacked actuators require prestraining, since they can delaminate when in tension. A single crystal piezo element only needs prestraining in the case of large tensile forces.

For our application, small displacements at high frequencies are required. In that case, a single crystal piezo is suitable, considering its high stiffness and the fact that it does not require prestraining. We choose the PIC181 PZT ceramic, supplied by Physik Instrumente. This material has a high Young’s modulus (\(E_{33} = 166\) GPa), high mechanical quality factor (\(Q_m = 2000\)) and dissipates little heat. This makes it very suitable for our application, because we want to operate it at high frequencies and heat generation should be as little as possible to avoid its influence on the cantilever. The required operating voltage, however, is quite high, due to its low piezoelectric constant. The relevant properties of PIC181 are listed in Table 2.1 and Appendix B. The chosen element is a thin plate with dimensions 10x10x0.5mm.

Amplifier design

To operate the piezo, an excitation signal is generated, amplified to a desired voltage amplitude and sent through the ceramic. The specifications of the amplifier are determined by the piezo characteristics and signal generator. At an excitation amplitude of 50 nm, the piezo expands and contracts over 100 nm. The required operating voltage is computed by:

\[
\Delta L = \pm Ed_{33}L_0,
\]

Table 2.1: Specifications of PZT PIC 181 (from [Phi01]). The piezoelectric and mechanical properties of interest are in the ‘33’ direction, perpendicular to the plate area.
2.2 Experimental setup design

100. To compute the current consumption, the capacitance $C$ [F] is an important parameter:

$$C \approx n\epsilon_{33}\frac{A}{d_s},$$  \hspace{1cm} (2.2)

where $n$ is the number of layers, $\epsilon_{33}$ is the permittivity [As/Vm], $A$ is the electrode surface area and $d_s$ is the layer thickness. The permittivity is often expressed as a multiple of the electric constant $\epsilon_0 = 8.854 \cdot 10^{-12}$ C²/N m². For the single crystal piezo plate with size 10x10x0.5mm, the capacitance is $C = 2.12$ nF. The maximum operating current is

$$i_{max} \approx f\pi CU_{p-p} = 250\text{mA},$$  \hspace{1cm} (2.3)

for sinusoidal operation, with frequency $f$ and peak-to-peak drive voltage $U_{p-p}$.

From (2.2) it is clear that stacked piezo’s have a much higher capacitance; $n$ increases and $d_s$ decreases. This introduces some problems concerning the operation of the actuator, because an amplifier has to supply a high frequency signal to the piezo, at the appropriate voltage amplitude. The operating voltage, though, is higher for a single crystal piezo element. To compare, for 10 stacked piezo elements (100 µm thick), also capable of a displacement of 50nm, only $\pm 19$V is required. The capacitance is 106nF, giving a current consumption of 1.2A at 100kHz. High frequency amplifiers cannot handle these high capacitance loads, while a high operating voltage is less problematic.

The amplifier circuitry was designed by Michael Hopper and built by the Central Technical Service of the TU/e. The electrical circuit layout is found in Appendix D. The amplifier operates between 0-400V and can handle a current consumption up to 600 mA. Preliminary testing shows that a drive frequency of up to 100 kHz with an amplitude of 200V can be reached, using an input signal with an amplitude of 2V, so the amplifier is designed well within specifications. More on the operation of the piezo will be discussed in Section 2.3.

Heat dissipation

During operation, the piezo generates heat, proportional to its dielectric loss factor, $\tan \delta$:

$$P \approx \frac{\pi}{4} \tan \delta fCU_{p-p}^2.$$  \hspace{1cm} (2.4)

The loss factor for PIC181 PZT is 3%, giving a thermal power of $P \approx 0.7$W. To dissipate that heat, the piezo is placed on top of an aluminum heatsink, so overheating is avoided.

2.2.3 Measurement stage

The cantilever is excited at the fixed end within a range of frequencies and the dynamic response is recorded by measuring the vibration at the free end of the cantilever. The response of the tip depends on the cantilever’s mechanical and dynamical properties and its geometry. To measure the deflection, a contactless measurement principle is required. An all-in-one solution for measuring the deflection of the cantilever is a Laser Detector Grating Unit (LDGU).
An LDGU consists of a laser source, photodiodes and a grating. Additionally, mirrors and lenses are placed to guide and focus the laser light [Pri02]. In Figure 2.6, a schematic representation of the general working principle is shown. The laser beam is split by a beam splitter, guided through the grating and focused on the surface. When exactly in focus, the reflected light follows the exact same path back through the lens, but at the grating it is refracted and directed to the diodes. The diodes are placed such that when the light beams are in focus, the reflected beams are also focused on the diodes, resulting in a small light spot and a low output current of the diodes. When the object moves towards or away from the focal point of the laser beam, the light spots grow and move - due to different diffraction through the grating - and the output currents differ. The analog signal of the diode couples is subtracted, which is a measure of the displacement of the object.

Figure 2.6: Measurement principle of a Laser Detector Grating Unit (LDGU) [Pri02].

The LDGU, which is used in this setup, comes from a Philips VAM1202/12 CD Mechanism. The function of this LDGU in CD players is to focus the laser spot on the CD surface and to follow the data track, using voice coils, actuating the projector lens. Additional information about the LDGU is found in [Phi00]. Five diodes are placed as in Figure 2.7. The diodes $D_2$ and $D_3$ are used for measuring z-movement. The so-called 'satellite diodes', $D_1$ and $D_5$, are used for detecting radial offset of the data track on the CD, and are of no interest. $D_4$ is a central diode, giving an indication of the overall intensity of the reflected light. The voice coil actuators are not necessary and can even be a disturbing factor, because the lens has two degrees of freedom in the plane, perpendicular to the cantilever oscillation. The actuators are therefore disabled and the leaf-spring guidance for the lens is glued to the frame of the LDGU.

The diode signal $D_2 - D_3$ gives information about the displacement of the surface. On approaching the surface, the signal $D_2 - D_3$ qualitatively evolves as shown in the graph in Figure 2.7, called the S-curve. Starting out of focus, the laser spot moves towards the diodes when approaching the surface. At a certain distance, the laser spot moves over diode $D_2$, so
2.2 Experimental setup design

$D_2 - D_3$ is positive. Further approaching the surface, both $D_2$ and $D_3$ are illuminated equally, so $D_2 - D_3 = 0$. Moving along, $D_3$ receives more light, so the signal becomes negative. If the surface is too close, the laser spot is not illuminating the diode anymore, and the signal vanishes again. The output currents can be related to the displacement of the surface. This LDGU can detect amplitudes up to 12 $\mu$m.

**Data acquisition**

A flat cable connects the LDGU to a power source and the diode signals can also be acquired. The output currents (in the order of $\mu$A) are amplified and converted to a voltage by an instrumentation amplifier. The amplifier circuit can be found in Appendix E. To shield the measurement stage and amplifier from surrounding electromagnetic noise, a brass Faraday cage is built. This cage also protects the equipment and makes positioning and handling of the measurement stage easier. BNC connectors and coax cables shield the diode signals, directed to the data acquisition hardware. A picture of the LDGU and the final measurement head is shown in Figure 2.8.

To acquire the diode signals and to excite the piezo at high frequency, high speed Data Acquisition (DAQ) hardware is required. The resonance frequency of the cantilevers is expected in a range up to 100 kHz. The Nyquist Shannon sampling theorem states that for a frequency to be reconstructible, the sampling rate of the signal must be at least two times higher than this frequency. A rule of thumb is to use five times the frequency, to avoid aliasing and to be working well within the limits of the hardware.

The NI-USB 6251 from National Instruments is chosen, for its easy connection and high sample rate (1.25 MHz). When two signals are acquired simultaneously, the sample rate is roughly half the maximum rate. BNC connectors are made in the casing of the NI-USB 6251, because the standard supplied flat cable introduced unacceptable noise levels in the
measurement. National Instruments LabView is used for data acquisition, filtering and signal generation.

To align the laser spot on the cantilever, the excitation stage is mounted on a manual XYZ translation stage and \( \theta \phi \) tilt stage. Manual positioning is sufficient to align the laser properly. The LDGU diode signal \( D_4 \) is used to align the cantilever. A maximum signal from \( D_4 \) implies that the cantilever surface is reflecting optimal, so the surface is placed perpendicular to the LDGU. The measurement head is fixed on a stiff frame and the complete setup is placed on a vibration isolated optical table.

2.2.4 Heating

The temperature of the cantilevers is controlled by connecting the NiTi film to a current source. The current source is operated by sending commands through an RS232 port. Numerical values are sent as strings, and the corresponding constant current is offered to the film. Using this heating method (Joule heating), the film only needs to be connected to the current source with two wires. This makes it an easy and compact way of heating the film, compared to using a heating/cooling plate or temperature controlled environment, where the complete excitation stage would be placed in. The connecting wires may influence the dynamic excitation, but not in the frequency range of interest.

Wirebonding was first considered for connecting wires to the film. Several attempts to bond both gold and aluminum wires did not result in good connections. Possibly, the native oxide layer on the surface of the film prevents good bonding. Another reason can be that the material damps the ultrasonic vibrations, used to bond the wire to the surface. Finding the good configuration for wirebonding is an elaborate task. Alternatively, wire contacts are glued on the film, using a conductive epoxy glue (Amicon C805-1#500).

The current source is calibrated, to check the operation through the RS232 port. The resulting calibration curve is shown in Figure 2.9. The output saturates for large resistance values, because the supply voltage is limited to 12V. Resistance measurements on the film will be
discussed in Chapter \[4\].

![Figure 2.9: Calibration of the current source. The input ticks are strings, send to the current controller by an RS232 port.](image)

### 2.2.5 Assembly

For easy (re)placement and safe storage of the fragile cantilevers, the steel supporting plate is placed on a kinematic mount. The mount consists of an aluminum base, three sapphire spheres and two Neodymium magnets (see Figure 2.10(a)) and it is inspired on mounts, generally used in Atomic Force Microscopes. The supporting plate has a hole and a slot, so the plate can be positioned statically determined, while kept in place by the two magnets. Removing and replacing the cantilevers is easy, while the position of the chip is maintained. A finite element analysis of the resonance modes of the plate shows that the first two modes at $\approx 21$ kHz and $\approx 28$ kHz are the wobbling of the freestanding corners of the plate. The third mode at $\approx 56$ kHz, can be seen as tilting of the plate (see Figure 2.10(b)). In Section 2.3 it is discussed what influence these vibrations have on the frequency response measurements of the cantilevers.

The piezoelectric element is glued between the heatsink and kinematic mount using HBM X60 two component adhesive, also used for fixing strain gauges. The connecting wires are soldered with silver solder and isolated using silicone glue. Figure 2.11 shows a picture of the final excitation stage.
Experimental setup

(a) Kinematic mount design and the cantilever chip with supporting plate.

(b) COMSOL finite element eigenfrequency analysis. Here the third resonance frequency of the steel plate at $\approx 56kHz$ is shown.

Figure 2.10: Kinematic mount design and analysis.

The dynamic peak force during excitation can be determined, if the oscillating mass is known. The effective oscillating mass is one third of the piezo mass and the additional mass of the kinematic mount, the cantilever holder and the cantilever chip. The total mass of this assembly is $1.09g$. With a total length change of $\Delta L = 100nm$ at $f = 100kHz$, the peak force is

$$F_{dyn} = \pm 4\pi^2 m_{eff} \left(\frac{\Delta L}{2}\right) f^2$$  

$$= 21.5N.$$
2.3 Characterization of the experimental setup

The required stiffness of the construction then is

\[ k_T = \frac{F_{dyn}}{\Delta L/2} = 430 \cdot 10^6 \text{N/m}. \]  

(2.7)

The stiffness of the piezo in the excitation direction is

\[ k_{33} = \frac{C_{33}^D A}{L} = 33.2 \cdot 10^{10} \text{N/m}, \]  

(2.8)

which is much higher than the required stiffness, so the piezo element does not need prestraining and will hold the dynamic forces for our purposes.

2.3 Characterization of the experimental setup

To isolate the cantilever’s frequency response from the piezo excitation, the influence of the peripheral equipment is investigated.

To investigate the operation of the piezo amplifier, the Frequency Response Function (FRF) of the amplifier output signal is compared to the FRF of the amplifier input signal. The power supply and piezo amplifier will distort the input signal, generated by the software. In Figure 2.12 it can be seen that the amplifier contains a highpass filter. Excitation frequencies in the range of 10 kHz to 100 kHz are amplified best, according to the specifications. A maximum amplification level of ±80 is achieved in this frequency range.

![Figure 2.12: Frequency Response Function of the amplifier output to the signal generator input.](image)

If the piezo element is connected to the amplifier, the capacitive load influences the operation of the amplifier. This is shown in Figure 2.13. The piezo load only affects the FRF of the amplifier output above 100 kHz. This implies that for our purposes, the amplifier is able to handle the capacitive load of the piezo element.
Experimental setup

Now that the input characteristics of the excitation stage are known, the characteristics of the LDGU are investigated. To investigate crosstalk between the piezo amplifier electronics and the LDGU amplifier, the output signal of the diodes is analyzed, while the LDGU is not measuring the piezo excitation. Therefore the LDGU is focused on a fixed plane, while the piezo element is in operation. The FRF of the diode voltage $D_2 - D_3$ should not pick up any excitation signal, except the background noise and possibly some electronic disturbances in the peripheral equipment. Furthermore, vibrations of the excitation stage to the measurement stage through the mounting table is assessed. In Figure 2.14 the result of this FRF measurement is shown. A sharp peak can be distinguished at 24.5 kHz. This frequency and its second and third harmonic are also appearing in the FRF of the input voltage in Figure 2.13. It is identified as a disturbance, due to the electronics of the piezo amplifier. The diode signals are contaminated by it, because the DAQ picks up this signal through the connecting cables.

From Figure 2.14 we also conclude that the background noise is low; only at very high frequencies (> 200kHz) some peaks are observed, probably due to the LDGU amplifier electronics or analog to digital conversion (ADC) of the DAQ. A lowpass filter with a cutoff frequency of 100kHz is added to reduce the influence of these high frequency disturbances. From the figure it can be concluded that no mechanical transfer of vibrations through the mounting table occurs.

Figure 2.13: Frequency Response Function of the amplifier output with or without piezo load. Above 100 kHz, the amplitude of the response without load decreases, while the response with piezo load peaks.
2.3 Characterization of the experimental setup

2.3.1 Test measurements

To check whether the operation of the setup is according to the requirements, a measurement is done with an uncoated Silicon micro cantilever, cantilever ‘E’ (see Appendix C). The lateral dimensions are measured with the Sensofar optical profiler and the thickness is first assumed to be as specified in Appendix C, giving \( l \times w \times h = 348x35x2 \ \mu m \). The Young’s modulus is depending on the crystal orientation of the Silicon and the appropriate stiffness matrix has to be used. Assuming uniform thickness, the first resonance frequency is calculated with COMSOL Multiphysics. Given the geometrical and material parameters, the first bending mode is found at 20.9 kHz.

To generate a frequency response function of the cantilever, a uniform white noise signal with an amplitude of 0.1 V is supplied to the piezo amplifier. The sample rate for signal generation and acquisition is 700 kHz and the number of samples in one measurement interval is \( 10 \cdot 10^3 \). The FRF output is averaged over 200 measurements, with a Hanning window to reduce the influence of signal leakage. The result is shown in Figure 2.15. Two resonance modes at 16.0 kHz and 16.9 kHz can be distinguished. One mode is most likely the first bending mode. The appearance of the second peak will be discussed in Chapter 4.

The resonance mode of the steel mounting plate at 56 kHz is also clearly distinguished. The electronic disturbance at 24.5 kHz does not reach the order of amplitude of the cantilever resonance. To obtain the transfer function \( H_{\text{cant}} \) of the cantilever we use

\[
H_{\text{cant}} = \frac{\hat{y}_{\text{tip}}}{\hat{y}_{\text{chip}}}, \tag{2.9}
\]

where \( \hat{y}_{\text{tip}} \) is the amplitude of the frequency response at the cantilever tip and \( \hat{y}_{\text{chip}} \) is the amplitude of the frequency response at the clamped end of the cantilever, which in fact is the cantilever chip. The result for the uncoated cantilever E is shown in Figure 2.16.
plate resonance has nearly vanished and only the two resonance modes remain.

Figure 2.15: Frequency Response Function of the uncoated Silicon cantilever E with length $l=348 \mu m$.

The resonance frequency differs significantly - almost 25% - from the calculations. This can be addressed to uncertainty in the thickness of the cantilever. In [Mik07] the resonance frequency of the cantilever is also given: 21 kHz with 20% uncertainty, due to the geometric variations. The measured resonance frequency does not fall within these specifications.

If the thickness is changed to $1.6 \mu m$, the calculated resonance frequency shifts to 16.7 kHz,
2.3 Characterization of the experimental setup

matching the measurement within 5%. The thickness now does not fall within the specified geometric uncertainty (2±0.3 μm) of the supplier, which is logical, given the big difference in resonance frequency.

Other reasons for the mismatch in resonance frequency can be a non-uniform thickness and elasticity of the fixation of the cantilever with the chip. This will not be discussed here.

Discussion

In this chapter we have discussed the design and calibration of the experimental setup for dynamic measurements. The setup is specialized for high frequency excitation and the measurement stage is capable of detecting small displacements at these high frequencies. Using a current source as a means of heating the film is a simple and flexible solution, but it can only heat the film from room temperature. The thermomechanical characteristics of the samples will show whether the temperature range of the current source is sufficient to observe phase transformations in the material.

Calibration and test measurements show that the setup is working according to the specifications, stated in Section 2.1. In Chapter 4 the setup is used to obtain Frequency Response Functions (FRFs) of the coated cantilevers as function of the current through the film. The FRFs give insight in the dynamical behavior of the cantilevers. The next chapter deals with modeling the thermomechanical and dynamical behavior of the coated cantilevers and modeling the heat transfer through the film. These models are useful to predict and explain the behavior, observed in the measurements in Chapter 4.
Chapter 3

Modeling of the coated cantilever

In Chapter 2, the experimental setup for investigating the thermal-dynamical properties of the cantilevers is discussed. Before conducting experiments with the NiTi coated cantilever samples, knowledge about the thermomechanical behavior is desired. This gives insight in the transformation temperatures of the material and stress variations due to the phase transitions. Using a current source as a means of resistive heating of the NiTi film requires knowledge about the electrothermal properties of the material, as well as heat transfer to the surroundings.

The dynamic behavior of the cantilevers is influenced by the thermomechanical behavior. An analysis of the resonance frequencies and a way of investigating of the damping properties is explained in this chapter.

A general framework of the relation between the processes discussed above, is visualized in Figure 3.1. It must be noted that we do not have the intention of constructing a complete model of the influence of the input current to the dynamics of the cantilevers. Every process is analyzed and the important parameters influencing the thermal, mechanical and dynamical behavior of the cantilever are discussed.

**Figure 3.1:** Block diagram of the model. The current $i$ results in a film temperature $T$. The temperature changes the stress state $\sigma$, resulting in a deflection $\delta(T,t)$. If the cantilever is excited with an harmonic input $\omega$, the deflection in the frequency domain $\delta(T,\omega)$ can be analyzed.
### 3.1 Thermomechanics

An analysis of the thermomechanical behavior of the coated cantilever gives insight into its material properties and transition temperatures. This information is valuable for investigating the dynamical properties of the cantilevers and the possibilities of changing the dynamics by varying the temperature. In the following analysis, we assume that the temperature of the cantilevers is known. In other words, the output of the Joule heating analysis is the input for the thermomechanical model. The output of this model part is the stress in the NiTi film and the accompanying deflection profile of the cantilever.

The coated cantilever experiences two mechanisms, related to temperature variations and introducing stress in the NiTi film and the Silicon substrate. Due to a difference in thermal expansion coefficients the two materials exert a force upon each other, depending on their stiffness and geometrical properties. Furthermore, the crystal reorientation during phase transitions, as discussed in Chapter [1](#), adds stress variations within the NiTi film. Finally, the crystal structure of the material changes during phase transformations, changing the Young’s modulus and thermal expansion coefficient of the NiTi film.

To analyze these processes separately, we divide the total stress in the NiTi film in two parts: the stress $\sigma_b$ due to a mismatch in thermal expansion coefficients, which from now on we call the **bimorphic stress**, and stress variations due to phase transformation $\sigma_t$, called the **transformation stress**.

$$\sigma = \sigma_b + \sigma_t$$  \hspace{1cm} (3.1)

Here $\sigma_b$ also includes the initial stress $\sigma_0$ in the film, due to sputter deposition and annealing.

**Timoshenko’s equation for bimetallic thermostats**

The bimorphic stress is modeled, using Timoshenko’s equation for bimetallic thermostats [Tim25]. In his classical publication, the equilibrium between the forces and bending moments in a bimorph strip gives an expression for the maximal stress in the film. If the thermal expansion of the film is larger than the substrate, e.g. $\alpha_f > \alpha_s$, an increasing temperature results in compressive (negative) stress in the film, as shown in Figure 3.2. The initial stress and curvature can be tensile or compressive, but for this analysis that is not relevant. The following equation is obtained for the curvature $\rho = \frac{1}{R}$ and maximal stress $\sigma_{b,\text{max}}$ in the film.

$$\sigma_{b,\text{max}} = \rho \left( \frac{2}{(h_s + h_f)h_f} (E_s I_s + E_f I_f) + \frac{h_f E_f}{2} \right)$$  \hspace{1cm} (3.2)

$$\rho = \rho_0 + \frac{h_s + h_f}{2} \frac{(\alpha_f - \alpha_s)(T - T_0)}{\frac{1}{E_s I_s} + \frac{1}{E_f I_f} + \frac{1}{E_f h_f w}}$$  \hspace{1cm} (3.3)

where $E_f$, $\alpha_f$ and $h_f$ are the Young’s modulus, thermal expansion coefficient and thickness of the film, respectively. $w$ is the width of the cantilever. For the Silicon substrate, we use the index $s$. The area moment of inertia $I_f$ and $I_s$ are discussed in the next paragraph. An initial curvature $\rho_0$ at $T = T_0$, caused by sputtering and annealing, causes initial stress in the film. The bimorphic stress is at its maximum at the interface between the film and substrate,
3.1 Thermomechanics

where the effect of the thermal mismatch has the largest influence. In further analysis we consider this interface stress, and replace $\sigma_{h,max}$ by $\sigma_b$.

In Timoshenko's approach, the assumption is made that the width of the strip is small, compared to its length. Furthermore, it is assumed that the thermal expansion and inertia remains constant during heating. We extend this analysis to a situation with temperature dependent stiffness and thermal expansion. As can be seen in (3.3), the stress varies proportional to the temperature. Note that if we assume the film thickness is much less than the substrate thickness, (3.3) reduces to Stoney’s equation for the stress in thin films.

Area moment of inertia

Uncoated Silicon cantilevers are assumed to have a rectangular cross section. Neglecting the tapered sides, the area moment of inertia $I_y$ of the rectangular cross section with width $w$ and height $h_s$ around its neutral axis $y$ is

$$I_y = \frac{wh_s^3}{12}. \quad (3.4)$$

Residual stress in the NiTi coated cantilever results in bending along the length of the beam, but also along the width of the beam. This is shown schematically in Figure 3.3. Here we assume that the cross section can be considered as a sector of a hollow circle and we use polar coordinates for convenience. If the radius of curvature $R$ and the initial width $w$ of the beam are known, we compute the angle as $\alpha = \frac{2R}{w}$ [rad]. The area moments of inertia of the film and substrate, for bending around the $y$-axis, are computed as follows.

$$I_f = I_{yf} + (y_f - y_c)^2 A_f \quad (3.5)$$

$$I_s = I_{ys} + (y_s - y_c)^2 A_s, \quad (3.6)$$

where $I_{yf}$ is the moment of inertia of the film around its centroid $y_f$, measured from the center of curvature $O$, as indicated in Figure 3.3. The same holds for the substrate, with index $s$. The inertia $I_f$ and $I_s$ around the centroid $y_c$ of the total cross section is computed using Steiner’s law. Derivations of the expressions for the area moment of inertia are found

Figure 3.2: Bimorphic bending due to heating along the length of a beam element. $R$ is the radius of curvature of the cantilever.
in Appendix F. The temperature dependence of the moment of inertia lies in a change of curvature, due to the thermal mismatch and phase transitions.

![Diagram of a cantilever cross section](image)

**Figure 3.3:** Dimensions of the NiTi coated cantilever cross section.

### Material properties

Due to the phase transitions, the material properties of the NiTi film change significantly. If we consider two-step transformation, the Young’s modulus of NiTi can take the values $E_m$, $E_a$ and $E_r$ for Martensite, Austenite and R-phase, respectively. The thermal expansion coefficients are $\alpha_m$, $\alpha_a$ and $\alpha_r$. The material properties are constant when no transformation takes place and we assume that they vary linearly with the temperature during a phase transformation. In fact, this means that the phase fraction is assumed to vary linearly with the temperature, so

$$\xi_m = \frac{A_f - T}{A_f - A_s}, \quad \text{for } A_s < T < A_f \quad \text{and } \dot{T} > 0 \quad (3.7)$$

$$\xi_m = \frac{M_s - T}{M_s - M_f}, \quad \text{for } M_f < T < M_s \quad \text{and } \dot{T} < 0 \quad (3.8)$$

$$\xi_r = \frac{R_s - T}{R_s - R_f}, \quad \text{for } R_f < T < R_s \quad \text{and } \dot{T} < 0. \quad (3.9)$$

This assumption is only made to estimate the evolution of the material properties. A piecewise linear model can be made to calculate the Young’s modulus at a specific temperature, also depending on the temperature history. The result of such a model is shown in Figure 3.4. It can be seen that the modulus changes at the transition temperatures. The thermal expansion coefficient follows a similar profile.
3.1 Thermomechanics

Figure 3.4: Evolution of the Young’s modulus as function of temperature. The solid line indicates an increasing temperature, the dashed line indicates a decreasing temperature.

Accounting for the changing material parameters, the bimorphic stress evolves as shown in Figure 3.5. Starting from the initial positive stress $\sigma_0$ at $T_0$, the stress in the film (at the interface between the film and substrate) decreases as function of temperature, because the thermal expansion of the film is larger than the substrate.

Figure 3.5: Bimorphic stress $\sigma_b$ due to heating. The solid line indicates an increasing temperature, the dashed line indicates a decreasing temperature.
3.2 Transformation stress – hysteresis modeling

As discussed in Chapter 1, the phase transitions induce a change in the crystal structure of NiTi, leading to stress and deformation. The transformation behavior depends on the composition and annealing treatments, as well as residual stress and pretension/compression in the material. If the stress levels in the material are generally low, the R-phase appears, an intermediate phase between Austenite and Martensite phase transformation. For a NiTi thin film on a substrate, the temperature-stress curve can have several forms. A one-step transformation from Martensite to Austenite is shown in Figure 3.6(a), while in Figure 3.6(b), a two-step transformation can be seen, indicating the presence of the R-phase [Wan07].

![Temperature-stress curve of 910nm NiTi film on various substrates.](image1)

(a) Temperature-stress curve of 910nm NiTi film on various substrates.

![Temperature-stress curve for 910nm NiTi film on Corning glass substrate.](image2)

(b) Temperature-stress curve for 910nm NiTi film on Corning glass substrate.

Figure 3.6: Typical temperature-stress curves of NiTi on various substrates. The temperature is cycled between 20 °C and 120 °C [Wan07].

Before measuring the thermal response of the samples, it is difficult to say which graph resembles the transformation behavior of our samples. We therefore discuss the outcome of thermomechanical experiments in Chapter 4. In this section, a proposal is made for modeling approaches, possibly able to model the transformation stress. Because the transformation is hysteretic, we will investigate several models of hysteresis.

The transformation stress $\sigma_t$, together with the computed bimorphic stress $\sigma_b$ from the former section, give a full overview on the thermomechanical behavior of a NiTi coated cantilever.

3.2.1 Hysteresis models

The word hysteresis is a derivation of the greek word υστέρημας, which literally means to come late or to lag behind. Hysteresis occurs in various physical phenomena, such as structural mechanics, electromagnetism and phase transformations. A system with hysteresis has memory dependent behavior; the output of the system depends on the input and the output history. Furthermore, the system shows different behavior for an increasing or decreasing input. Hysteresis occurs for example in magnetization and demagnetization of soft iron and in mechanical elements like actuators or gears, with play or backlash. From
these varying backgrounds in engineering and science, models of hysteresis arised in various forms.

In general we can subdivide hysteresis models in two classes, physical and geometric models. In physical models, the parameters and their dependence are motivated on the physical processes causing the hysteresis. Geometric models are based on finding equations that represent measured hysteresis curves. The equations have no physical motivation, but simply map the input on the output as good as possible. In the following paragraphs, existing models of hysteresis in shape memory alloys of both classes are reviewed shortly.

**Physical models of hysteresis**

Ikuta et al. [ITH91] have made a start in modeling the behavior of shape memory alloys, using a variable sublayer approach. The phases of the material are modeled separately and the total behavior is modeled as a parallel connection of these sublayers of material models. The sublayers consist of basic models of mechanical play in series with a spring. The model has some physical motivation, but the actual hysteresis is simply modeled as play. Boyd and Lagoudas [BL96] have developed a thermodynamical constitutive model, using the Gibbs free energy and a dissipation potential. Hardening effects are also incorporated. Bekker and Brinson [BB98] have formulated a model, based on the transformation kinetics of the material and basic piecewise continuous functions, describing stress and strain relations. The phase fraction in the material is calculated from transformation kinetics. The model is mainly concerning about computing the phase fraction as function of temperature.

**Mathematical models of hysteresis**

Mathematical models of hysteresis exist in various forms. Some are inspired on physical models, but adapted for other applications. We can distinguish models that are a sum of basic elements - the hysteresis operators - and differential models. Other geometrical models use the so-called *hysteron*, a general formulation of hysteresis, applied to some input-output behavior. In all of these three classes models of shape memory alloys are found.

The Preisach model [Pre35] is based on a summation of relay operators with switching values and weight factors. The physical relevance of summing the relay operators is given as being the phase transition of separate crystals in an SMA or switching dipoles in magnetic hysteresis. Mayergoyz [May91] discusses the model's properties and limitations extensively. Hughes and Wen [HW97] have worked on Preisach modeling of SMA hysteresis and proposed an identification method.

The Duhem model [MNZ93, OB05] includes a large class of differential models with an extensive amount of properties. The models can be rate-dependent or rate-independent, linear or nonlinear. The motivation for this model is found in electromagnetic charging, where the output changes its behavior when the input changes direction. Derivations of the Duhem model are the Bouc-Wen and Madelung model. Dutta et.al. [DGD05, DG05] propose a method to model and control a Shape Memory Alloy wire actuator. The model is a combination of the Duhem model and Ikuta’s variable sublayer model. It describes Joule heating, phase transformation and stress-strain relations and uses a large amount of
nonlinear curve fits to describe the temperature dependent material parameters.

Macki et.al. [MNZ93] give a general overview of a large amount of mathematical models of hysteresis. They distinguish between relay hysteresis and active hysteresis. The difference between these two is that in the case of active hysteresis, trajectories inside the hysteresis region (also referred to as the major loop) are possible. The Duham and Preisach model, as well as some other interesting hysteresis operators are discussed. The 'stop' operator was originally proposed as a model for plasticity-elasticity. An upper and lower boundary - the stops - are connected by monotone functions. The Krasnosel'skii-Pokrovskii hysteron is a generalized 'play' operator. The hysteron can be formulated with any function for the major loop up and down, and horizontal lines describe the minor loops.

Discussion and model requirements

Constructing a physical model of shape memory alloy hysteresis requires knowledge of transformation kinetics and energy balance in crystal structures. The parameters describing these processes are difficult to measure and to interpret. Understanding transformation kinetics and crystal morphology is out the the scope of this project. We therefore focus on geometrical (or mathematical) models of hysteresis. This conclusion is supported by the work of van der Wijst [Wij98]. He discusses 1D and 3D constitutive models, as proposed in [BL96] and [BB98], as well as geometrical and plasticity based models. The conclusion is that constitutive models, based on free energy or transformation kinetics, are very complicated, while still not accurate. Plasticity based models often require measurement of phase fraction, which is difficult and sometimes impossible. He proposes a geometrical model based on constitutive relations, being a tradeoff between accuracy and simplicity.

Requirements of the model are as follows.

- The phase transformation forms a closed loop, when the material is temperature cycled between fully Martensite and fully Austenite phase. This is called the major loop. In full Martensite or Austenite phase, no transformation takes place anymore. This plateau is called the dead zone. When the major loop is not fully cycled, a so-called minor loop appears. A realistic model has to be able to describe these three mechanisms. Furthermore, the appearance of the R-phase should be modeled if necessary.

- From a control point of view, a simple model would be appealing. A differential model, for example, can easily be included in a control loop and frequency response analysis is relatively easy, making it possible to analyze and control the dynamic behavior of the system. A complex model would increase computational effort, possibly adding time-delay to the system. Other models can eventually be included in a control loop as well, for example as a look-up table or piecewise continuous system. This, however, makes control more complicated.

- The hysteresis in shape memory alloys is rate-independent. This implies that the input velocity does not affect the shape or behavior of the output. A physical explanation is found in the fact that the phase transformation occurs almost instantaneous throughout the material and no physical input can reach a higher velocity than the propagation of the transformation.
In the following sections, we will explore the suitability of some mathematical models to describe the phase transformation hysteresis in SMA. Advantages and disadvantages are discussed, as well as simulation results. The models are evaluated on the above requirements and a final decision for modeling the hysteresis in our system is made.

### 3.2.2 Preisach model

The Preisach model [May91, Vis94, HW97] consists of a weighted sum of relay operators (Figure 3.7). The output can have 'up' and 'down' values, 1 and −1. Depending on the direction of the input, the output switches values at \( \alpha \) or \( \beta \). The Preisach model is a weighted sum of a number of operators with different switching values. The general formulation of the model is

\[
y(t) = \Gamma u(t) = \int \int_S \mu_{\alpha\beta} [\gamma_{\alpha\beta} u(t)] d\alpha d\beta,
\]

where \( \mu_{\alpha\beta} \) is the weighting factor, \( \gamma_{\alpha\beta} \) relay state, depending on the values \( \alpha \) and \( \beta \) and \( u(t) \) is the input.

To form a hysteresis curve, a number of switching values and weighting factor is to be determined. \( S \) is the plane, spanned by the individual relay element switching values \( \alpha \) and \( \beta \). The plane \( S \) is also referred to as the Preisach plane and is shown in Figure 3.8. At every coordinate \( (\alpha_i, \beta_i) \), a specific weight factor \( \mu(\alpha_i, \beta_i) \) determines the change in the output values. Two sets \( S^+ \) and \( S^- \) are formulated, containing the relay elements in the up or down state, respectively. The sets are bounded by the saturation values, defining the boundaries of the Preisach plane. For an increasing input value a number of relay elements switches up when passing the threshold values \( \alpha_i \), giving an increase in the output value (Figure 3.8(a)). When the input switches direction and thus decreases, the output value decreases when passing the threshold values \( \beta_i \) (Figure 3.8(b)). If the hysteresis curve is not fully traveled, a minor loop appears. The minor loops can also be visualized in the Preisach plane, seen in Figure 3.8(c). It can be seen that the output, i.e. the number of relay elements in \( S^+ \), depend on the history of output.

Because infinitely many relay elements can be added, the Preisach model is a very versatile model, capable of modeling complex curves, deadzones and minor loops. Identification of the
model requires the availability of measured hysteresis curves and the model can be made as accurate as desired by choosing the number of relay elements. The output is incremental, but the input can be continuous. The model is rate-independent and is able to model minor loops and deadzones. An example of a Preisach model of hysteresis is shown in Figure 3.9. Clearly the switching points are distinguished. Using more relay operators obviously gives a smoother fit.

Figure 3.9: A Preisach hysteresis curve. Clearly the thresholds for positive and negative velocity can be distinguished.
3.2.3 Duhem model

The Duhem model is based on the fact that in the case of hysteresis, the output of a system changes its behavior when the input changes direction. The general formulation of the Duhem model is [Vis94, MNZ93, OB05, DG05]

\[
\dot{x}(t) = f[x(t), u(t)] g(\dot{u}(t)), \quad x(0) = x_0, t \geq 0 \\
y(t) = h[x(t), u(t)].
\]

For modeling rate-independent hysteresis, the following definition is stated [OB05]:

**Definition 1.** The continuous and piecewise \(C^1\) function \(\tau : [0, \infty) \rightarrow [0, \infty)\) is a *positive time scale* if \(\tau(0) = 0\), \(\tau\) is nondecreasing, and \(\lim_{t \to \infty} \tau(t) = \infty\). A function \(x(t)\) is called *rate-independent* if and only if

\[
x(t) = x(\tau(t)), \quad \text{for all} \quad \tau.
\]

The Duhem model can represent rate-dependent and rate-independent hysteresis, depending on the function \(g(\dot{u}(t))\). Oh and Bernstein state that it is rate-independent if the function \(g(\dot{u}(t))\) is *positively homogeneous*, that is, if \(g(hv) = h(g(v))\) for all \(h \geq 0\) and \(v \in \mathbb{R}\). Furthermore, the input-output behavior of the model is hysteretic if \(f[x(t), u(t)]\) is different for an increasing or decreasing input. Such a model for example is

\[
\dot{x}(t) = [-x(t) + u(t)] |\dot{u}(t)|, \quad x(0) = x_0, t \geq 0 \\
y(t) = x(t).
\]

The notion of a rate-dependent hysteresis model is somewhat confusing, since hysteresis is considered as an intrinsic rate-independent process. It can be clarified by the fact that the hysteretic behavior of a system can be rate-independent, while the dynamics are rate-dependent. An example is the dynamic behavior of a mass-spring-damper system with play. The play itself is a rate-independent mechanism, while the dynamic response of the mass-spring-damper system is strongly rate-dependent. The Duhem model is rate-dependent if \(g(\dot{u}(t))\) is not positively homogeneous, for example when \(g(\dot{u}(t)) = \dot{u}^2\). A property closely related to rate-independence is dependence of a function \(f(u(t))\) on the shape of the input \(u(t)\), as discussed in [OB05].

In Figure 3.10 two sets of simulations are shown for the Duhem model. Figure 3.10(b) shows the rate-independent Duhem model, while Figure 3.10(c) is the rate-dependent model. Clearly we see a difference in the output for the rate-dependent system, when the rate of the input or the shape of the input is changed. For the rate-independent model the output is exactly the same for every input.

In Figure 3.10(d) we see that the model can describe minor loops, but does not model dead zones. Simulations show that no combination of system parameters is able to model these dead zones, if the system is formulated as in (3.11). However, for a positively homogeneous \(g(\dot{u}(t))\) we can separate (3.11) into two functions for positive and negative velocity of the input \(\dot{u}(t)\).
Modeling of the coated cantilever

(a) Input time signals for Figure 3.10(b) and 3.10(c).
The input frequency is varied from 0.1 Hz to 10 Hz.
The fourth input signal is a triangular waveform with frequency 1 Hz. The amplitude is 3

(b) Input-output for system with \( g(\dot{u}(t)) = |\dot{u}| \).

(c) Input-output for system with \( g(\dot{u}(t)) = \dot{u}^2 \).

(d) Input-output for system with \( g(\dot{u}(t)) = |\dot{u}| \). The input amplitudes are varied from 4 to 1. The input frequency is 1 Hz.

Figure 3.10: Rate-dependent and rate-independent behavior of the Duhem model.

\[
\ddot{x}(t) = \left\{ \begin{array}{ll}
 f_+ [x(t), u(t)] g (\dot{u}(t)), & \text{for } \dot{u} \geq 0 \\
 f_- [x(t), u(t)] g (\dot{u}(t)), & \text{for } \dot{u} < 0 \\
 \end{array} \right., \quad x(0) = x_0, t \geq 0
\]

\[
y(t) = h [x(t), u(t)].
\]

This formulation is referred to as the Madelung model. This model switches between functions, depending on the sign of \( \dot{u}(t) \). This formulation may be useful for describing more complex forms of hysteresis, such as hysteresis with dead zones.

**Bouc-Wen model**

An application of the Duhem model for hysteresis is found in the Bouc-Wen model, often used for mechanical hysteresis [Sch04]. It uses the Duhem model to describe some sort of restoring force \( z \) in a dynamical system. The restoring force \( z \) is rate-independent, but the dynamics are not. This can be seen as a mass-spring-damper system with nonlinear play.

\[
\ddot{x} + z = f(t)
\]

\[
\ddot{z} = a\dot{x} - b|\dot{x}| \cdot z \cdot |z|^{n-1} - c|\dot{x}|z^n
\]

\[f(t) = A \sin(\omega t).\]
The influence of the model’s parameters \(a, b, c, n\) and input parameters \(A\) and \(\omega\) on the hysteretic behavior is thoroughly analyzed by Schwanen [Sc04]. Because we are only interested in the hysteretic behavior, only the input-output behavior from \(x\) to \(z\) is analyzed here. We thus consider a harmonic input \(x\). The model becomes

\[
\dot{z} = a\dot{x} - b|\dot{x}| \cdot z \cdot |z|^{n-1} - c\dot{x}|z|^n \tag{3.16}
\]

\[
x(t) = A\sin(\omega t).
\]

With parameters \(a = b = c = 1\) and \(n = 2\), the hysteresis curve in Figure 3.11 is obtained for varying input amplitude (input frequency is not relevant, since this model is rate-independent). We can directly point out big differences with the general Duhem model. First of all, the hysteresis loop is following clockwise. Secondly, the minor loops are different. They don’t increase or decrease along the major loop, but follow a separate trajectory. The behavior of the restoring force \(z\) differs much from the phase transformation hysteresis curves, seen in Figure 1.5.

![Figure 3.11: The restoring force \(z\) as function of input \(x\) with amplitude \(A = 4, 3, 2, 1\).](image)

Identification of the parameters can be a complex procedure. In [Sc04] a two-step identification procedure, based on the Levenberg-Marquardt algorithm, is used to find the parameters to fit on measurements on a wire rope spring showing mechanical hysteresis.

Another derivation of the Duhem model, directly mapping the input and output on physical processes, describes magnetization in soft iron as a function of a varying magnetic field [OB05, MNZ93].

\[
\dot{B}(t) = a |\dot{H}(t)| [bH(t) - B(t)] + c\dot{H}(t), \tag{3.17}
\]

where the output \(B(t)\) is the magnetic flux density as a result of the input magnetic field strength \(H(t)\) and \(a, b\) and \(c\) are positive constants. It can be seen that the model only slightly differs from the Bouc-Wen model with \(n = 1\). However, the input-output behavior changes significantly, similar to the general Duhem model (3.13).
3.2.4 Krasnosel’skii-Pokrovskii hysteron

The Krasnosel’skii-Pokrovskii hysteron \[ KP89, MNZ93 \] is a purely geometric model and can be seen as an extension of the model for play, shown in Figure 3.12. The bounds, linear functions in the play model, are replaced by two continuous nondecreasing functions \( \Gamma_1 \) and \( \Gamma_2 \). The two curves are connected by horizontal lines, being the minor loops in the system. Krasnosel’skii and Pokrovskii state a formulation of generalized play as follows.

\[
y(t) = L [t_0, y_0; \Gamma_1, \Gamma_2] u(t),
\]

with input \( u(t) \), output \( y(t) \). \( \Gamma_1 \) and \( \Gamma_2 \) are a function of the input \( u(t) \). For continuous monotone input \( u(t) \), \( t \geq t_0 \), (3.18) is reformulated to

\[
y(t) = \begin{cases} 
\max\{y_0, \Gamma_+[u(t)]\}, & \text{when } u(t) \text{ is nondecreasing} \\
\min\{y_0, \Gamma_-[u(t)]\}, & \text{when } u(t) \text{ is nonincreasing}.
\end{cases}
\]

We observe that the output \( y(t) \) depends on the input signal \( u(t) \) and the history of the output \( y_0 \). This formulation gives opportunities for using complex functions to describe the major hysteresis loop, while a disadvantage is that the minor loops are represented only by horizontal lines. This is not the case for transformation hysteresis in SMA (see Figure 1.5). Dead zones are easily modeled, simply by choosing the appropriate functions \( \Gamma_+ \) and \( \Gamma_- \) (for example a saturating function) and the model is computationally simple. Identification is also very easy, since \( \Gamma_+ \) and \( \Gamma_- \) can be fitted separately.

A simulation with this model is shown in Figure 3.13. Here, the functions \( \Gamma_+ \) and \( \Gamma_- \) are the sigmoidal functions

\[
\Gamma_+ = \left( 1 + e^{-a_1(u-c_1)} \right)^{-1}, \quad \Gamma_- = \left( 1 + e^{-a_2(u-c_2)} \right)^{-1},
\]

with \( u = u(t) \) and for this simulation \( a_1 = a_2 = 5, c_1 = 2 \) and \( c_2 = -2 \). For an input amplitude, large enough to follow the major loop, we get the solid line. If for some initial
value the input amplitude is lower than the saturation value of these functions and a minor loop appears, for example following the dashed line. If the input amplitude is such that the bounds at $\Gamma_+$ and $\Gamma_-$ are not reached at all, the output keeps moving along a horizontal line, for example the dotted line.

![Graph](image_url)

**Figure 3.13:** Input-output behavior of the Krasnosel’skii-Pokrovskii hysteron, using two sigmoidal functions.

**Discussion**

All of the above mentioned models have a certain advantage with respect to each other. The Preisach model is very versatile in modeling complex hysteresis behavior, because each step can be modeled separately. The advantage of such an approach is that irregular behavior, like different minor loops, can be modeled systematically. The disadvantage is that for every modeled relay, a set measurements should validate its properties. If lots of data points are available, this model can be accurate, but still does not predict behavior outside these data points unless fits on the curves are made. Furthermore, ‘filling’ the Preisach plane with the weighting factors and switching values can be time-consuming and computationally demanding. The result is a big matrix with numerical values, which may not be very useful for control purposes.

The Duhem model and its derivations are very flexible models and can be made as complex as necessary, while still relatively easy to use. Being differential models, they are very suitable for control. The model cannot describe dead zones, but minor loops can be represented easily, because one set of parameters fully describes the behavior of the model. Comparing this to the Preisach model, where every trajectory has to be ‘fitted’ to relay switching values and weight factors, the use of the Duhem model in modeling experimental curves and predicting the output for not measured trajectories is much more appealing. However, parameter identification can be very elaborate, especially if the hysteresis is not directly measurable or if the parameters are coupled (in the case of the Bouc-Wen model).
The Krasnosel’skii-Pokrovskii hysteron has an appealing mathematical simplicity and ease of use. It can model the major loop and dead zones without much effort and identification is straightforward. With well chosen functions $\Gamma_1$ and $\Gamma_2$, the model can describe complex behavior and a least squares fit is sufficient to make an accurate representation of a measured hysteresis curve. The disadvantage of this model is that the minor loops are represented poorly. Because the actual measurements determine which model is most suitable to use in our case, we will decide for the best hysteresis model in Chapter 4 where the measured temperature-stress curves are presented.

Now that the thermomechanics are discussed, the influence of these processes on the dynamical behavior of the system can be analyzed. We therefore present basic equations, describing the dynamics of cantilever beams.

### 3.3 Dynamics of cantilever beams

If the thermomechanical properties are investigated, we also have access to the right parameters for investigating the structural dynamics of the system. Having insight in the evolution of the area moment of inertia and the Young’s modulus as function of temperature gives the opportunity to analyze the vibrations of the cantilevers. The resonance frequencies can be computed, as well as the damping properties.

#### Flexural vibrations

The differential equation that describes the free flexural vibrations of a beam is [VZ65]

$$EI_y \frac{\partial^4 z(x,t)}{\partial x^4} + \rho A \frac{\partial^2 z(x,t)}{\partial t^2} = 0,$$

(3.21)

with Young’s modulus $E$, second moment of area $I_y$ around the $y$-axis, density $\rho$ and area $A$. The vertical displacement $z$ is a function of the position $x$ along the length of the beam, and time $t$. The solution of this partial differential equation gives the frequency equation and wave equation of the beam. The derivation is found in [VZ65]. The first resonance frequency of the beam is

$$\omega_f = \frac{\lambda_f^2}{2\pi L^2} \sqrt{\frac{EI}{\rho A}} \text{ [Hz]},$$

(3.22)

with beam length $L$ and where the wave number $\lambda_f = 1.87510407$ is determined by the boundary conditions.

Because the cantilever in our case is coated with NiTi and the temperature changes both the geometry and the material properties of the cantilever, (3.22) is of the form

$$\omega_f = \frac{\lambda_f^2}{2\pi L^2} \sqrt{\frac{E(T)I_y(T)}{\rho A}}.$$

(3.23)

The evolution of the Young’s modulus and moment of inertia as function of temperature is further discussed in Chapter 4.
3.3 Dynamics of cantilever beams

Torsional vibrations

For describing torsional vibrations we find [VZ65]

\[ GK \frac{\partial^2 \theta(x,t)}{\partial x^2} + \rho J_p \frac{\partial^2 \theta(x,t)}{\partial t^2} = 0, \]  

(3.24)

with polar moment of inertia \( J_p \), shear modulus \( G \) and the \textit{torsional stiffness constant} \( K \). The resonance frequency is

\[ \omega_t = \frac{\lambda_t}{2\pi L} \sqrt{\frac{GK}{\rho J_p}}, \]  

(3.25)

The polar moment of inertia is the sum of the moment of inertia around the y-axis and z-axis: \( J_p = I_y + I_z \). The wave number for the first torsional resonance is \( \lambda_t = \pi/2 \). The torsional constant for a sector of a hollow circle is found in [You89]

\[ K = \frac{2}{3} t^3 R \alpha, \]  

(3.26)

where \( t \) is the thickness of the beam, \( R \) the radius of curvature of the cross section and \( \alpha \) the angle between the centerline of the curve and the outer point (see Appendix F). Again, temperature dependence lies in the inertial parameters and in the modulus

\[ \omega_t = \frac{\lambda_t}{2\pi L} \sqrt{\frac{G(T)K(T)}{\rho J_p(T)}}, \]  

(3.27)

With these relations as a basis, Finite Element Analysis of the coated cantilever beams can be done. This is discussed in Chapter 4.

Damping

Structural damping of the cantilever can be investigated both in the time domain and in the frequency domain. In the time domain, the damping ratio can be deduced from the decay of amplitude in an impulse time response. This is shown in Figure 3.14(a). If we assume viscous damping, the amplitude decay \( Y(t) \) of the system is exponential:

\[ Y(t) = Y_0 e^{-\zeta \omega_n t}, \]  

(3.28)

where \( Y_0 \) is the initial amplitude, \( \zeta \) the damping ratio and \( \omega_n \) is the resonance or natural frequency. As stated above, the damping can be investigated only if the an impulse response is available.

In the frequency domain, damping is determined by computing the quality factor \( Q^{-1} \):

\[ Q^{-1} = \frac{f_0}{f_2 - f_1}, \]  

(3.29)

where \( f_0 \) is the resonance frequency and \( f_2 - f_1 \) is the \textit{half power bandwidth}. This is the frequency, at which the power of the oscillation is half the power of the natural frequency oscillation. In Figure 3.14(b), the frequency response of a single mass-spring-damper system
Figure 3.14: Damping in time and frequency domain.

is shown. Here the frequency is plotted against the amplitude. For computing the half power bandwidth, we note that the power is proportional to the square of the amplitude. The half power bandwidth thus is computed with \( f_1 \) and \( f_2 \) at an amplitude of a factor \( 1/\sqrt{2} \) of the amplitude at the resonance frequency. This is also shown in the figure. The relation between the quality factor \( Q^{-1} \) and the damping ratio \( \zeta \) is

\[
\zeta = \frac{1}{2Q^{-1}}.
\]  

(3.30)

To accurately compute the quality factor, the frequency resolution should be as high as possible. This means that the measuring time should be as long as possible.

### 3.4 Heat transfer

As discussed in Chapter 2, heating of the cantilevers is realized by passing a current through the film. Figure 3.15 is a schematic representation of the heat transfer over the thickness of the Silicon chip and NiTi film.

Figure 3.15: Schematic representation of the heat transfer in the Silicon chip and NiTi thin film.
3.4 Heat transfer

The film has an electrical resistance, resulting in an inward heat flow $q_i$ [W]. The film loses heat by conduction $q_{\text{cond}}$ through the film and Silicon substrate and by natural convection $q_{\text{conv}}$ to the surrounding air. Over time, the film stores the heat, given the specific heat $c_f$ of the material, resulting in a heat balance at a steady state temperature. Since heating of the film is our target, we will only consider the specific heat of the film and do not account for stored heat in the Silicon chip. This gives an indication of how fast the film can change its temperature, regarding the heat losses by conduction and convection.

Convective heat transfer is assumed to occur only from the top surface of the film. The temperature on the bottom of the chip is constant at $T = T_0$. This gives the following balance in the heat flow [Jan00]

$$q_i = q_{\text{transient}} + q_{\text{conv}} + q_{\text{cond}}$$

$$i^2 R = \rho_f V_f c_f \frac{dT}{dt} + \bar{h}_f A_f (T - T_\infty) + k_f \frac{A_f}{L_f} (T - T_0) + k_s \frac{A_s}{L_s} (T - T_0), \quad (3.31)$$

with current $i$, resistance $R$, density $\rho$, volume $V$, specific heat $c$, the overall convection coefficient $\bar{h}_c$, thermal conductivity $k$, characteristic length $L$ and area $A$. $f$ and $s$ indicate the film and substrate, respectively. Note that this is the heat transfer in one dimension and that contact resistance between the film and substrate is neglected.

The thermal conductivity of the NiTi film depends on its fraction and ranges between $8.6 - 16$ [W/mK]. For Silicon, the thermal conductivity also depends on temperature. This makes heat transfer analysis complex. However, if we can use the so-called lumped capacitance approach, internal resistance can be neglected and the thermal conductivity of both the film and the chip do not play a significant role in the heat transfer problem. In other words, the elements have a constant temperature throughout the material. The lumped capacitance approach can be used if the Biot number, specifying the ratio between the internal (conductive) and external (convective) resistance, is small:

$$Bi = \frac{\bar{h}_c L_{cw}}{k} \leq 0.1, \quad (3.32)$$

where $\bar{h}_c$ [W/m$^2$K] is the overall convection coefficient, $k$ [W/mK] is the conduction coefficient of the film or substrate, $L_{cw} = \frac{V}{A}$ [m] is the characteristic length (volume to surface ratio) of the film or substrate. For the time-being we neglect the cantilever dimensions, since they are small compared to the chip, so $A_s = A_f = A$. However, intuitively we can guess that the convective heat transfer may differ a lot from the chip surface, because it is a freestanding structure. The real temperature of the cantilever is expected to be lower than the film temperature. Estimating this temperature is discussed in Chapter 4. The convection coefficient $h_c$ will be estimated in the following paragraph.

**Convection coefficient**

Various correlations exist, estimating the convection coefficient for a horizontal surface in air. The Rayleigh dimensionless number, specifying the ratio between Buoyant forces and viscosity and diffusivity of a flowing medium, determines which correlation is most accurate. The Rayleigh number depends on the film temperature $T_w$, which in our case ranges from 300 K to 400 K.
\[ Ra_{L_{ca}} = \frac{g \beta}{\nu \alpha} (T_w - T_{\infty}) L_{ca}^3, \]  
(3.33)

where \( L_{ca} \) is the surface to perimeter ratio, \( g = 9.81 \text{ m/s}^2 \) is the gravity constant, \( \beta = \frac{1}{T_w} \) (for gases), \( \nu \) is the kinematic viscosity and \( \alpha \) is the thermal diffusivity of the surrounding air at \( T_a = \frac{1}{2} (T_{\infty} + T_w) \). The properties of air at specific temperatures are found in [Jan00]. Considering the dimensions of the coated Silicon chip and the properties of air at the appropriate temperatures, the Rayleigh dimensionless number varies between 0.003 and 0.06. For these low values, the following correlation is found for the Nusselt number [VDI02], giving the ratio between convection and conduction in the air.

\[ Nu = 0.766 \left[ Ra \cdot f_2(Pr) \right]^{1/5}, \quad \text{for} \quad Ra \cdot f_2(Pr) \leq 7 \cdot 10^4, \]  
(3.34)

and \( Pr = \frac{\nu}{\alpha}, \quad f_2(Pr) = \left[ 1 + \left( \frac{0.322}{Pr} \right)^{11/20} \right]^{-20/11} \),

for laminar flow. Here no lower bound is given for the Rayleigh number, so it can be used for any value below \( 7 \cdot 10^4 \). The Nusselt number \( \bar{Nu}_{L_{ca}} \) is defined as

\[ \bar{Nu}_{L_{ca}} = \frac{\bar{h}_c L_{ca}}{k_f}, \]  
(3.35)

where \( k_f \) is the conductivity of air. It must be equal to (3.34), so the overall convection coefficient can be computed from these two equations. This gives \( h \approx 10 - 20 \text{ W/m}^2\text{K} \) for \( T = 300 - 400 \text{ K} \). To check if the lumped capacitance approach applies to the problem (3.31), we need and expression for the overall conduction coefficient of the film and substrate. However, if we compute the Biot number both for Silicon and NiTi separately, we can conclude that \( Bi << 0.01 \) for both materials and for all relevant temperatures. We can thus neglect the internal resistance of the film and chip. This reduces (3.31) to a heat balance as function of time and input current

\[ i^2 R = \rho_f V_{fcf} \frac{dT}{dt} + \bar{h}_c A (T - T_{\infty}). \]  
(3.36)

This differential equation has a solution of the form

\[ T = \frac{B}{D} + Ce^{-Dt}, \quad \text{with} \quad B = \frac{i^2 R + \bar{h}_c A T_{\infty}}{\rho_f V_{fcf}}, \quad D = \frac{\bar{h}_c A}{\rho_f V_{fcf}}, \]  
(3.37)

and integration constant \( C \). Adding a boundary condition \( T(t = 0) = T_0 \) gives \( C = T_0 - \frac{B}{D} \).

The thermal time constant \( \tau = \frac{1}{D} = \frac{\rho_f V_{fcf}}{\bar{h}_c A} \) determines how fast the steady state temperature is reached, giving an indication of the maximum temperature 'switching' frequency

\[ f_{max} = \frac{1}{2\pi \tau}. \]  
(3.38)

Assuming constant material properties, as listed in Table 3.1 and a maximum convective heat transfer (so maximum cooling), the upper bound for the switching frequency is 1 Hz. This is low, if the cantilever’s temperature is to be actively controlled. However, we must keep in mind that we have now considered heating the complete NiTi film. If the film is heated locally, near the cantilever, higher switching rate may be reached.
3.4 Heat transfer

### Table 3.1: Heat transfer parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\rho_f)</td>
<td>6450 kg/m³</td>
</tr>
<tr>
<td>(c_f)</td>
<td>460 J/kg/K</td>
</tr>
<tr>
<td>(V_f)</td>
<td>3.4x1.6x0.001 mm³</td>
</tr>
<tr>
<td>(A)</td>
<td>3.4x1.6 mm²</td>
</tr>
<tr>
<td>(h_c)</td>
<td>20 W/m/K</td>
</tr>
</tbody>
</table>

**Resistance of the film**

As seen in (3.31), the resistance of the layer has to be known for computing the inward heat flux and the resulting temperature. The electrical resistance of the NiTi film is computed by

\[
R = \frac{L}{Wt\rho},
\]

(3.39)

with length \(L\), width \(W\) and thickness \(t\). The resistivity \(\rho\) depends on the fraction of Martensite in the film. Using the variable sublayer model [ITH91], the following relation between the resistivity, temperature and A-M fraction is obtained:

\[
\frac{1}{R} = \frac{1 - \chi_m}{R_A} + \frac{\chi_m}{R_M}, \quad \text{with} \quad R_A = \frac{L}{Wt\rho_A(T)}, \quad R_M = \frac{L}{Wt\rho_M(T)}
\]

(3.40)

\[
\frac{1}{R} = \frac{Wt}{L} \left[ \frac{1 - \chi_m}{\rho_A(T)} + \frac{\chi_m}{\rho_M(T)} \right],
\]

(3.41)

where \(R_A\) and \(R_M\) are the resistance of pure Austenite and Martensite, respectively and \(\chi_m\) is the Martensite fraction. The resistivity \(\rho_A\) and \(\rho_M\) are nonlinear functions of temperature and can be measured independently when the material is in full Austenite and Martensite phase. A disadvantage of this approach is that the Martensite fraction \(\chi_m\) has to be known, while it is hard to measure. We will discuss this in Chapter 4.

Inserting (3.41) into (3.36) gives an expression of the temperature as function of the input current \(i\) and martensite fraction \(\chi_m\).

**Discussion**

Note that in this analysis, a lot of assumptions are made. We assume that the inward heat flow directly heats the complete chip and film. The quality of the glued contacts will greatly influence the heat transfer in the film and will introduce local 'hotspots', where the contacts are attached to the film. Furthermore, the convection coefficient is an approximation and also depends on forced convection by air flows past the chip. As noted above, the cantilever experiences other convective heat losses than the chip, also severely influencing its final temperature. We therefore do not expect that the above heat transfer analysis will be a good estimation of real the cantilever temperature as a result of Joule heating. In Chapter 4 it is explained how the current to temperature relation of the cantilevers is measured indirectly.
Chapter 4

Experiments and simulations

In this chapter, measurements are shown and analyzed. The measured thermomechanics and dynamics are evaluated and a suitable modeling approach, inspired on the models discussed in Chapter 3, is chosen. First the thermomechanical behavior of the coated cantilevers is investigated by thermal cycling experiments. With the aid of experimental results, the thermomechanical model from Chapter 3 is completed and a choice for modeling the phase transformation hysteresis is made. Secondly, Joule heating of the film by the current source is discussed and experiments will be compared with the heat transfer model. This chapter is concluded with the analysis of the temperature dependent dynamic behavior of the coated cantilevers. The computed resonance frequencies and damping properties of the system are analyzed and compared with the measurements.

We used the NiTi coated cantilever chip as shown in Figure 4.1. Throughout this chapter we will name the cantilevers according to the specifications in [Mik07]. It can be seen in the figure that the cantilevers are bent, considering the change in reflected light and the sharpness of the cantilevers in the picture.

Figure 4.1: Optical microscope picture of the NiTi coated cantilever chip. The cantilevers on the right are named $D$, $E$ and $F$ and have a length of 300µm, 350µm and 250µm, respectively.
4.1 Thermomechanics

To investigate the transformation behavior of the NiTi coated cantilevers the sample is thermally cycled, while the deflection profile of the cantilever is measured. This can be related to the curvature of the beam, which in its turn is a measure for the stress in the film. The temperature-stress curve gives information about transition temperatures and material properties.

The setup for thermally cycling the sample consists of a Linkam heating and cooling stage and a confocal optical microscope, the Sensofar PLµ 2300 Optical Imaging Profiler. The measurement equipment is available at the Multi-scale laboratory of the Department of Mechanical Engineering at the TU/e. The heating stage is capable of controlling the temperature of the sample between -190°C and 150°C, using liquid nitrogen cooling and resistive heating. Temperature steps of 5°C are made. The N₂ atmosphere prevents condensation of water in the air during cooling. The confocal microscope measures the profile of the cantilever at a stable temperature. The sample is placed on a plate in the cooling stage, of which the temperature can be controlled with an accuracy 0.1°C.

The temperature of the cantilever, though, cannot be measured directly. Due to conduction and convection this temperature will differ from the measured temperature. A simulation with COMSOL, including conductive heat transfer from the steel plate through the epoxy and to the SMA film and cantilever, as well as convection of each element with the appropriate convective heat transfer coefficients (see Section 3.4), gives the following relation between the surface temperature \( T_{surf} \) and the cantilever temperature \( T_{cant} \). It can be seen in Figure 4.2, that for low temperatures the cantilever temperature is higher than the surface temperature, while at higher temperatures it is lower. The biggest difference is 7°C. We correct for this difference in the measured temperature-stress curves discussed later in this chapter, using the following linear fitted relation:

\[
T_{cant} = 0.96 T_{surf} + 1, \quad [^\circ C].
\]

\( T_{surf} \) and \( T_{cant} \) are plotted in Figure 4.2.

Figure 4.2: Simulated temperature difference between the heat source surface and the cantilever. Heat transfer simulations are done with temperature steps of 5 °C.
An example of a profile measurement is shown in Figure 4.3. This is the profile of cantilever E (L = 350 µm) at T = 23.5°C. The cantilever connects to the chip at X = 200µm and bends upwards, implying a tensile stress is acting on the film.

If we record the end deflection of cantilever E, while cycling the temperature between -100°C and 140°C, Figure 4.4 is obtained, after correcting the estimated temperature offset with (4.1). The response of the cantilever on the thermal cycle is as follows. First the sample is cooled down from room temperature, $T_s = 23.5°C$, to -100°C. Below $M_f = -56.6°C$, the film is fully in Martensite phase. Increasing the temperature again results in downward bending of the cantilever, purely because of the bimorph effect. At $A_s = 1°C$, the phase transformation to Austenite starts. During the phase transformation, the cantilever bends up again, until at $A_f = 63.4°C$ the Austenite stress level is fully recovered. Further increasing the temperature again results in a pure bimorph effect. The slope of this part of the graph differs from the slope in Martensite state, because both the thermal expansion coefficient and the Young’s modulus have changed under phase transformation.

If the temperature is decreased, a backward phase transition takes place. During cooling, an intermediate phase appears, the R-phase. Between $R_s = 58.6°C$ and $R_f = 49.0°C$ a small hysteresis curve is seen, being the Austenite to R-phase transition. After this transformation the material is in R-phase, until at $M_s = -3.8°C$ Martensite transformation starts, resulting in a stress relief and thus downward bending. Again, below $M_f$ the material is fully in Martensite phase.

To relate the deflection of the cantilever to stress in the NiTi film, the curvature $\rho$ is determined. Therefore a second order least squares fit is made on the curved cantilever. This curve is shown in Figure 4.3 with a slight offset, for visibility. From this fitted profile, the curvature is computed by

$$\rho = \frac{\partial^2 z}{\partial x^2} \left[ 1 + \left( \frac{\partial z}{\partial x} \right)^2 \right]^{-3/2}.$$

(4.2)
The curvature is related to the film stress according to Timoshenko’s equation (in the interface between the film and substrate)

\[ \sigma_{b,\text{max}} = \rho \left( \frac{2}{(h_s + h_f)h_f} (E_s I_s + E_f I_f) + \frac{h_f E_f}{2} \right). \]  

(4.3)

As discussed in Section 3.1, the material properties evolve during the temperature cycle, as well as the moment of inertia. The transformation temperatures determine the present properties. Taking into account the changing material properties of the film and inertia of the beam, the temperature-stress curve is as shown in Figure 4.5. We use the transformation temperatures, obtained from Figure 4.4 and selected material and geometric properties, all listed in Table 4.1.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( E_m )</td>
<td>30 GPa</td>
</tr>
<tr>
<td>( E_a )</td>
<td>80 GPa</td>
</tr>
<tr>
<td>( E_r )</td>
<td>70 GPa</td>
</tr>
<tr>
<td>( E_s )</td>
<td>130 GPa</td>
</tr>
<tr>
<td>( \alpha_m )</td>
<td>6.6 µm/m/K</td>
</tr>
<tr>
<td>( \alpha_a )</td>
<td>11 µm/m/K</td>
</tr>
<tr>
<td>( \alpha_r )</td>
<td>7 µm/m/K</td>
</tr>
<tr>
<td>( \alpha_s )</td>
<td>2.6 µm/m/K</td>
</tr>
<tr>
<td>( L )</td>
<td>348 µm</td>
</tr>
<tr>
<td>( w )</td>
<td>35 µm</td>
</tr>
<tr>
<td>( h_f )</td>
<td>1.2 µm</td>
</tr>
<tr>
<td>( h_s )</td>
<td>1.7 µm</td>
</tr>
<tr>
<td>( T_0 )</td>
<td>-70 °C</td>
</tr>
<tr>
<td>( \sigma_0 )</td>
<td>26 MPa</td>
</tr>
<tr>
<td>( A_s )</td>
<td>1.0 °C</td>
</tr>
<tr>
<td>( A_f )</td>
<td>63.4 °C</td>
</tr>
<tr>
<td>( R_s )</td>
<td>58.6 °C</td>
</tr>
<tr>
<td>( R_f )</td>
<td>49 °C</td>
</tr>
<tr>
<td>( M_s )</td>
<td>-3.8 °C</td>
</tr>
<tr>
<td>( M_f )</td>
<td>-56.6 °C</td>
</tr>
</tbody>
</table>

Table 4.1: Chosen material parameters, geometrical parameters and transformation temperatures of the NiTi coated cantilever.

The graph gives information about various processes in the material. We can check the material properties of fully Martensite phase and fully Austenite phase from the slope of the graphs below \( A_s \) and above \( A_f \), respectively. In these regions no phase transformation takes place. From \( R_f \) down to \( M_s \) the material is in constant R-phase, so the material properties in this phase can also be estimated. In constant phase the bimorphic stress is causing the slope
of the graph, so subtracting the bimorphic stress results in horizontal lines in the remaining transformation stress graph. We therefore compute the curvature and film stress due to the bimorph effect, using (3.3). Because no other stress mechanisms are present in constant phase, the total stress is

\[ \sigma(T) = \sigma_b(T), \quad \sigma(T = T_0) = \sigma_0. \]  

(4.4)

Figure 4.5: Temperature-stress curve for cantilever E.

Figure 4.6: Bimorphic stress in cantilever E. The stress is computed at the interface between the film and substrate. In the figure the start and finish temperatures of phase transformations are indicated.
The initial stress $\sigma_0$ is determined at $T_0 = -70^\circ C$, when the stress in the film is purely due to the thermal mismatch. Figure 4.6 shows the bimorphic temperature-stress curve.

Subtracting the bimorphic stress from the total measured stress results in horizontal lines for constant phase, provided the material parameters are chosen well. We can see this in Figure 4.7, where the remaining stress due to the phase transitions ($\sigma_t = \sigma - \sigma_b$), is plotted against temperature. The horizontal lines in constant phase imply that the material parameters are well chosen. The hysteresis in the R-phase is small and hard to measure, considering the accuracy of the temperature controller and uncertainties in the real temperature of the cantilevers due to heat transfer.

![Figure 4.7: Stress due to phase transformation in cantilever E.](image)

The appearance of the R-phase is explained by the fact that the stress levels in the film are relatively low. The stress 'bump' and the different slope after transformation (between $R_f$ and $M_s$) in Figure 4.5, indicate that indeed a two-step transformation takes place. We also note that the width of the Austenite-Martensite hysteresis is almost $60^\circ C$, which is large for equiatomic NiTi. This observation can be addressed to fact that the phase transformation is both temperature- and stress-induced. Recalling Figure 1.4, we can draw the transformation behavior of the NiTi coated cantilever as shown in Figure 4.8. It can be seen that the region for phase transformation is large, because both temperature and stress are activating the transformation and that the R-phase is present, because the residual stress is low.
4.1 Thermomechanics

![Diagram showing phase transitions and stress-induced phase transformation](image)

**Figure 4.8:** Critical temperature and stress for phase transformation the NiTi coated cantilever. The Clausius-Clapeyron relation is drawn as the solid lines. The dashed line indicated the R-phase transition temperature. Temperature-induced and stress-induced transformation is indicated with the solid line and arrows. The combined transformation is the lowest line.

**Modeling the phase transformation**

In the above section we have discussed the thermomechanical measurement and modeling of the bimorph stress $\sigma_b$. To complete the thermomechanical model we are left with modeling the transformation stress $\sigma_t$ in the NiTi film, which behaves as in Figure 4.7. In Section 3.2 we have discussed some appealing options for modeling hysteresis. Considering the measured hysteresis curve we select the model, which is most suitable for the measured temperature-stress response.

The two-step transformation - from Martensite to R-Phase to Austenite and back - introduces problems in modeling the hysteresis with a differential model. Since temperature-stress behavior of the material is asymmetric, the generalized Duhem model (3.11) is difficult to implement. The Bouc-Wen model (3.16), capable of modeling more complex forms of hysteresis, follows the loop clockwise, while our measured curves are followed anti-clockwise. Furthermore, both models are unable to describe dead zones. A derivation of the Duhem model, the Madelung model of (3.14), allows the formulation of separate functions for $\dot{T} > 0$ and $\dot{T} < 0$. The Preisach model is capable of modeling the complex hysteresis curve, simply by adding as much relay elements as necessary. This model, however, is difficult to implement for control purposes. Another disadvantage is that the minor loops have to be measured, in order to model them correctly.
The Krasnosel’ski-Pokrovskii hysteront is a good choice to model this hysteresis curve. At first sight, this model has some similarities with the Madelung model, though the hysteront is not a differential model. This makes it possible to make a least squares fit simply on the measured hysteresis loop, while the Madelung model requires more a sophisticated identification procedure. Although the minor loops are simply represented by horizontal lines, we still approach the behavior of the material well in accordance with the minor loops, as shown qualitatively in Figure 4.5. Only on approaching the major loop, a significant difference is expected between the measured and modeled curves.

Since the major loop shows saturation behavior in both the A-R transformation and the R-M transformation (the dead zones), it is evident that using saturation functions for $\dot{T} \geq 0$ and $\dot{T} \leq 0$ could give good results. Recalling the formulation of the Krasnosel’ski-Pokrovskii hysteront, we define the transformation stress as

$$\sigma_t(T) = \begin{cases} \max \{\sigma_{t,0}, \Gamma_+(T)\}, & \text{when } T \text{ is nondecreasing} \\ \min \{\sigma_{t,0}, \Gamma_-(T)\}, & \text{when } T \text{ is nonincreasing} \end{cases},$$

where $\sigma_{t,0}$ is the stress history variable and

$$\Gamma_+(T) = S(T, p_1)$$
$$\Gamma_-(T) = S(T, p_2) + S(T, p_3)$$
$$S(T, p_i) = p_{i1} + p_{i2} \left(1 + e^{p_{i3}(T-p_{i4})}\right)^{-1}, \quad i = 1, 2, 3. $$

Here we use one sigmoidal function for a nondecreasing input and the sum of two sigmoidals for a nonincreasing input. The function $S(T, p_i)$ requires four parameters, so the total model has 12 parameters, to be fitted on the measured data. We therefore divide the major loop into three intervals, where the sigmoidal functions are fitted:

$$S(T, p_1), \quad \text{for } \dot{T} > 0$$
$$S(T, p_2), \quad \text{for } \dot{T} < 0 \text{ and } T < R_f$$
$$S(T, p_3), \quad \text{for } \dot{T} < 0 \text{ and } T > M_s.$$

The results of the identification procedure are shown in Figure 4.9. The error stays within 5 MPa, except at the R-phase transitions, where the slope of the graph changes abruptly, and thus the function fails to fit well over the data. A simulation with the model for an input of changing amplitude gives Figure 4.10. Clearly we see the minor loops appear when the amplitude decreases.
Figure 4.9: Results of parameter identification on the transformation stress. In the lower graph, the error between the model and measurement is shown. The arrows indicate the direction of the temperature profile.

Figure 4.10: Simulation of the transformation stress in cantilever E. The arrows indicate the direction of the temperature profile. The numbers indicate a change in the direction, also seen in the temperature profile in the bottom figure.
Discussion

We have formulated a thermomechanical model, fully capable of describing the temperature-stress behavior of the NiTi coated cantilever. The stress is computed at the interface between the film and substrate, where the bimorphic stress $\sigma_b$ is at its maximum. The model is based on physical information for the bimorphic stress, while the transformation stress is a geometrical model.

Now we have fully analyzed the thermomechanical behavior at controlled environment, an analysis of the influence of the thermomechanics on the dynamics of the system can be done. We therefore heat the cantilever not with the $N_2$ heating and cooling stage, but with the current source. The next section discusses measurements with the current source, to obtain relation between the induced current and the resulting temperature of the cantilever.

4.2 Joule heating

In Chapter 2 it is explained how Joule heating of the cantilever chip is realized by attaching two wires to the chip, using conductive glue. A current source supplies a constant current, while the resistance of the film changes under varying temperature. In Chapter 3, a 1D model of the heat transfer and a model of the resistance of the NiTi film is formulated. The heat transfer coefficient is estimated and the resistivity is computed, using the phase fraction.

As discussed in Chapter 2, the wire bonds are made with conductive glue, which is an epoxy with silver particles. A lot of uncertainties arise when measuring the resistance of the film. If the voltage is measured at a certain current input, the resistance of the glue, as well as contact resistance between the glue and the NiTi surface, is measured. Furthermore, the epoxy could have some trapped air in between, resulting in a capacitive effect. Resistance measurements give rise to problems, severely increasing the complexity of the heat transfer problem. The resistance measurements are not reproducible and even fluctuate severely at a constant current, due to air flows and unpredictable behavior of the glue.

Because the thermal response of the cantilever to an induced current cannot be measured directly, an indirect measurement approach has to be followed. This approach is to measure the deflection profile of the cantilever when heating and cooling with a calibrated heating source and to repeat the measurement with the current source. The temperature-stress response for the calibrated heat source is compared with the current source measurement, which should be similar in the same temperature range.

The measured current-stress curves are compared to the temperature-stress curves in Figure 4.11. The slope of the current-stress curve is not linear, since Joule heating is not proportional to the input current. Heating from room temperature, we distinguish the R-phase transition in both graphs, indicating that at around 250 mA, the cantilevers are at a temperature of 60 $^\circ$C. However, repeating the measurements did not give reproducible results, because of the fluctuating resistance of the system. Where therefore can not accurately estimate the relation between the induced current and the cantilever temperature. Wirebonding the contacts would give a better quality bond, but to do so, the surface quality must be better.
4.3 Frequency Response Functions

The experimental setup discussed in Chapter 2 is used for dynamic measurements on the cantilevers. The mounted cantilever chip is excited by the piezo element, which is supplied with a white noise signal. During the measurements, the current through the film is varied to change the temperature. In Figure 4.12 the measured Frequency Response Functions (FRF) of the coated cantilevers (solid line) at room temperature are shown. In the same figure the FRF of the uncoated cantilever (dashed line) is shown.

We see that for every cantilever, two peaks are located close to each other. In every FRF, a small sharp peak is seen at 24.5 kHz. As discussed in Section 2.3 this likely is electronic noise from peripheral equipment. The peak almost coincides with the resonance mode of cantilever D. The first bending modes of the uncoated cantilevers, as specified in [Mik07] (with large uncertainty), are listed in Table 4.1.

The $\sim 1\mu$m NiTi film adds mass and stiffness to the cantilevers, so the resonance frequency...
Table 4.2: Specified resonance frequencies of the cantilevers with different length $L$.

<table>
<thead>
<tr>
<th>cantilever</th>
<th>$L$ [$\mu$m]</th>
<th>$f_0$ [kHz]</th>
</tr>
</thead>
<tbody>
<tr>
<td>D</td>
<td>300</td>
<td>28 ±6</td>
</tr>
<tr>
<td>E</td>
<td>350</td>
<td>21 ±4</td>
</tr>
<tr>
<td>F</td>
<td>250</td>
<td>41 ±8</td>
</tr>
</tbody>
</table>

shifts. Apparently, the increase in the stiffness is larger than the increase in mass, because for all three cantilevers the resonance frequencies increase (recall that $f_0 = \sqrt{k/m}$).

Figure 4.12: Frequency Response Function of the three cantilevers at room temperature. The cantilevers D, E and F have a length of 300$\mu$m, 350$\mu$m and 250$\mu$m. The solid lines are the FRFs of the coated cantilevers. The dashed lines are the FRFs of the uncoated cantilevers.

To obtain the transfer function $H_{\text{cant}}$ of the cantilever, we measure the response of the cantilever tip $\tilde{y}_{\text{tip}}$ and of the fixed end $\tilde{y}_{\text{chip}}$, which is the real excitation signal of the cantilever.
The transfer function is computed as
\[ H_{\text{cant}} = \frac{\dot{y}_{\text{tip}}}{\dot{y}_{\text{chip}}}. \quad (4.12) \]

Let us now look at the behavior of the coated cantilevers as function of the induced current. In Figure 4.13 the FRFs of cantilever E, the longest one, are shown. For clarity we only show several curves. More results are found in Appendix H.

![Figure 4.13: Frequency Response Function of cantilever E (L = 350µm) for varying current.](image)

In Figure 4.13 we see that the resonance frequencies shift as function of the current. For increasing current, both peaks decrease. When the induced current is greater than 180 mA, the right peak jumps to the right, while the left one keeps decreasing. The small shifts at lower currents are explained as follows. For low currents, the material is in constant R-phase, which can be deduced from Figure 4.11. The temperature increase results in bending stress in the cantilever, so the cross section of the cantilever changes, decreasing the overall bending stiffness slightly (recall that in Appendix F, the inertia is a function of the radius of curvature of the cross section). This is also seen in the FRF, where the decrease in stiffness results in a decrease of the resonance frequency. Above 180 mA, the phase transformation takes place, having a twofold influence. First of all, the stress in the film (and thus the curvature) slightly increases again (see Figure 4.11). Secondly, the Young’s modulus changes from 70 GPa to 80 GPa, having a bigger influence on the dynamics. The sudden increase of the Young’s modulus, increases the bending stiffness of the beam, so the flexural resonance frequency increases. Considering this transformation behavior, we conclude that the right peaks in Figure 4.13 are the flexural resonance frequencies. We also note that for both modes, the amplitudes first increase, but for higher currents decrease. After transformation, further increasing the current gives an increase in the amplitude for the left mode, while not much changing the frequency.
Figure 4.14: Frequency Response Function of cantilever F ($L = 250 \mu m$) for varying current.

For cantilever F, we observe a similar process (see Figure 4.14). However, here we see that the left peak decreases, while the right peak slightly increases at low currents. This indicates that the flexural mode is in this case not the right peak, but the left one. Another interesting phenomenon is that the peaks merge after phase transition, almost doubling the amplitude. This is seen in reverse order for cantilever E.

Figure 4.15: Frequency Response Function of cantilever D ($L = 300 \mu m$) for varying current.
4.4 Damping

Cantilever D (Figure 4.15) shows only one resonance mode. The small dip at 24.5 kHz is caused by the electronic disturbance. Because the disturbance appears both in the FRF of the cantilever tip and in the FRF of the chip, it disappears in the transfer function. Apparently the piezo element does not perform well at this frequency, since the cantilever is not excited at 24.5 kHz.

If the flexural resonance frequency of each cantilever is plotted against the input current, Figure 4.16 is obtained. Here it can also be seen that the flexural modes first decrease, and during phase transformation increase. The current at which transformation takes place is roughly equal for all three cantilevers, indicating that for these measurements the resistance is constant and a current \( i = 180 \text{ mA} \) results in a temperature of \( T \approx 55^\circ \text{C} \), which is the start temperature for R-phase to Austenite transformation. The two resonance modes located close to the flexural modes for cantilever E and F also change for varying input current, but much less. This is seen in Figure 4.17. Here it can also be seen that the phase transformation does not have as much influence on the resonance frequency as is seen for the flexural resonance modes.

4.4 Damping

As noted in Chapter 1, large damping may be present during phase transformation. From the measured frequency responses, however, it is hard so say if structural damping is responsible for the amplitude fluctuations. Because two resonance modes merge, the width of the peak is influenced by more than only damping. We do not further assess damping in these measurements.
4.5 Simulations

To validate the observation of the flexural resonance frequencies a numerical model is made. Possibly, the model can also explain the appearance of the second resonance peaks close to the flexural mode. To compute the resonance frequencies of the coated cantilevers we combine the evolution of the material properties from Section 3.1 with the measured curvature of the cross section. The Silicon cantilevers are known to have anisotropic material properties, possibly having a big influence on the location of the natural frequencies.

To compute the resonance frequencies as function of the temperature, we need to account for the following processes:

- The Young’s modulus is of single crystal Silicon is anisotropic;
- The Young’s modulus of the NiTi film changes during phase transitions;
- The curved cross section of the beam changes with the stress state in the material.

We assume the NiTi film has isotropic material properties. For the Silicon substrate, we formulate the anisotropic stiffness matrix

\[
C = \begin{bmatrix}
C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\
C_{21} & C_{22} & C_{23} & 0 & 0 & 0 \\
C_{31} & C_{32} & C_{33} & 0 & 0 & 0 \\
0 & 0 & 0 & C_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & C_{55} & 0 \\
0 & 0 & 0 & 0 & 0 & C_{66}
\end{bmatrix},
\]  

(4.13)
4.5 Simulations

for the translations and rotations $\mathbf{r} = [x \ y \ z \ \theta_x \ \theta_y \ \theta_z]^T$ in the crystal directions. According to the specifications in [Mik07], the cantilevers are cut from a $<100>$ wafer and the edges of the cantilever are along the $<110>$ direction. This implies that in bending, the Young’s modulus in the $<001>$ direction has to be used. For torsion, the shear modulus for torsion around the $<001>$ axis is used. This is clarified in Figure 4.18.

![Figure 4.18: Crystal structure directions for the Silicon cantilever.](image)

Table 4.3: Stiffness and compliance coefficients of Silicon. For the other parameters holds: $C_{22} = C_{33} = C_{11}, C_{12} = C_{13} = C_{21} = C_{23} = C_{31} = C_{32}$ and $C_{44} = C_{55} = C_{66}$.

$$
\begin{array}{c|c}
C_{11} & 165.7 \text{ GPa} \\
C_{12} & 63.9 \text{ GPa} \\
C_{44} & 79.6 \text{ GPa} \\
S_{11} & 7.68 \cdot 10^{-12} \text{ Pa}^{-1} \\
S_{12} & -2.142 \cdot 10^{-12} \text{ Pa}^{-1} \\
S_{44} & 12.56 \cdot 10^{-12} \text{ Pa}^{-1} \\
\end{array}
$$

For Silicon, only three different constants exist in the stiffness matrix. The inverse of (4.13) gives the compliance matrix $S$. The stiffness and compliance constants are listed in Table 4.3.

The Young’s modulus in the $<001>$ direction and the shear modulus for torsion are computed, using the direction cosine matrix $M$ of the respective axes and transposing the compliance constants to the axis of interest. This is done as follows [WE65]

$$
s'_{11} = s_{11} + s_c (l_1^4 + l_1^4 + l_1^4 - 1) \tag{4.14}
$$

$$
s'_{44} = s_{44} + s_c (l_2^2 l_1^2 + m_2^2 m_1^2 + n_2^2 n_1^2 - 1) \tag{4.15}
$$

$$
s_c = s_{11} - s_{12} - 1/2 * s_{44} \tag{4.16}
$$

where $l_i, m_i$ and $n_i$ are the elements of the cosine matrix for rotation around the desired axis.

$$
M = \begin{bmatrix}
l_1 & m_1 & n_1 \\
l_2 & m_2 & n_2 \\
l_3 & m_3 & n_3
\end{bmatrix}
= \begin{bmatrix}
1 & 0 & 0 \\
0 & \cos \theta & \sin \theta \\
0 & -\sin \theta & \cos \theta
\end{bmatrix}.
\tag{4.17}
$$
For our analysis, however, no rotation is necessary, so \( \cos \theta = 1 \) and we simply compute the compliance constants by substituting \( l_1, m_2 \) and \( n_3 \) by 1 and all other elements of the cosine matrix by zero. The Young’s modulus and shear modulus are computed using

\[
E_i = \frac{1}{s_{ii}}, \quad \text{and} \quad G_r = \frac{1}{s_{rr}}
\]

This results in a Young’s modulus for bending \( E_y = 130 \) GPa and the shear modulus for torsion \( G_{xx} = 64 \) GPa. For the NiTi thin film we assume isotropic, temperature dependent behavior. The parameters are shown in Table 4.1. Again, to compute the curvature of the beam, Timoshenko’s equation (3.3) is used.

\[
\rho = \rho_0 + \frac{(\alpha_f - \alpha_s)(T - T_0)}{\frac{h_s + h_f}{2} + \frac{2(E_s I_s + E_f I_f)}{E_s h_s w + E_f h_f w}}
\]

Knowing that the area moment of inertia is of the form \( I = wh^3 \), we conclude that the width of the beam has no influence on the curvature. This implies that the curvature of the beam is equal in every direction, provided that the Young’s moduli are equal. In our case, the Young’s modulus in the <001> direction is equal to the Young’s modulus in the <010> direction, so the curvature in these directions are also equal.

In Figure 4.11 the current-stress curves are presented. Due to the fluctuating resistance, the curves are not reproducible. From the measurements, however, we can conclude that at a current of 180 mA, the phase transformation starts. In Figure 4.11, the most left graph comes the closest to this observation, so we take this evolution of the curvature \( \rho \) as an estimation in our simulations. In Figure 4.19(a), the measured radius of curvature \( R = 1/\rho \) of the beam is shown. From this figure, we can assume that from 180 mA to 210 mA, the phase transformation takes place. The resulting (linear) change of the Young’s modulus is as shown in Figure 4.19(b).

(a) Radius of curvature \( R \) of the cross section as function of the induced current. This radius of the induced current.
(b) Young’s modulus of the NiTi film as function of the induced current. The curvature is the reciprocal of the curvature \( \rho \), shown in Figure 4.11.

**Figure 4.19:** Parameter evolution for the simulations.

For the Finite Element Analysis, we use an Euler beam element and add the stiffness and mass terms for torsion, as specified in Appendix G. Torsion is included, in order to check
whether the second peak, close to the flexural mode, is a torsional mode. The inertial terms are computed as in Appendix F. The analysis was done with 20 beam elements. For the three cantilevers with varying length, the simulation results are shown in Figure 4.20. Here the flexural resonance frequency is shown.

Figure 4.20: Simulated flexural resonance modes for varying current $i$ [mA]. The cantilevers D, E, and F have length 300 $\mu$m, 350 $\mu$m and 250 $\mu$m, respectively.

In Figure 4.20, we observe that the bending mode of cantilever E, 18.4 kHz at room temperature, first decreases slightly and during transformation increases. Although the relative change of the resonance frequency is less than measured, the qualitative behavior corresponds well. Cantilevers D and F show similar behavior. This is a strong indication that the measured shifts in the flexural resonance frequencies are indeed caused by the change in curvature and the change in the Young’s modulus. Possibly the curvature of the beam changes more than measured. Furthermore, the Young’s modulus in R-phase is estimated, using the measured temperature-stress curves from Section 4.1. This modulus may be lower than estimated, enlarging the shift in the resonance frequencies during phase transformation.

According to the simulations, the first torsional resonance frequency for all three cantilevers is higher than 200 kHz, so we can conclude that the measured resonance mode, close to the flexural mode, is not a pure torsional resonance frequency. It is not clear where this peak comes from. In the simulations, we did not include coupled bending and torsion and did not consider warping. Using coupled equations for bending and torsion, e.g. as discussed in [Ban89], and
Experiments and simulations

Including warping may change the location of the resonance frequencies drastically. Especially when the cross section of the cantilever has a large curvature, the influence of warping may be large. However, it is unlikely that with this analysis the torsional resonance mode of the cantilever shifts with more than a factor 6 down.

Discussion

In this chapter, we have fully characterized and modeled the thermomechanical behavior of the NiTi coated cantilever. Thermomechanical measurements provide interesting information about the transformation temperatures. The appearance of the R-phase in the working range of the current source gives the opportunity to switch between R-phase and Austenite phase during dynamic excitation.

Because the wire contacts on the film are glued, the resistance of the film severely fluctuates. This makes modeling heat transfer by Joule heating problematic. Alternatively, we have indirectly measured the temperature of the cantilevers during Joule heating and corrected for the temperature difference between the chip and the freestanding cantilevers.

From the frequency response functions of the cantilevers it can be concluded that the dynamic behavior is significantly influenced by the phase transitions in the material. The thermal mismatch between the Silicon and NiTi results in a change of curvature of the cross section, subsequently changing the flexural resonance frequency. This change is small though, compared to the frequency shift of 10%, caused by the phase transformations. Additionally, these shifts cause separation or merging of the two modes, resulting in a drastic change of their amplitudes. Because of this phenomenon, it is rather difficult to say anything about damping. If the experiments were conducted with other geometries, e.g. triangular shaped cantilevers, the flexural resonance mode may be more isolated and it would be less complex to assess the damping behavior of the film. It is not clear why two resonance peaks appear close to each other in the measurements. Both peaks shift for varying currents, implying that they are part of the dynamics of the coated cantilever.

Simulations of the flexural resonance frequencies correspond well with the measured responses. The small change due to a change of cross section curvature is observed, as well as the larger frequency shift due to the change of Young’s modulus. The model does not show the second resonance peak, close to the flexural mode. A dynamical model, capturing more of the dynamics than only the flexural and torsional modes, could clarify what the origin of this resonance peak is.

We conclude that with the SMA coating it is possible to actively influence the dynamics of the cantilevers in this setup. Whether this change is significant enough to cancel or damp unwanted vibrations needs to be further investigated. It is interesting to investigate the possibility to switch between Martensite phase and Austenite phase, instead of the R-phase and Austenite phase. Because Martensite has a much lower Young’s modulus than Austenite (30 GPa and 80 GPa, respectively), the frequency shifts are larger. Furthermore, investigation of the damping properties is necessary to gain a complete insight in the dynamics and in the applicability of this design. It can be concluded though, that the coated cantilever gives good opportunities for vibration control on micro scale and to add functionality in micro devices.
Chapter 5

Conclusions and recommendations

5.1 Conclusions

In this research we have investigated the possibility to actively influence the dynamical behavior of micro cantilevers by using a Shape Memory Alloy coating. A equiatomic Nickel-Titanium (NiTi) thin film is deposited on commercially available Silicon micro cantilevers and consequently annealing the film results in a crystallized NiTi film, showing temperature- and stress-induced Martensitic transformations. Because NiTi shows large differences in its material properties for different phases, the material is useful as a structural solution for influencing the dynamics of the cantilever.

Thermomechanics

First the thermomechanical behavior is investigated, using a calibrated heat source and optical profiler. From temperature-stress curves, the phase transformation temperatures are determined. The hysteresis curve is large, because the phase transformations are both temperature- and stress-induced. Because the stress levels in the material are low, a two-step transformation takes place when cooling from Austenite phase. This is the R-phase, which appears at 58.5 °C.

Experimental setup

An experimental setup is designed, suitable for investigating the dynamic response of micro cantilevers to an excitation, while the temperature of the NiTi film can be varied by an electrical current. The setup is capable of measuring small displacements (< 100 nm) at high frequency (up to 100 kHz). In the frequency domain, accurate frequency response measurements are possible.

Modeling

To understand and predict the behavior of the coated cantilevers, a thermomechanical model is made. This model gives insights in the transformation behavior of the material. The bimorph effect due to a mismatch in thermal expansion is modeled using Timoshenko’s equation for bimetallic thermostats, extended to include temperature dependent stiffness. For describing the hysteretic transformation behavior several mathematical models of hysteresis
are proposed. To fit the measurements the Krasnosel’skii-Pokrovskii hysteron is used, because it has an appealing simplicity, while still capable of modeling the complex hysteresis loops. Minor loops are represented by horizontal lines, but still the model represents the phase transformation behavior of the material well.

A 1D heat transfer model is formulated, describing the conduction, convection and Joule heating of the NiTi film. For estimating the electrothermal interaction, the temperature dependent resistivity is required. This is hard to measure, because the electrical circuit experiences resistance fluctuations due to the quality of the glued contacts. Because the temperature of the cantilever cannot be measured directly, validation of the model is not possible. Alternatively the thermal response is measured indirectly by measuring the profile of the cantilever for a controlled temperature and comparing this to the thermomechanical response on the electrical current. Because the resistance fluctuates, it still is difficult to determine the exact temperature of the cantilevers.

Experiments

The measured frequency response functions of the cantilevers show that changing the temperature of the film significantly influences the dynamics of the cantilevers. The measurements show two resonance peaks close to each other, of which one is identified as the flexural resonance mode. The appearance of the second resonance peak is not clear. Due to a change in the profile of the cross section, the flexural stiffness decreases for an increasing current. Phase transition from R-phase to Austenite phase increases the Young’s modulus, resulting in an increase of the flexural resonance frequency of as much as 10%. The second mode only change slightly. The two resonance peaks merge and separate under varying input currents, resulting in a change of amplitude. Because of this observation it is difficult to assess damping of the system.

The resonance frequencies of the beam are computed, using standard formulations of flexural and torsional stiffness and a finite element analysis. In these simulations the changing area moment of inertia and Young’s modulus are taken into account. The simulations show that the flexural resonance mode indeed is influenced by the changing cross section of the cantilever and by the change in the Young’s modulus. The frequency shifts are smaller than the measured ones, probably because the cross section curvature changes more than assumed and because the Young’s modulus in the R-phase is not estimated correctly. The simulations also show that the second peak, close to the flexural resonance mode, is not likely to be a torsional resonance frequency.

We can conclude that a NiTi coating on these cantilevers significantly influence its dynamic response. The amount of damping that the NiTi film adds is difficult to determine, because the modes are close to each other.
5.2 Recommendations

For future work on this topic, recommendations are done in both the experimental and in the analytical part of the research. We also discuss future work.

Experiments

For more accurate experiments, the setup for dynamic experiments can be improved. Although the LDGU is suitable for qualitatively measuring small displacements at a high frequency, its performance depends greatly on the reflectivity of the sample and how well it is aligned. Furthermore, the focusing lens has to be within 3mm of the sample, making access to the sample and shielding from the electromagnetic radiation of the piezo element problematic. A measurement principle for larger distance, for example a calibrated laser-diode combination as used in Atomic Force Microscopes, would greatly improve the flexibility of the setup.

Sample preparation was a very elaborate task in this project. Sputtering and annealing of the thin film on the cantilever chip was done very well, but attaching wires to the chip did not give satisfactory electrical properties. Sputter coating the chip partially with gold or silver can solve these problems, because then ultrasonic wirebonding will have more chance of giving good bonds. Moreover, a reproducible and accurate measurement of the film resistance as function of the induced current is useful for understanding both the electrothermal properties of the film and the phase transformation behavior. A four-point resistivity measurement is desired and could give good results, provided the wirebonds are of good quality. Because the resistance of the film is fluctuating severely, an accurate estimation of the cantilever temperature for varying current is not possible. It is recommended to investigate possibilities to directly measure the cantilever temperature. If the film is well connected to a current source, an indirect measurements could also give satisfactory results.

It is recommended to repeat the experiments with coated cantilevers with other dimensions and geometries. This may make interpretation of the frequency response functions less complex, because separate resonance peaks can be analyzed. It may also give the opportunity to investigate the damping properties of the material, which in our case is problematic.

Modeling

For modeling hysteresis in the phase transformations, several modeling approaches are presented. These are only a few options for mathematical modeling of hysteresis. A thorough investigation on other available models, as well as improvement of the proposed models can lead to a more accurate approximation of the hysteresis curves. Furthermore, a physical model describing the kinematics of transformation can improve the understanding of the thermomechanical behavior of the NiTi coated cantilever.

The measured frequency response functions only partially explain the dynamic behavior of the coated cantilevers. It is recommended to investigate the dynamical behavior of the coated cantilevers in more detail. If the model is extended to describe more than flexural and torsional modes, we may be able to better understand the measured dynamical behavior of the cantilevers.
Future work

In this research we have made choices for the dimensions of the cantilevers, the thickness of the deposited SMA film and the way of heating the film. These decisions have determined the frequency range of interest, the temperature range of interest and the thermomechanical characteristics of the samples. Concerning the range of applications of the cantilevers as a sensor or actuator, other geometries may be more favorable. A study on specific applications for coated cantilevers will provide requirements for the cantilever dimensions and may help in choosing the right film composition and thickness. The transformation temperatures can vary severely for varying film composition and film thickness, so it is important to select the right material for the right application. These choices are of great influence on the effect of the phase transformation on the dynamics of the cantilever and thus determine the success of this solution for structural vibration control of micro systems.

Shape Memory Alloys have several extraordinary properties. In this research we have investigated the influence of a change in the material properties on the dynamics of a micro cantilever. We did not consider the Shape Memory Effect or Superalasticity Effect. These two phenomena - also a result of phase transformations - are very interesting to use for actuating a (micro) device. Numerous publications (e.g. [KLR+96] and [DGD05]) report the use of these mechanisms in micro and macro applications. It is recommended to investigate whether a combination of our research with existing knowledge about SMA materials gives more opportunities for using it to add functionality and efficiency in the design of sensors and actuators.
Bibliography


Appendix A

Atomic Force Microscopy

One of the major fields of applications of micro cantilevers is Atomic Force Microscopy. The Atomic Force Microscope (AFM) was invented by Binnig, Quate and Gerber in 1986 and it was one of the first microscopes performing better than optical microscopes in terms of resolution.

An AFM is a type of Scanning Probe Microscope (SPM) and is capable of measuring structures with a resolution of fractions of a nanometer [Vel05]. The first SPM was not an Atomic Force Microscope, but the Scanning Tunneling Microscope, invented by Binnig and Rohrer in 1981. The name scanning probe implies that a probe is used to scan an object. The name microscope implies looking and is a bit contradicting with the former, since probing has more affinity with feeling.

(a) Schematic representation of the design of an AFM.
(b) The Van der Waals force as function of the relative distance between the atoms on the tip and the atoms on the substrate.

Figure A.1: Atomic Force Measurement principles.
In Figure A.1(a) the schematic layout of a certain type of AFM is shown. A micro cantilever with a very sharp tip - at the end only several atoms wide - is brought close to the surface of the substrate, in the order of a few nanometers. The interaction between the substrate surface and the cantilever tip results in deflection of the tip. One of the important forces in this range is the Van der Waals force, shown in Figure A.1(b). When the tip is relatively far away from the surface, the forces are small and the tip does not feel the surface. Coming closer, the tip is firstly attracted to the surface due to the attractive Van der Waals force. When brought closer, this force will become repulsive. Other interacting forces are mechanical contact forces, capillary forces, chemical bonding, electrostatic and magnetic forces. Analysis of these forces gives opportunities to investigate several properties of the substrate.

The cantilever is connected to a Z-stage, consisting of a piezo element. The stage has a resolution in the range of 0.1 nm (1 Å). The substrate is placed on an XY-stage, that moves the sample under the tip. This way, the tip probes the surface area of the sample. The tip will encounter a change in topography when the XY-stage is scanning it. This will bring the tip closer or further to the surface and consequently the interacting forces result in deflection of the cantilever. This deflection is measured by a laser-diode combination and fed back to the Z-stage, which will correct for the position change. The effort, needed to bring the tip back to its original position, gives information about the surface topography and other mechanical or physical properties. If the cantilever is charged or heated, electrical and thermal properties can be investigated.

Disturbing vibrations of the cantilever introduce measurement errors and noise. These disturbances can come from the surroundings or from moving parts in the AFM. The setup is therefore isolated from the surroundings as good as possible and sophisticated design principles and control algorithms for the piezo stages are used.
Appendix B

PIC181 PZT material properties

The specifications of the used piezo ceramic material are shown in Figure B.1. The piezo elements are fabricated by Physik Instrumente (PI) and distributed by Applied Laser Technologies in Best, the Netherlands. At PI, two types of piezo ceramics are available: ‘soft’ and ‘hard’ ceramics. ‘Soft’ refers to piezo’s which require less power to expand, but have a low quality factor (high damping), so dissipate more energy. ‘Hard’ piezo’s need more power, but dissipate less energy, are very stiff and have a high quality factor. They are suitable for high power applications and oscillators [Phi01]. PIC181 is a hard ceramic with an extremely high quality factor and a high curie temperature, making it suitable for high frequency applications.
### Material Data

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Further information:
The following values are valid approximations for all PZT materials from R. Ceren.

Specific heat capacity | HC = approx. 350 J / kg K
Specific thermal conductivity | TC = approx. 1.1 W / m K
Poisson’s ratio | v = approx. 0.34
Coefficient of thermal expansion | αx = approx. 4 to 6 x 10⁻⁶ / °C (in the polarization direction, molded)
Static compressive strength | larger than 600 Mpa

Figure B.1: PIC181 PZT material properties [Phi01].
Appendix C

NSC12/Tipless/No Al Cantilevers

Single crystal (100) Silicon chips, containing six cantilevers of different length, are supplied by µMasch. In Figure C.1 the dimensions of the cantilevers are given. Due to the production method - lithography and chemical etching - the dimensions have a significant uncertainty. The resonance frequencies and force constants are also given.

![Image of SEM image of the short-tipless tipless cantilever A, B, C on chip of the NSC12 series.]

**Figure C.1:** NSC12/Tipless/No Al Cantilever chip specifications [Mik07].
Appendix D

Piezo amplifier design

The electrical design of the piezo amplifier is shown in Figure D.1. The layout was designed by Michael Hopper. The Central Technical Services (GTD) of the Eindhoven University of Technology have made some adjustments and have built the amplifier with additional cooling facilities. It uses a 400 V, 600 mA power supply and the input is generated using NI Labview and a 1.25 MHz NI-USB 6251 data acquisition card.
Figure D.1: Electrical design of the piezo amplifier.

Piezo amplifier design
Appendix E

LDGU amplifier design

The electrical design of the LDGU amplifier is shown in Figure E.1. The Central Technical Services (GTD) of the Eindhoven University of Technology has designed and built the amplifier, according to specifications in [Phi00].
Figure E.1: Electrical design of the LDGU amplifier.
Appendix F

Area moment of inertia of a sector of a hollow circle

For computing the area moment of inertia of the beam around several axes of rotation, we use the following approach. The cross section of the beam is considered as a sector of a hollow circle. It is convenient to use polar coordinates. Schematically the cross section and relevant dimensions are shown in Figure F.1.

![Figure F.1: Dimensions of the NiTi coated cantilever cross section.](image)

First we compute the centroid for bending around the y-axis.

\[
y_c = \frac{1}{A} \int_A ydA = \frac{1}{A} \int_{-\alpha}^{\alpha} \int_{R}^{R+t} r \cos \theta r dr d\theta, \]

where

\[
A = \int_{-\alpha}^{\alpha} \int_{R}^{R+t} r dr d\theta = \alpha t (2R + t). \]
This gives
\[ y_c = \frac{2 \sin \alpha \left( 3R^2 + 3Rt + t^2 \right)}{3\alpha} = \frac{2 \sin \alpha}{3\alpha} \left( R + t + \frac{R^2}{2R + t} \right). \] (F.3)

The inertia around the x-axis is computed through
\[ I_{y0} = \int_A y^2 dA = \int_{-\alpha}^{\alpha} \int_{R}^{R+t} r^2 \cos^2 \theta r dr d\theta, \] (F.4)

Steiner’s law gives the inertia around the centroid \( y_c \)
\[ I_y = I_{y0} - y_c^2 A = \frac{1}{4}t(2R + t)(2R^2 + 2Rt + t^2)(\cos \alpha \sin \alpha + \alpha) - \frac{4 \sin^2 \alpha \left( 3R^2 + 3Rt + t^2 \right)^2 t}{9\alpha \left( 2R + t \right)} . \] (F.5)

Similarly, for the moment of inertia around the z-axis we write
\[ I_z = \frac{1}{4}t(2R + t)(2R^2 + 2Rt + t^2)(- \cos \alpha \sin \alpha + \alpha). \] (F.6)

For both the film and the cantilever, the inertia with respect to their own centroid is computed, simply by integrating over \( R + h_f \) and \( R \) for the film and over \( R + h_f + h_s \) and \( R + h_f \) for the cantilever. The centroid of the total cross section is computed by integrating over \( R + h_f + h_s \) and \( R \). The inertia of the elements with respect to this centroid can be computed using Steiner’s law.
Appendix G

Mass and stiffness matrices for FE analysis

In Figure G.1, a representative beam element is shown.

Deflection $z$ of the cantilever and bending around the $y$-axis $\theta_y$ are represented by the Euler beam element matrices. The stiffness and mass matrix for an Euler beam element are

$$K^e = K_f^e \begin{bmatrix} 12 & 6L & -12 & 6L \\ -6L & 4L^2 & -6L & 2L^2 \\ 12 & -6L & 4L^2 \end{bmatrix} _{sym},$$  \hspace{1cm} (G.1)

where $K_f^e = \frac{E I_y}{L^3}$.

$$M^e = M_f^e \begin{bmatrix} 156 & 22L & 54 & -13L \\ 22L & 4L^2 & 13L & -3L^2 \\ 54 & 13L & 156 & -22L \\ -13L & -3L^2 & -22L & 4L^2 \end{bmatrix} _{sym},$$  \hspace{1cm} (G.2)

where $M_f^e = \frac{\rho A L}{420}$. 

Figure G.1: Beam element, representing bending and torsion.
For torsion, the element stiffness and mass are

\[ K^e_t = \frac{GK}{L}, \quad M^e_t = \frac{\rho J_p L}{3}, \]  

(G.3)

where \( K \) is the torsional constant and \( J_p = I_y + I_z \) is the polar moment of inertia. If we assume uncoupled torsional and flexural vibrations, the element mass and stiffness matrices for are

\[
K^e = \begin{bmatrix}
12K^e_f & 6K^e_f L & 0 & -12K^e_f & 6K^e_f L & 0 \\
4K^e_f L^2 & 0 & -6K^e_f L & 2K^e_f L^2 & 0 \\
K^e_t & 0 & 0 & -K^e_t & 0 \\
12K^e_f & -6K^e_f L & 0 & 4K^e_f L^2 & 4K^e_f L^2 \\
sym
\end{bmatrix}
\]

(G.4)

\[
M^e = \begin{bmatrix}
156M^e_f & 22M^e_f L & 0 & 54M^e_f & -13M^e_f L & 0 \\
4M^e_f L^2 & 0 & 13M^e_f L & -3M^e_f L^2 & 0 \\
M^e_t & 0 & 0 & M^e_t/2 & 0 \\
156M^e_f & -22M^e_f L & 0 & 4M^e_f L^2 & 0 \\
sym
\end{bmatrix}
\]

(G.5)

where the degrees of freedom are

\[
\tau = \begin{bmatrix}
z_1 \\
\theta_{\phi 1} \\
\theta_1 \\
z_2 \\
\theta_{\phi 2} \\
\theta_2
\end{bmatrix}
\]

(G.6)
Appendix H

Dynamic measurements

The dynamic measurements were conducted on the three coated cantilevers with the approximate length of 250 µm (Figure H.1), 300 µm (Figure H.2) and 350 µm (Figure H.3). The cantilevers and chip are excited by a white noise signal with an amplitude of 0.05 V, amplified to ~5 V by the piezo amplifier. The diode signal $D_2 - D_3$ is acquired by the NI USB-6251 at a rate of 700 kHz, with 10,000 samples per measurement interval. The Nyquist frequency - the highest reconstructible frequency - thus is 350 kHz. The frequency resolution is

$$f_r = \frac{f_m}{N} = 70 \text{ Hz},$$

(H.1)

where $f_m$ is the sample rate and $N$ is the number of samples per measurement interval. The measurement time interval is 14.3 ms. The shown responses are an average over 200 measurements and a Hanning window is used to reduce signal leaking.

To properly visualize the difference between the responses, they are given a certain offset. This gives a clear sight on the evolution of both the location and the amplitude of the resonance frequencies in the relevant frequency range.
Figure H.1: Frequency response functions of cantilever D, with length $L = 300\mu$m.
Figure H.2: Frequency response functions of cantilever E, with length $L = 350\mu m$. 
Figure H.3: Frequency response functions of cantilever F, with length $L = 250\mu m$. 