University of South-Eastern Norway

Per Kristian Bolstad

Evaluation of Metallurgical Bonding for Ultrasound Transducers

Dissertation for the degree of Ph.D
Applied micro- and nanosystems

Faculty of Technology, Natural Sciences and Maritime Sciences

University of
South-Eastern Norway

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Dedication

Ultrasound was not a field I was especially familiar with that day I approached Professor Lars Hoff to ask about his available master's projects. A few days before, I had heard about an exchange program to Los Angeles, so I was California Dreamin'. It was through my master's project that I got to know the supervisor trio of Professor Lars Hoff, Associate Professor Tung Manh, and Associate Professor Martijn E. Frijlink. This excellent team sparked my interest in ultrasound transducer design. A PhD was not a part of the plan, but the opportunity was hard to resist, knowing this trio, along with Associate Professor Hoang Vu Nguyen, would help me through it.

Whether intentionally or not, my parents inspired interests which served as helpful background for this PhD. Picking apart electronics, keeping up to date on the latest developments in technology, and making noise on expensive HiFi systems and cheap guitar amps, are in essence what transducer design is about. You motivated me to take on this challenge and have supported me the whole way. This thesis is dedicated to my mother and father, Kari and Olbrigt.

My free time, I dedicate to you, Victoria, the most patient and loving person a PhD candidate can hope for. Bonding is a central topic of this work, and while metallurgical bonds might be strong, the bond we have is stronger.

I have great appreciation for all those who have shown me support through this. I miss the close group of friends from USN, yet I'm thankful for the great colleagues I have gained from Kongsberg's transducer team.

Brødrene B og gutta backer.

Preface

This doctoral thesis is submitted in partial fulfilment of the requirements for the degree of Philosophiae Doctor at the Faculty of Technology, Natural Sciences and Maritime Sciences at the University of South-Eastern Norway.

The work was carried out at the Department of Microsystems, University of South-Eastern Norway, Campus Vestfold, Norway, under the supervision of Professor Lars Hoff, Associate Professor Martijn E. Frijlink, Associate Professor Tung Manh, and Associate Professor Hoang Vu Nguyen. All supervisors are with the Department of Microsystems, University of South-Eastern Norway, Campus Vestfold, Norway.

This work was supported by the Research Council of Norway through the Centre for Innovative Ultrasound Solutions (CIUS), project number 237887. The Research Council of Norway is acknowledged for the support to the Norwegian Micro- and Nano-Fabrication Facility, NorFab, project number 295864.

Abstract

Common ultrasound transducers use a piezoelectric element to convert between electrical and mechanical energy. The piezoelectric layer is sandwiched between acoustic matching and backing layers to maximize energy transfer and bandwidth. The bonding layers joining these materials are conventionally made of polymers, and decades of experience verify that it functions excellently in many ultrasound transducer applications. These bonding layers should be very thin compared to the acoustic wavelength to avoid reverberations and reduced bandwidth. Transducers for operation in harsh environments rely on careful selection of materials, and polymer materials are known to degrade at high temperatures.

Metallurgical bonding is proposed as an alternative to polymeric adhesives. Metals form strong, electrically- and thermally conductive bonds with a high characteristic acoustic impedance. Solid-liquid interdiffusion (SLID) bonding is a technique which relies on the formation of intermetallic compounds, which are stable at temperatures above the processing temperature. SLID bonds are not reflowable, as compared to bonds formed through soldering.

This thesis explores the binary metal system of gold (Au) and Tin (Sn) for bonding essential layers of ultrasound transducers, such as piezoelectric ceramic materials. Metallurgical bonding techniques, Au-Sn SLID and Au-Sn soldering, are evaluated and compared to conventional polymeric adhesives. Testing of bonding performance during and after exposure to high temperature and high pressure indicated high-temperature stability with high mechanical strength.

A common challenge related to metallurgical bonding is the formation of voids within the layer, which are gas or vacuum-filled pockets. Reliable estimation of the influence from such voids on the acoustic performance is important if metallurgical bonding is to be used in acoustic transducers. A finite element method modelling technique is presented in this thesis, which can be applied to accurately estimate effective medium parameters of layers containing voids of arbitrary size, concentration, and distribution.

Keywords: Ultrasound, Piezoelectric, Transducers, Bonding, Solid-Liquid Interdiffusion, Soldering, Characterization, Finite Element Method.

List of papers

Papers omitted from online publication due to publisher's restrictions

Article 1

P. K. Bolstad, M. E. Frijlink, T. Manh and L. Hoff, "Estimating Effective Material Parameters of Inhomogeneous Layers Using Finite Element Method," in *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, vol. 69, no. 12, pp. 3402-3410, Dec. 2022.

Article 2

P. K. Bolstad, M. Frijlink, and L. Hoff, “Evaluation of bonding techniques for ultrasound transducers,” in *Microelectronics Reliability*, vol. 151, p. 115272, Dec. 2023.

Article 3

P. K. Bolstad, D. Le-Anh, L. Hoff and T. Manh, "Intermetallic Bonding as an Alternative to Polymeric Adhesives in Ultrasound Transducers," in *2019 IEEE International Ultrasonics Symposium (IUS)*, Glasgow, UK, 2019.

Article 4

P. K. Bolstad, S. L. Kuziora, H. -V. Nguyen, T. Manh, K. E. Aasmundtveit and L. Hoff, "Impact of High Pressures on Au-Sn Solid Liquid Interdiffusion (SLID) Bonds," in *2020 IEEE 8th Electronics System-Integration Technology Conference (ESTC)*, Tønsberg, Norway, 2020.

Article 5

P. K. Bolstad, M. Frijlink and L. Hoff, "Metallurgical AuSn Bonding of Piezoelectric Layers," in *2022 IEEE International Ultrasonics Symposium (IUS)*, Venice, Italy, 2022.

Article 6

P. K. Bolstad, L. Hoff, H. Torp and T. F. Johansen, "Design, Fabrication and Testing Highly Sensitive Single Element Doppler Transducers," in *2018 IEEE International Ultrasonics Symposium (IUS),* Kobe, Japan, 2018.

List of figures

Abbreviations

USN	University of South-Eastern Norway
SLID	Solid-Liquid Interdiffusion
IMC	Intermetallic Compound
PZT	Lead-Zirconate Titanate
PMN-PT	Lead Magnesium Niobate-Lead Titanate
AC	Alternating Current
FEM	Finite Element Method
SAM	Scanning Acoustic Microscopy
SEM	Scanning Electron Microscopy
EDX	Energy Dispersive X-ray Spectroscopy
Au	Gold
Sn	Tin
Si	Silicon
Cr	Chromium

Table of contents

1 Introduction

1.1 Background and Motivation

Common ultrasound transducers use a piezoelectric element to convert between electrical and mechanical energy. The piezoelectric material is usually sandwiched between an acoustic backing material and one or more acoustic matching layers to enhance the acoustic performance. This is called the acoustic stack. The material choice and thicknesses of these backing and matching layers are optimized towards desired properties for the selected application, such as bandwidth, sensitivity, and directivity, where robustness and complexity must be considered. Optimization is done by carefully selecting materials with specific acoustic properties and fine-tuning the layer thicknesses.

The acoustic matching and backing layers need to be bonded to each other and to the piezoelectric material. This is commonly done using a polymer adhesive, e.g. epoxy resin, and decades of experience verify that it functions excellently in many ultrasound transducer applications. However, there are drawbacks associated with this method, most notable in some special applications.

Any bonding layer can influence the acoustic performance of the transducer. The normal way to mitigate this is to make the layer thin; ideally, the layer thickness should be negligible compared to the acoustic wavelength inside the layer [1]. This can be challenging for high-frequency transducer designs where wavelengths are short. Designs incorporating de-matching techniques are also especially sensitive to influences from the bonding layer.

Transducers for operation in harsh environments are reliant on careful selection of materials. In this thesis, harsh environments are defined according to the definition by the American Petroleum Institute as temperatures and pressures exceeding 177 °C and

1030 bar [2]. Epoxy adhesives are generally sufficiently strong at room temperature but degrade at elevated temperatures [3].

Metallurgical bonding techniques are proposed as an alternative to polymeric adhesives to address the mentioned challenges. Such bonding techniques are common in the interconnect industry for packaging of electronic components; however, their applications for bonding layers of ultrasound transducers are less common. Metals have a higher speed of sound compared to polymers, which allows for relaxed requirements on bonding layer thickness and uniformity. Certain metal systems are stable at elevated temperatures.

A common challenge related to metallurgical bonding is the formation of voids, which are gas or vacuum-filled pockets scattered throughout the bonding layer. Reliable estimation of the influence from such voids on the acoustic performance is important if metallurgical bonding is to be used in acoustic transducers.

1.2 Research Goals and Tasks

This research project aims to investigate novel methods for bonding layers in the acoustic stack in ultrasound transducers. The project is based on the long experience and expertise in bonding technology in the packaging group at USN, combined with the acoustic expertise in the ultrasound group. The main topic will be applying solid-liquid interdiffusion bonding, SLID, to build ultrasound transducers. The fabricated transducers shall be tested for behaviour during and after exposure to high pressure, high temperature, and for mechanical robustness.

The research questions are:

1. Can metallurgical bonding be used to bond layers of the acoustic stack?
2. What are the limitations of developed metallurgical bonding techniques?
3. How will high pressure and high temperature influence transducer performance?

1.3 Thesis Outline

The following introductory chapters serve as relevant background for concepts presented in articles and proceedings. The aim is to give the reader an understanding of ultrasound applications and the importance of ultrasound transducer design, including modelling, fabrication and characterization. This is the topic of Chapter 2.

Since the focus of this thesis is to develop novel techniques for bonding layers of a piezoelectric ultrasound transducer, a fair understanding of the state of the art is important. The conventional polymer adhesive bonding technique and metallurgical bonding techniques developed in this thesis are the topic of Chapter 3. This chapter is relevant background for Paper 2, Paper 4, and Paper 5.

The metallurgical bonding techniques developed in this thesis are applicable to most applications. Yet, applications involving harsh environments, such as high temperatures and high pressures, are of special interest. In Chapter 4, the effects of harsh conditions on essential materials in ultrasound transducers are discussed.

Metallurgical bonding layers often contain voids, which may influence the elastic properties, and consequently, the acoustic properties of the layer. Influence from voids and delamination of the bonding layer of a transducer was studied in Paper 3. This inspired the development of the FEM-based approach to the estimation of effective material parameters of inhomogeneous layers, which is the topic of Paper 1. Chapter 5 presents the background and motivation for the development of the FEM-model.

The articles included in this thesis are summarized in Chapter 6.

Chapter 7 concludes the thesis.

2 Introduction to Piezoelectric Ultrasound Transducers

2.1 Ultrasound Transducers

You might have heard about ultrasound, but you might never hear it. Humans perceive sounds at frequencies ranging from approximately 20 Hz to 20 kHz [4]. Sound with frequencies above this range is called ultrasound. The field of application for ultrasound includes medicine, marine operations, non-destructive evaluation, seismology, wireless energy transfer and harvesting, and more [5][6][7].

The transducer is the part of the system that transmits and receives sound waves. Many ultrasound transducers rely on the piezoelectric effect, a phenomenon in which a material mechanically deforms upon the application of an electrical field and vice versa. The direct piezoelectric effect refers to the phenomenon in which the application of stress causes a net electric charge in the material. In the reverse, or inverse effect, a potential difference across the material will induce a deformation of the material. This deformation causes tensile or compressive stresses and strains in the material and is dependent on the direction of the electric field, the direction of which the piezoelectric material is polarized, the geometric dimensions of the material and mechanical clamping of the material. When the piezoelectric material is subjected to an alternating electric field, the material will vibrate, creating alternate compression and rarefication in the surrounding media.

For this reason, the piezoelectric layer is often referred to as the active layer. Acoustic matching techniques are often employed by use of acoustic matching layers, sometimes referred to as passive layers. Their purpose is to increase energy transfer from the active layer to the measurement target and to limit ringing.

2.2 Piezoelectric Materials

Piezoelectric materials occur naturally, and Quartz was implemented in the first piezoelectric transducer, invented in the early 1900s by Paul Langevin [8]. Now, a variety of synthesized ferroelectric materials with stronger piezoelectric coupling are available. Lead-Zirconate Titanate (PZT) ceramics are the most common piezoelectric material in ultrasound transducers to date. Single-crystal materials, such as Lead Magnesium Niobate-Lead Titanate (PMN-PT) have seen increased use in recent years due to the increased electromechanical coupling coefficient of the materials. Several other piezoelectric materials exist but will not be covered.

In a piezoelectric ultrasound transducer, the active layer can be either bulk ceramic or single crystal. Piezoelectric composites are another common implementation, where piezoelectric ceramic or single crystal is bound to a polymer material. Newnham et al. [9] first described the dimensional connectivity of such composites as m-n connectivity, where m refers to the connectivity dimensions of the piezoelectric material and n refers to the connectivity of the polymer matrix. Figure 2-1 shows the most common connectivity configurations used in ultrasound transducers, which are the 2-2, 1-3 and 0-3 connectivity composites [10]. Ordered piezoelectric composites can achieve high bandwidth and sensitivity with the benefit of reduced characteristic acoustic impedance relative to the load medium.

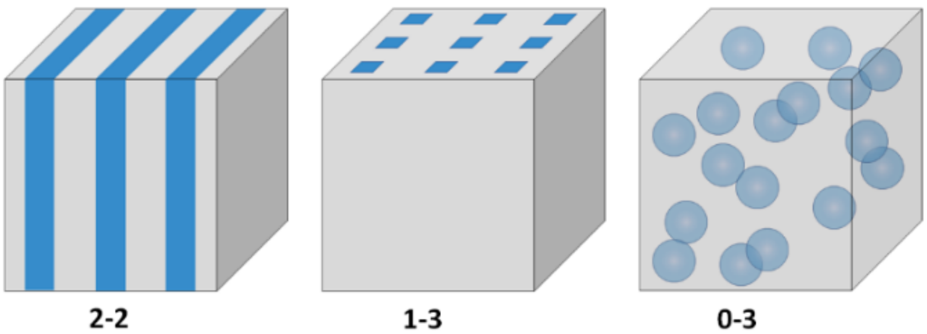


Figure 2-1 – The most common connectivity configurations used in ultrasound transducers.

An understanding of key material characteristics of the piezoelectric layer is important when designing a transducer for a specific application. There are several different piezoelectric compositions, each with distinct properties. A brief summary of properties relevant to this thesis is given below.

Electromechanical coupling coefficient: The electromechanical coupling coefficient, k, is a measure of the material's ability to convert mechanical energy to electrical energy and vice versa. This is a unitless quantity ranging from 0 to 1. Single-crystal materials possess the highest electromechanical coupling coefficient of all materials. The coefficient for PZT materials ranges from 0.4 to 0.7. Piezoelectric composites are known to increase the coupling coefficient due to the altered resonance mode of the piezoelectric ceramic material.

Losses: Piezoelectric materials are lossy, meaning some of the energy input will be converted to heat. This is also the case for non-piezoelectric materials. In the lumped representation of a piezoelectric material, losses can be represented by the mechanical quality factor Q_m and dielectric loss tan(ϑ). In distributed-parameter models and FEM, Holland's representation of loss is commonly used. Here, material parameters are represented as complex numbers and losses are represented by the imaginary component. In these representations, losses are defined by mechanical loss tangent $tan(\varphi)$, dielectric loss tangent tan(ϑ) and piezoelectric loss tangent tan(δ) [11].

Permittivity: This property describes how the material behaves in the presence of an electric field. Relative permittivity, also often referred to as dielectric constant, is the permittivity of a material relative to the permittivity of vacuum. The permittivity of the piezoelectric material is important to consider since the capacitance, and consequently, the electrical impedance magnitude of the piezoelectric is defined by the product of permittivity and surface area over the thickness of the piezoelectric.

Curie temperature: The Curie Temperature of a piezoelectric material is an important property to consider, both during transducer fabrication and in transducer operation. At the Curie Temperature, the piezoelectric material will lose polarization and will no longer possess piezoelectric properties. Piezoelectric materials already exhibit signs of depolarization at temperatures below the Curie Temperature. A general rule of thumb is to not expose piezoelectric materials to temperatures above half of the Curie Temperature [8]. The Curie temperature is dependent on the composition of the piezoelectric material but generally ranges from 150 °C to 350 °C for piezoelectric ceramics. A depolarized piezoelectric material may regain polarity by application of an electrical field greater than the coercive field. The coercive field is the electrical field strength required to alter the domain polarity of the piezoelectric. This field strength varies with temperature, and polarization is often performed by first heating the material in order to reduce the required electric field strength for polarization saturation. During transducer operation, the drive voltage should be kept sufficiently low as to not exceed the coercive field, causing depoling [8].

Characteristic acoustic impedance: Matching the acoustic impedance of the piezoelectric layer to the load is important for optimal energy transfer. This is the topic of the following section.

2.3 Acoustic Matching

The acoustic impedance of the piezoelectric material should match the impedance of the load medium to achieve optimal energy transmission. Bulk ceramic PZT materials and piezoelectric single crystals generally have an acoustic impedance close to 30 MRayls. With piezoelectric composites, acoustic impedances in the range from 10 MRayls to 20 MRayls are possible. Common loads such as water and biological tissue have characteristic impedances around 1.5 MRayls [12]. Selecting a piezoelectric material with low acoustic impedance can improve energy transfer to the load since a large impedance mismatch between the piezoelectric layer and the load will cause strong reflections. Acoustic matching layers are often used to compensate, where this layer acts as an intermediate step. The transmission line model [8] can be adapted to give the impedance of a piezoelectric layer, Z_p, seen by the acoustic load Z_L through a matching layer Z_m of thickness L as

$$Z_p = Z_m \frac{[Z_L + jZ_m \tan kL]}{[Z_m + jZ_L \tan kL]} \qquad (2.1)$$

where k=2π/λ_m is the wavenumber in the matching layer. For a layer thickness of $\lambda_m/4$, the acoustic impedance of the matching layer is given as

$$Z_m = (Z_p Z_l)^{1/2}. \qquad (2.2)$$

Desilets et al. [1] found that effectively lower target values for the matching layer impedances could be derived based on the KLM equivalent circuit model. For a single matching layer, the matching layer impedance is

$$Z_m = (Z_p Z_l^2)^{1/3}. \qquad (2.3)$$

Two matching layers may be used to improve wideband performance. The acoustic impedance of the first and second matching layers is calculated as

$$Z_{m1} = (Z_p^4 Z_l^3)^{1/7} \qquad (2.4a)$$

$$Z_{m2} = (Z_p Z_l^6)^{1/7}. \qquad (2.4b)$$

Furthermore, Lau. et al. [13] demonstrated that two matching layers of λ/8 thickness results in improved wideband performance compared to two matching layers of λ/4 thickness. The two λ/8 layers can be considered as one λ/4 layer with graded acoustic impedance.

The back surface of the piezoelectric also has to be considered for optimal energy transfer and is referred to as acoustic backing. With an air-backed transducer, most of the acoustic energy that reaches the back of the piezoelectric element will be reflected into the forward direction due to acoustic impedance mismatch between the piezoelectric material and air. The reflected energy will reverberate inside the piezoelectric element, causing a ringing effect, which lengthens the pulse duration. Backing materials can be used to damp out the ringing effect and thereby increasing bandwidth. The backing material should not only absorb part of the energy from the vibration of the back face but also minimize the mismatch in acoustic impedance. This suppression of ringing or shortening of pulse duration is achieved by sacrificing sensitivity since the backing material absorbs a large portion of the energy.

De-matching is a technique in which the backside of a piezoelectric layer is coupled to a layer of high acoustic impedance, typically tungsten-based materials. For an air-backed de-matching layer (DML) of thickness $\lambda_{dml}/4$, the impedance seen from the piezoelectric layer is infinitely high, thereby effectively clamping the back end of the piezoelectric layer at the fundamental resonance. Paper 3 employs these mentioned front matching and de-matching concepts in simulations of a transducer.

2.4 Modelling Techniques

There are several ways of representing a piezoelectric transducer. Mechanical components, such as masses, springs, and dampers, may be represented by equivalent electrical components as capacitors, inductors, and resistors. The simplest form of representing a transducer is by the lumped parameter model. The lumped model is shown in Figure 2-6B in Ch 2.6.

More complex and accurate representations are the distributed-parameter models [8]. The Mason’s model [14] is a commonly used distributed model where the transducer is treated as a three-port network of two mechanical ports, representing the front and back surfaces of the transducer and one electrical port. xTrans [15] is an implementation of the Mason’s model in a MATLAB (MathWorks Inc. Natick, MA) program, developed at the Department of Circulation and Medical Imaging at NTNU and was used for one-dimensional modelling in this work. One limitation of the distributed models is that only effects from thickness mode resonances are predicted. Despite this, the modelling technique is frequently used in transducer design and evaluation.

More detailed analysis is performed using the finite element method (FEM). FEM is a numerical technique for finding approximate solutions to partial differential equations. The modelled structure is divided into smaller, finite elements. The equations that model the finite elements are then assembled into a larger system of equations that models the entire structure [16]. COMSOL Multiphysics (COMSOL Inc. Burlington, MA) was used for FEM simulations in this thesis.

2.5 Fabrication Techniques

The production of bulk or composite piezoelectric ultrasound transducers is a labour-intensive process, involving multiple fabrication steps. Each original transducer design requires a distinct fabrication process, yet certain steps are common in most fabrication processes. To illustrate, the process-flow for fabrication, testing and characterization of samples in Paper 2 is shown in Figure 2-2. Green boxes are all fabrication steps, which are summarized below. Blue boxes are characterization steps, which are summarized in 2.6. Characterization is often performed in between fabrication steps for quality control and traceability of the fabrication process. Characterization was also performed before and after exposure to harsh environments, as shown by the red-orange box marked “expose”.

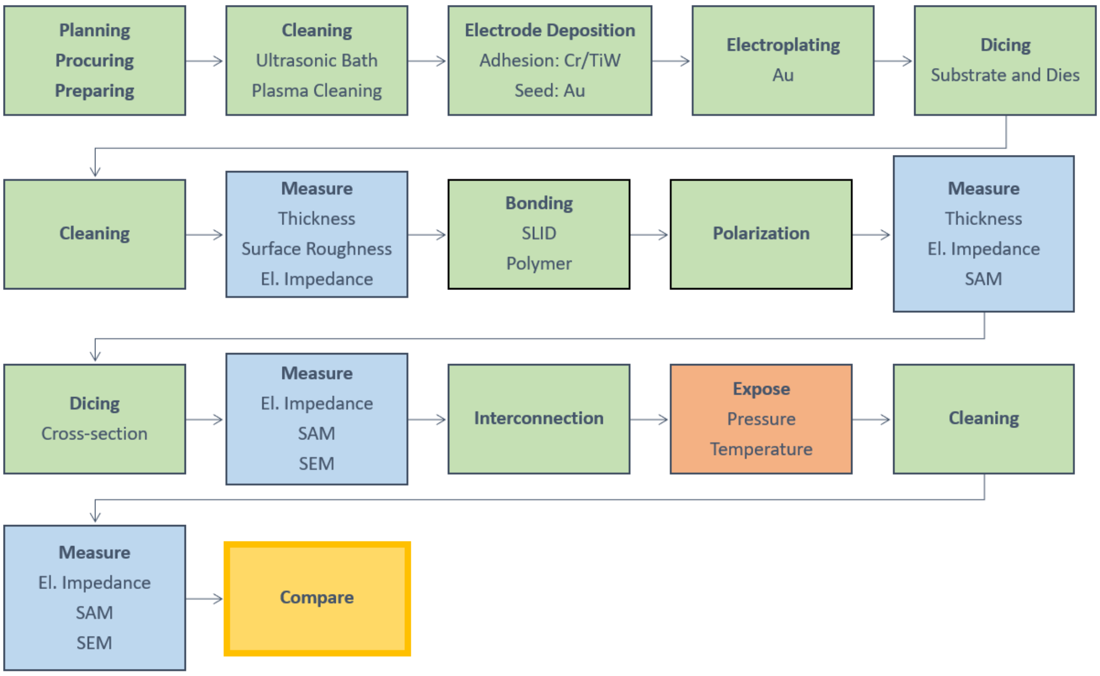


Figure 2-2 – Process-flow for fabrication and characterization of samples.

3xP: The first step is 3xP, namely planning, procuring, and preparing. The plan was to do a comparative study on different bonding techniques for ultrasound transducers. The necessary materials, such as piezoelectric ceramics, epoxies and an Au-Sn pre-form and Si substrates were procured. Since the fabrication of samples involves a variety of laboratory equipment, booking instruments is important.

Cleaning: This step is important prior to any deposition step. This ensures the sample is free from contaminants and is often performed to ensure good adhesion between layers. Cleaning in a heated ultrasonic bath with various solutions such as soaps, alcohols, or de-ionized water is common. Prior to electrode deposition, plasma cleaning may be performed to remove any remaining contaminants and to further improve adhesion. Plasma cleaning may introduce significant heat to the sample and care must be taken not to overheat temperature-sensitive materials.

Electrode Deposition: Metallization of surfaces to be used as electrodes can be performed by electron beam deposition (E-Beam) and sputtering techniques. Electrodes are often multi-layered metal films where each layer has a specific purpose. Certain metals may provide multiple functions.

- *Adhesion Layer: Ensures adhesion between adherend and succeeding metal films.*
- *Diffusion Barrier Layer: Prevents interdiffusion between adherend and deposited metal films.*
    - *Cr, Ni and $Ti_{90}W_{10}$ were explored as adhesion/diffusion barrier layer.*
- *Top Metallization Layer: The top surface layer which should be conductive and ensure good wetting characteristics, i.e., how well liquids spread over the surface.*
    - *Au was used as the top metallization layer in this work.*

Electroplating: Electroplating processes are used when thicker metal layers are required. These thicknesses are not obtainable through sputtering or E-beam alone. Since µm-thick layers of Au are needed for SLID bonding, Au electroplating was performed.

Dicing: A saw is used to cut a larger sample into smaller samples. These tools require careful programming of settings such as cut-length, cut-depth and cut-speed, as well as specification of cut-locations.

Bonding: Two layers are coupled together mechanically and acoustically through a bonding process. This processing step is much of the focus of the thesis and will be explained in detail in Chapter 3.

Polarization: Piezoelectric materials are often polarized prior to transducer fabrication. However, fabrication steps involving high temperatures may depolarize the piezoelectric material. In this work, repolarization was necessary after SLID bonding. Polarization was performed in room temperature without the application of stress.

Interconnection: Connecting wires for electrical connection can be done through a variety of ways and should consider the sample material, electrode configuration and environment of operation. Soldering is a common interconnection technique which produces strong connections. The challenge with this technique is the high localized temperatures involved. Conductive epoxy is a common alternative for temperature-sensitive materials but may be unsuitable for high operating temperatures.

Grinding/Lapping/Polishing: The process flow in Figure 2-2 does not involve grinding, lapping and polishing, yet this process deserves a mention. Grinding, lapping, and polishing are common processes for tuning a layer’s thickness and surface finish.

2.6 Characterization Techniques

This section introduces characterization techniques frequently used throughout the thesis. Visual inspection, material characterization and electrical measurements were essential in assessment of bond performance during fabrication as well as before and after exposure to harsh environments.

Optical Microscopy: Optical microscopy is a useful technique for visual inspection limited to unobstructed surfaces. Magnification is achieved through focused lenses. Most advanced optical microscopes available today are controllable by a computer, making image capture and general characterization simple. Figure 2-3 shows two micrographs where 200× magnification was used to capture the cross-section of a Si-SLID-Si sample (top) and Si-SLID-PZT sample (bottom). Both samples have bonds of uniform intermetallic compounds (IMCs) containing voids. These samples are discussed further in Paper 4.

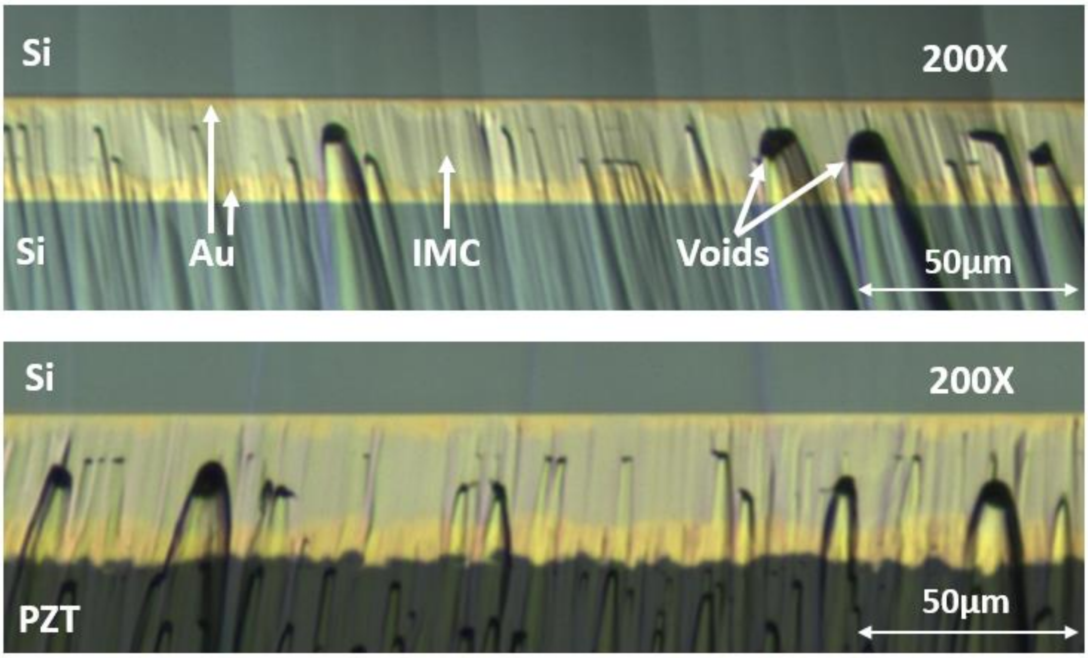


Figure 2-3 – Optical micrograph of cross sections of a Si-SLID-Si sample (top) and Si-SLID-PZT sample (bottom).

Scanning acoustic microscopy (SAM): Scanning acoustic microscopy uses acoustic waves to create visual images of variations in the mechanical properties of samples. This technique is frequently used in microelectronic packaging as an efficient non-destructive testing (NDT) technique where internal defects can be visualized in two and three dimensions. Bertocci et al. [17] provided a detailed overview of the characterization technique and demonstrated the use of SAM to detect defects during the fabrication of ultrasound transducers.

The three main scanning modes are A-, B-, and C-scans. An A-scan measures the reflected signal at a single point in pulse-echo mode. B-scans are similar to A-scans, but now a line scan is performed across the width of the sample instead of a single point. C-scans make scans through the sample of planes parallel to the top surface.

There are some limitations to SAM. Imaging edges in the sample is challenging due to distortions of the reflections from edges. An acoustic coupling medium, typically de-ionized water, is needed for the propagation of the acoustic wave back- and forth between the transducer and the sample. Resolution can be improved by using high-frequency transducers, but at the cost of reduced penetration depth [18].

SAM was frequently used in this work for inspection of bonding layers. The capability to detect improper bonds was verified, and one approach of verification is shown in Figure 2-4. Two PZT layers were bonded using metallurgical Au-Sn-solder bonding, where the bond was intentionally made uneven. A SAM C-scan of the sample revealed different contrasts in sections across the sample, suggesting poor bonding. The diced cross-section of the sample was inspected using an optical microscope. Optical micrographs captured across the width of the sample corresponded to sections of imperfect bonding outlined by the SAM micrograph.

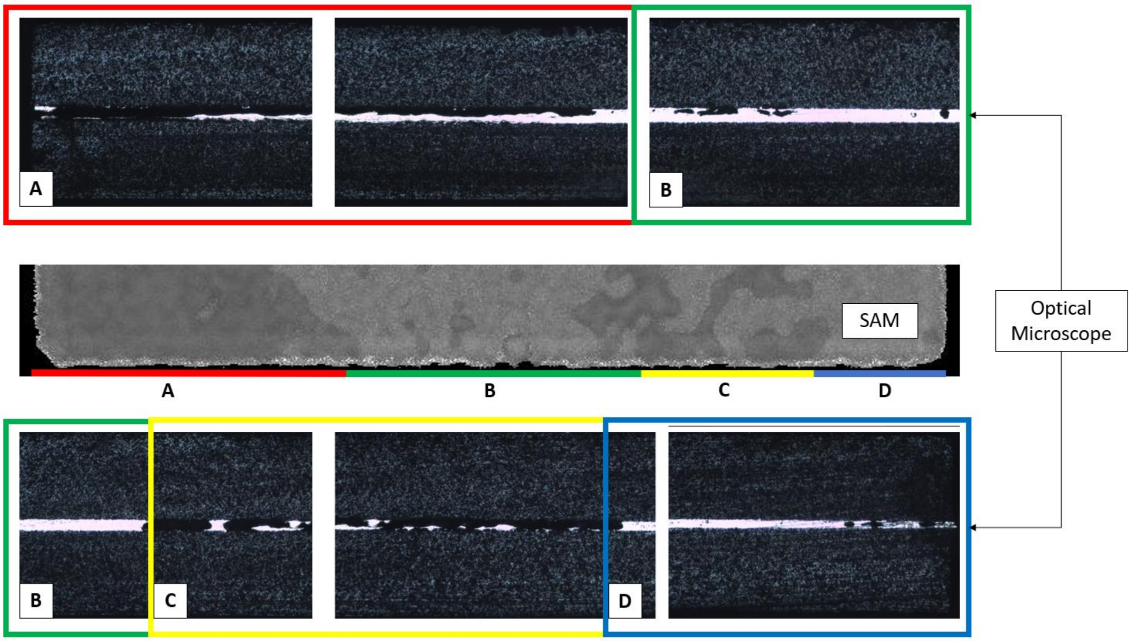


Figure 2-4 – SAM C-scan of two PZT layers bonded using metallurgical Au-Sn-solder bonding where optical micrographs of the cross-section are referenced on the SAM micrograph.

Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDX): Scanning electron microscopy is an imaging technique which forms micrographs of a sample by scanning the surface with a focused electron beam. Electrons accelerate towards the sample and interact with atoms, which produce various signals that carry information about the atomic structure and topography of the sample. The electron beam can remove inner-shell electrons from the sample, causing higher-energy electrons to fill the shell and release energy in the form of characteristic X-rays. Energy-dispersive X-ray spectroscopy measures this energy which contains information about the elemental composition in the sample and maps their distribution.

In this thesis, SEM with EDX was used to study the composition of the metallurgical bond to determine intermetallic phases. Figure 2-5 shows an SEM micrograph of the cross-section of a PZT layer bonded to a Si substrate using the SLID technique. The five points distributed over the sample cross-sections are locations where EDX was performed. The contrast in the SEM micrograph was modified for presentation purposes.

Point	Location
1	PZT
2	Gold
3 & 4	Intermetallic Compound
5	Silicon

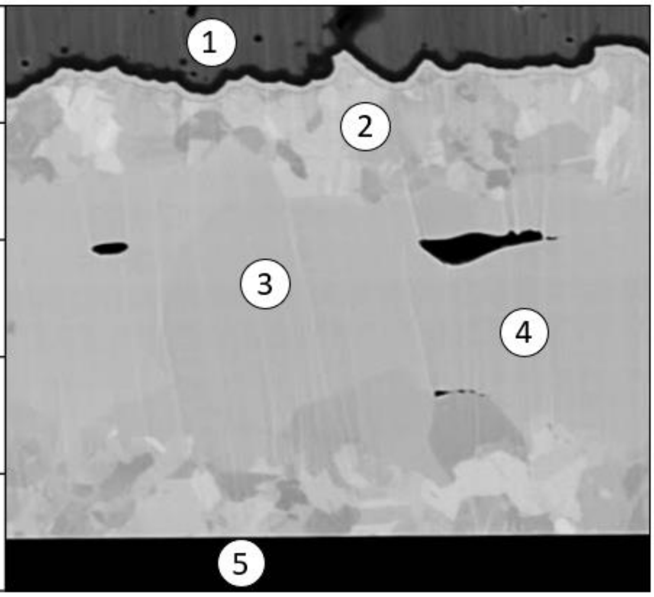


Figure 2-5 - SEM micrograph of the cross-section of a PZT layer bonded to a Si substrate using the SLID technique. The five points distributed over the sample cross-sections are locations where EDX was performed.

Electrical impedance measurements: This measurement technique is a quick, non-destructive characterization method which yields useful information about the performance of the transducer. Electrical impedance measurements are performed to evaluate the electromechanical response of the transducer, for control during fabrication, comparison towards theoretical design and for electronics system integration. Measurements are often performed in air during the fabrication phase and later in water or similar fluids. Most of the impedance measurements performed in this work were done in air, hence this introduction will be limited to measurements under air-loading. The same principles apply to measurements under water loading, however, in such cases the radiation impedance of water loading will influence the measurement. Measurement of the electrical impedance as a function of frequency is done using an impedance analyzer or a vector network analyzer. An electrical representation of the measurement circuit is shown in Figure 2-6A, where an AC source of known Voltage V_0 and a resistor of known resistance R is connected in series with the transducer of impedance Z to be measured. Calibration of the measurement equipment against precision resistors, usually 50 Ohm, is often performed prior to measurement to compensate for electrical cables [8].

Measurement of the impedance magnitude |Z| of a piezoelectric transducer in air determines the resonance frequency, f_r and antiresonance frequency, f_a. From these two frequency points, the effective dynamic coupling coefficient is given by

$$K_{eff}^2 = 1 - \left({f_r}/{f_a} \right). \quad (2.5)$$

In the Van Dyke equivalent circuit, also known as the Butterworth-Van Dyke circuit, shown in Figure 2-6B, all elements of the transducer are represented by electrical components. In the equivalent circuit in Figure 2-6C, the transducer is separated into sections representing the electrical, mechanical, and acoustical parts. By measurement of free capacitance and dissipation factor at low frequencies well below resonance, combined with the measurement of impedance magnitude around resonance, all parameters of the Van Dyke equivalent circuit can be calculated. Furthermore, calculation of all parameters of the equivalent circuit in Figure 2-6C is possible through a coupled set of relations, presented in [8: Ch.12]. This demonstrates the usefulness of impedance measurements as a tool in transducer development.

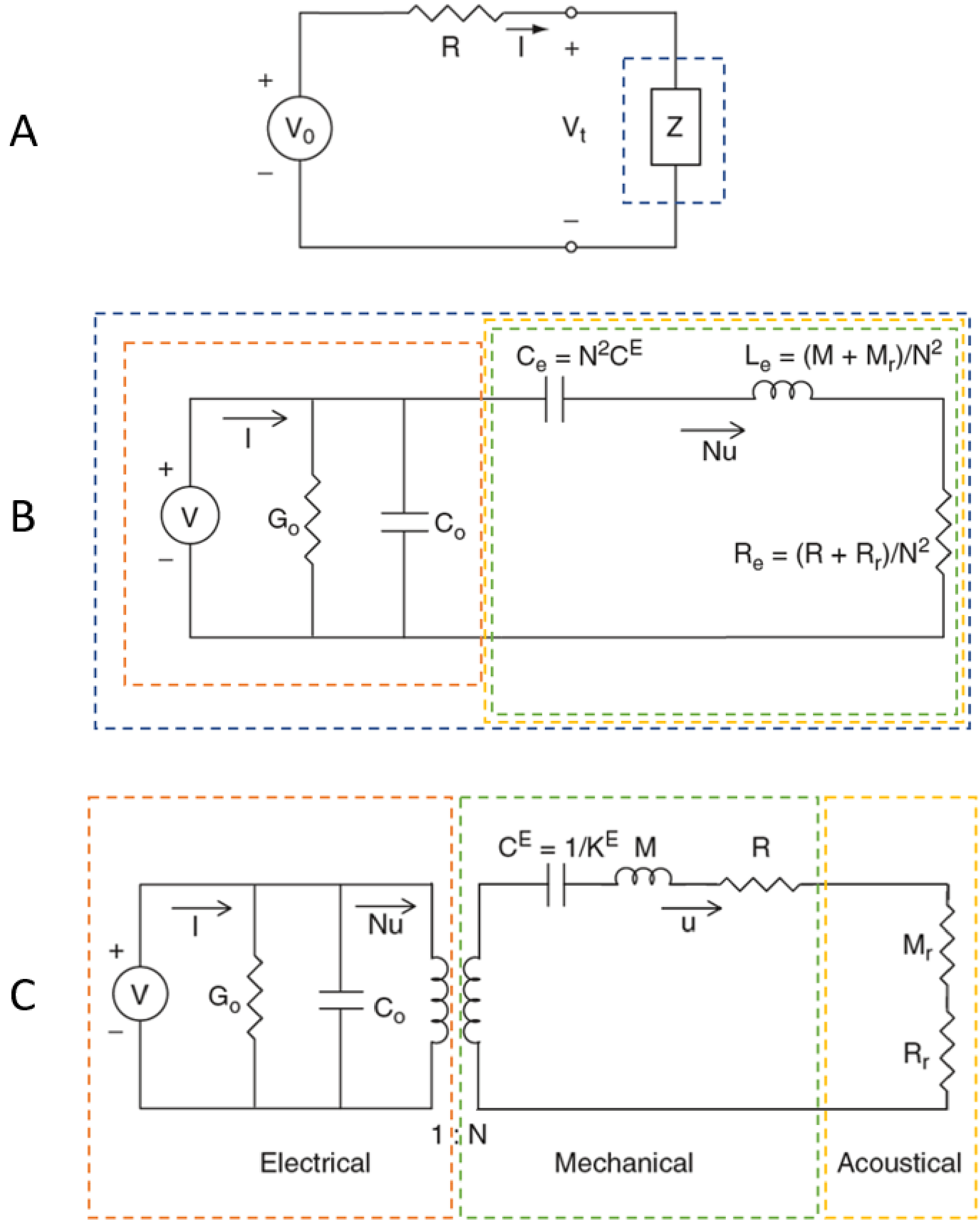


Figure 2-6 – The electrical impedance measurement circuit is shown in A. Van Dyke representation of a transducer is shown in B. In the equivalent circuit shown in C, sections representing the electrical, mechanical, and acoustical parts are separated. Figure adapted from [8] and reprinted with permission.

Acoustic performance measurements: Acoustic measurements are essential in the evaluation of the performance of ultrasound transducers, and common measurement techniques include pulse-echo and hydrophone measurements. The samples produced in this thesis were limited to inspection of the bonding layers. In Paper 3, the electroacoustic transfer function was evaluated through simulations of transducers comprised of polymer bonds and metallurgical bonds. However, acoustic performance measurements were not performed physically. Interested readers are referred to Paper 8. In this paper, sensitive single element doppler transducers were developed and measured acoustically by hydrophone and pulse echo measurements.

3 Bonding Techniques for Ultrasound Transducers

This chapter includes background information on bonding, a central topic of the thesis. Some of the information presented in this chapter is not included in the published articles but may serve as useful information for interested readers. In Chapter 3.1, an overview of the relevant bonding techniques is presented. In Chapter 3.2, the micro-scale mechanisms responsible for bond formations are discussed. Chapter 3.3 is about polymer materials with a focus on epoxy adhesives. Chapter 3.4 discusses the metallurgical bonding techniques explored in this thesis. A summary of the central bonding techniques evaluated in this thesis is presented in chapter 3.5.

Bonding is used consistently in this thesis to refer to the process of coupling two layers mechanically and acoustically. The primary function of any bonding layer is to hold substrates together for the life of the product. Secondary functions are also possible and may in general cases be such as sealers, vibration dampers, insulators, and gap fillers [3]. In the context of ultrasound transducers, secondary functions, which may also introduce challenges, include acoustic matching, electrical conduction, heat transport, and serving as an absorber for thermally induced stresses. Most importantly, the bond should be stable and keep the acoustic front- and back-matching layers in contact with the piezoelectric through the lifetime of the transducer.

For successful bonding, it is important that the coupling medium spreads out and coats the entire adherend surface. This is known as wetting and is achieved when the surface tension of the liquid is lower than the surface tension of the adherend. Factors that influence the wettability of the adherend layer include surface roughness and preparation, and bonding process parameters [3].

The surface roughness of the PZT is determined by the grain size and is for most conventional PZT ceramics in the range of 3 to 5 µm [19]. Single crystal ferroelectric materials, such as PMN-PT, can be polished to a finer surface finish of >1 µm, comparable to that of Si.

Piezoelectric materials need electrodes to convert between electrical and mechanical energy. The placement of the electrodes depends on the polarization direction. The PZT materials used in this thesis were all polarized in the 3-direction and excited electrically in the same direction. This means electrodes of the PZT and matching layers, where relevant, are in direct contact with the bonding layer. One must therefore also consider the adhesive strength between the electrodes and the PZT. Electrode deposition was explained in Chapter 2.5. It is critical that the electrode adheres well to the adherends and that the electrode composition is suited for the selected bonding material and bonding process.

3.1 Fabrication Techniques

As the process-flow in Figure 2-2 illustrates, transducer fabrication involves multiple steps, with each step being a complex process. There are many possible techniques for bonding, and each case requires consideration of the materials involved, the operation environment of the transducer, available equipment, and cost.

Bonding can be divided into three classes which depend on the structure of the bonding layer during operation. Only solid coupling is considered in this thesis. Interested readers are referred to [20] and [21] for extensive reviews of bonding techniques, piezoelectric materials, and transducer designs for high temperature operation.

Dry coupling: High pressures clamp the layers together during operation.
Liquid coupling: The bond is liquid during operation.
Solid coupling: The bond is solid during operation.

Common solid-coupling bonding techniques can be grouped into three categories, as illustrated in Figure 3-1. These categories differ in how the bond between the piezoelectric or any layer of the transducer is formed. This thesis considers the two latter bonding categories, in which a bond is formed through the application of a separate layer.

Some polymer-based materials may be applied (cast) on top of the piezoelectric layer where it hardens (cures) and bonds to the piezoelectric material. A mould may be used to obtain the desired thickness of the polymer layer. It is also common practice to adjust the layer thickness by grinding after the layer has cured.

Materials which are not castable have to be bonded through a separate fabrication step. The relevant techniques for this thesis are bonding using polymeric epoxy adhesives and metallurgical techniques.

Bonding using epoxy adhesives relies on the adhesion through a polymer layer. The epoxy is distributed over the mating surfaces and cured through the application of force and heat. It is important that layers are clean prior to bonding. The layer thickness and surface finish may be adjusted by grinding and polishing before and/or after bonding. This technique is well established.

Metallurgical bonding in ultrasound is less common. Bonds formed through metallurgical processes may require additional metallization steps, which depend on the bonding method and metal-systems involved. The application of force and temperature is critical to form successful bonds between the mating surfaces. Some piezoelectric materials experience depolarization due to the high bonding temperatures and in these cases repolarization will be needed.

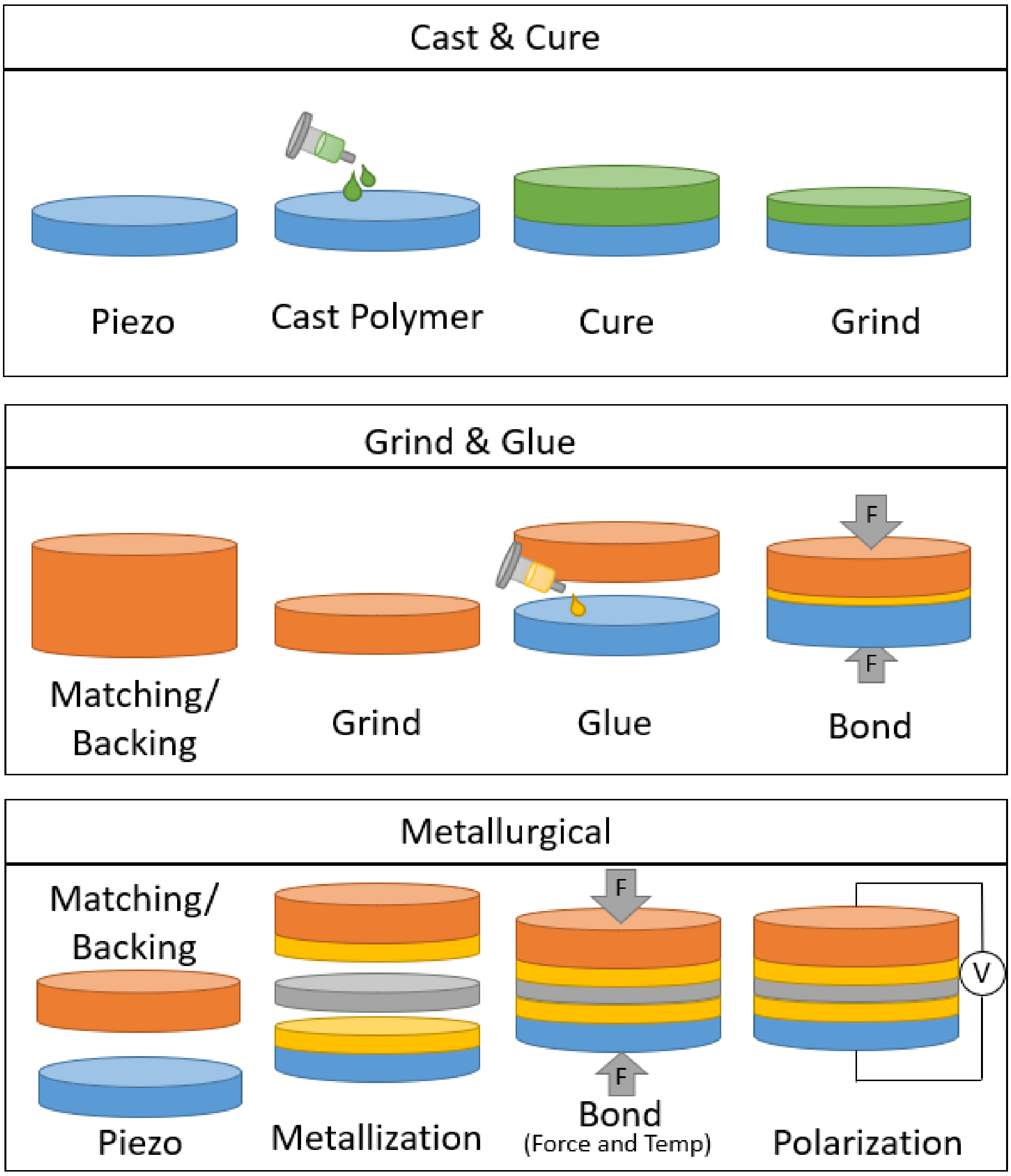


Figure 3-1 – Illustration of central process steps in the common solid-coupling bonding techniques.

3.2 Microscale Mechanisms

Cohesive strength describes the stability of the bonding material itself, while adhesive strength relates to coupling at the interface between the adherend layer and the bonding layer, as illustrated in Figure 3-2. Adhesive- and cohesive strengths are the result of forces existing between atoms or molecules. These forces arise due to unlike charge attractions between atoms or molecules and are commonly referred to as short-range forces and long-range forces, or primary forces and secondary forces, respectively [3: Ch.2].

Primary/ Short Range:

- Covalent: Sharing of valence electrons between atoms.
- Ionic: Electrostatic attraction between two ions of opposite charge due to transfer of valence electrons.
- Electrostatic: Similar to ionic bonds but between molecules.
- Metallic: Electrostatic attraction between positively charged metal atoms (cations) and free electrons.

Secondary/ Long Range/ Van der Waals forces:

- Dispersion: Intermolecular forces acting between atoms and molecules which are electrically symmetric but get induced dipoles due to electrostatic attraction to neighbouring atoms or molecules.
- Polar: Like dispersion but regards atoms and molecules with permanent dipoles.
- Hydrogen Bonds: Electrostatic attraction between a hydrogen atom which is covalently bonded to two electronegative atoms (typically oxygen, nitrogen, fluorine) from two separate molecules.

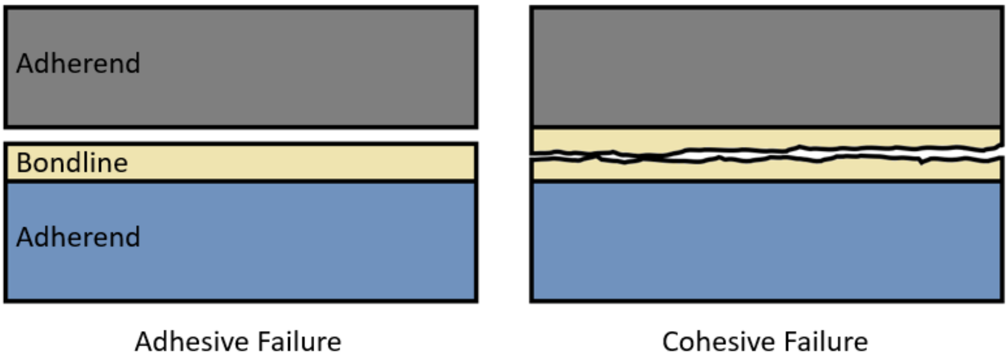


Figure 3-2 – Illustration of adhesive- and cohesive strength

There are several theories for adhesion which all differ in how the mechanism of adhesion is formed. Which theory applies depends on the adhesive system used and the materials involved [3: Ch.2]. The most relevant adhesion theories are summarized here:

Adsorption theory: Adhesion is a result of adsorption of adhesive molecules onto an adherend surface which results in attractive secondary forces between molecules. Intimate contact is needed for these forces to develop and is achieved through proper wetting. Bonds between epoxy adhesives and metallic surfaces are largely attributed to this mechanism [22].

Chemical theory: This theory is similar to adsorption theory but implies the formation of stronger primary bonds, such as covalent, ionic and hydrogen bonds, or Lewis acid-base interactions across the interface.

Mechanical theory: Increasing the contact area by roughening the surface will improve adhesive strength. The surface roughness also contributes to increased adhesive strength from the interlocking effect.

Electrostatic theory: Electrostatic forces arise between the adhesive and the adherend.

Diffusion theory: Adhesion arises through the diffusion of molecules or atoms in the adhesive and adherend.

Weak boundary layer theory: A boundary layer forms at the interface between the adhesive and adherend surface. Such layer may develop from the adhesive, the adherend, environmental exposure, impurities, or a combination of any of the four.

3.3 Polymer Adhesives

Adhesives are all around us and have been utilized for thousands of years. Early hunters used beeswax to glue feathers to their arrows to improve accuracy, and prehistoric civilizations would repair broken pottery with tree sap. The science of adhesives has evolved since then, and in the present day, we have polymer adhesives which can be tailored to most applications and intricate designs.

Polymer materials are common in ultrasound transducers. Piezoelectric composites combine piezoelectric ceramic separated by a polymeric filler. Acoustic matching and backing layers are often based on polymers, where the acoustic properties can be tuned by fillers. Acoustic lenses of silicone are another common implementation of polymers in ultrasound transducers. Bonding using polymer adhesives is the most common technique for bonding layers in acoustic transducers.

A wide variety of polymer adhesives exist. For brevity, the introduction to polymer adhesives is limited to epoxies. Epoxies primarily consist of an epoxy resin and curing agent. Single-component epoxies are a mix of resin and curing agent, whereas two-component epoxies require mixing prior to application. Curing may be performed at room temperature or at elevated temperatures. The cured epoxies have thermosetting cross-linked networks of macromolecules [23]. Thermoset polymers do not melt but become softer at high temperatures.

Epoxy adhesives offer good wetting characteristics and bond well to glass, metals, ceramics and certain plastics. Epoxies are of the most versatile family of adhesives as properties of the cured epoxy can be modified by [3: Ch.9 & 10]:

1. Additions of organic and inorganic fillers and components to tune the electrical, thermal, and mechanical properties. Epoxies are inherently not electrically conductive with poor thermal conductivity. This allows epoxies to function as electrical and thermal insulators. The addition of silver particles can make electrically conductive epoxies, while boron nitride fillers may be used to improve thermal conduction. As mentioned above, tuning properties of polymer-based composites by incorporating filler particles is common practice in transducer fabrication.
2. Selection of epoxy resin or combination of multiple epoxy resins.
3. Selection and concentration of curing agent as well as curing parameters such as time and temperature. Curing at elevated temperatures generally increases the glass transition temperature and degree of crosslinking in the cured epoxy.

The two epoxies explored in this thesis are listed in Table 3-1. The motivation for using polymer adhesives was mainly to compare the bonding process, and resulting bond line performance, to that of metallurgical bonding.

Table 3-1 – Overview of the polymer epoxies used in this thesis.

Tradename	Manufacturer	Temperature Range	Form	Resin/Curing Agent	Datasheet
Duralco 4460	Cotronics	High temperatures	Two-component, liquid	Epoxy/Anhydride	[24]
DP460	3M	Moderate temperatures	Two-component, liquid	Epoxy/Amine	[25]

3.4 Metallurgical Bonding

The history of metallurgical bonding is fascinating. The bronze age is a historical period characterized by the use of bronze, an intermetallic formed by mixing copper and tin or certain other metals. Since bronze is harder and more resistant to wear than copper, new tools and weapons were made possible by the discovery of this intermetallic. Recent excavations in Serbia recovered a piece of bronze foil which is estimated to be approximately 6500 years old and is the earliest known tin bronze artifact to date [26]. It is also around the Bronze Age when we have the first trace of solder bonding using tin to create jewellery and other decorative pieces. Now, some thousand years later, we can rely on the same principles for bonding layers in an ultrasound transducer.

Diffusion bonding can be performed in either solid or liquid state. Solid-state bonding often requires long processing time under high pressure, while liquid bonding requires high temperatures to melt the metals. Liquid bonding is often faster than solid-state bonding. A combination of solid and liquid state interdiffusion exists, referred to in literature as solid-liquid interdiffusion (SLID) [27], and transient liquid phase bonding (TLP) [28].

The SLID bonding process involves two metals, one with high and one with low melting temperature (T_{high} and T_{low}). Processing temperature is above T_{low} and below T_{high}. The melting of the T_{low}-metal allows a relatively fast diffusion process, yielding intermetallic compounds (IMCs) that are stable at temperatures above the processing temperature. The SLID bonding technique offers a well-defined, metallurgical bond with excellent mechanical strength and electrical conductivity, well suited for interconnections in high temperature electronics [29]. The high melting temperature of the IMCs makes the method suitable for operation in high temperature environments. In addition, the high characteristic acoustic impedance of the metallurgical bonding layer is acoustically beneficial compared to the softer polymeric bonds.

Soldering is performed by melting a metal alloy between two mating surfaces. This is a reflowable process, i.e., the bonding layer will melt if reheated to the temperature at which the soldering was performed. Soldering is a wide term, including techniques where the metallurgical solder composition is applied in different forms, e.g., as a string, a thin-film, a paste or a preform. Preform-soldering utilizes a shaped thin sheet of the solder-metal composition sandwiched between the mating surfaces.

SLID and soldering can be done by a wide variety of metal systems. This work was focused on the binary metal system of gold (Au) and tin (Sn). The metallization can be achieved through direct deposition of metals on mating surfaces or by application of paste or preform of eutectic composition. The Au-Sn metallurgical system was selected for these reasons:

1. USN has expertise in Au-Sn SLID, where developed bonding processes produce uniform bonds.
2. Bonds are mechanically strong, ductile, and stable up to high temperatures.
3. Minimal oxide formation on Au surfaces.
4. Preforms of eutectic composition are available.

A eutectic AuSn preform (Indalloy 182 ribbon, Indium Corp., USA) of 25 µm thickness was used for all metallurgical bonds produced in this work. This commercially available preform has the composition 80 wt% Au – 20 wt% Sn, which corresponds to 71 at% Au – 29 at% Sn.

Figure 3-3 shows the Au-Sn phase diagram, where the red solid indicator illustrates the SLID process, while the blue dashed indicator illustrates the soldering process. The general approaches of SLID and soldering used in this work are described here:

SLID:

1. Heat up to the melting temperature of the preform.
2. The diffusion between Au and Sn depends on time and temperature, so the bonding process requires some holding time at the bonding temperature. This diffusion process dilutes the concentration of Sn, such that the overall Sn concentration in the developed bond is less than that of the eutectic preform.
3. Cooling.

Soldering:

1. Heat up to the melting temperature of the preform.
2. Cooling.

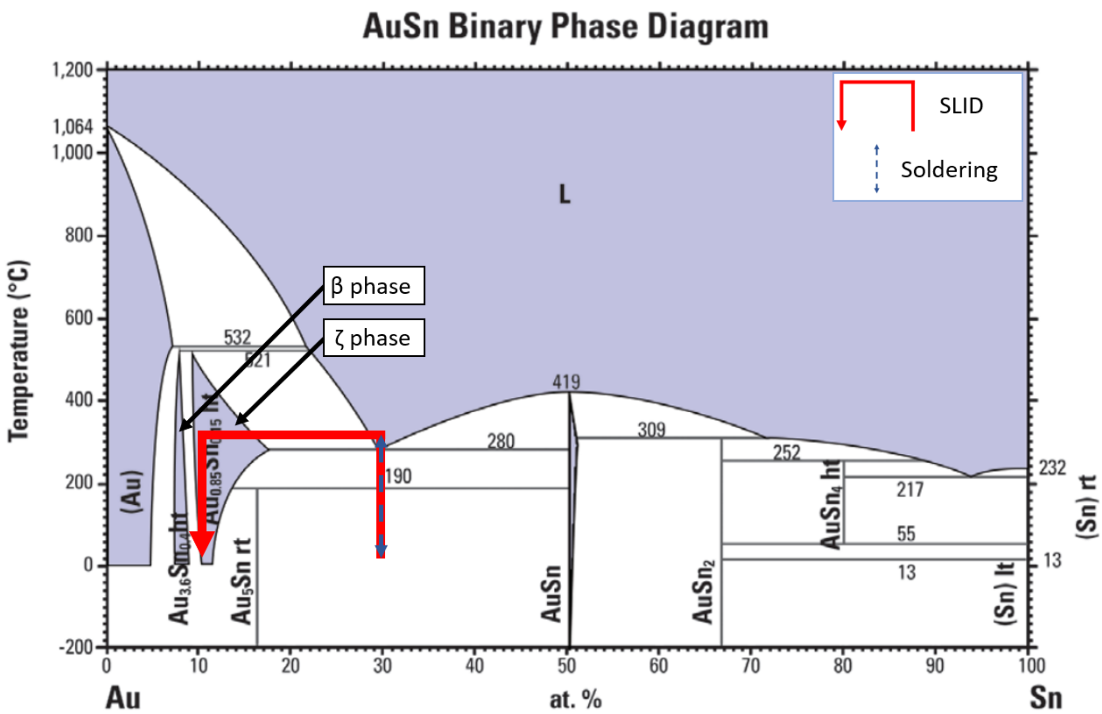


Figure 3-3 – Au-Sn phase diagram adapted from [30] and reprinted with permission.

Figure 3-3 shows that the main conceptual difference between soldering and SLID is the diffusion process, in which SLID forms IMCs and soldering does not. Thin intermetallic layers may be formed through the soldering process if nm-thick Au electrodes are used. However, most of the bonding layer will still be of a composition at or close to the eutectic composition, such that the bond is still reflowable.

Bonds formed through the SLID process are not reflowable since this process targets uniform phases of low Sn concentration. The target phase of the intermetallic after bonding can be achieved by depositing an Au layer of a specific thickness. The ζ-phase is often desired as this phase has a high melting temperature with adequate ductility. If an excess of gold is left after bonding, high temperature storage will induce further diffusion of the bond and a β-phase may be reached. This phase has a higher melting temperature than the ζ-phase but is reported to be stiffer [29]. Figure 3-4 shows the calculated required Au thickness to reach the ζ-phase (red line) and β-phase (blue line) if a preform is used with the composition 71 at% Au – 29 at% Sn. Note that the Au thickness on the y-axis is the total Au thickness. Hence, if both mating surfaces are plated with Au layers of equal thickness, it is the combined Au thickness of the two layers that is relevant. Also, a force is often applied to the stack which causes the liquified preform to squeeze out, reducing the effective thickness of the preform. The Au thicknesses in Figure 3-4 were calculated from the number of moles of Au and Sn in the preform and substrate where the target composition of Sn was set to 11.1 at% for the ζ-phase and 9 at% for the β-phase.

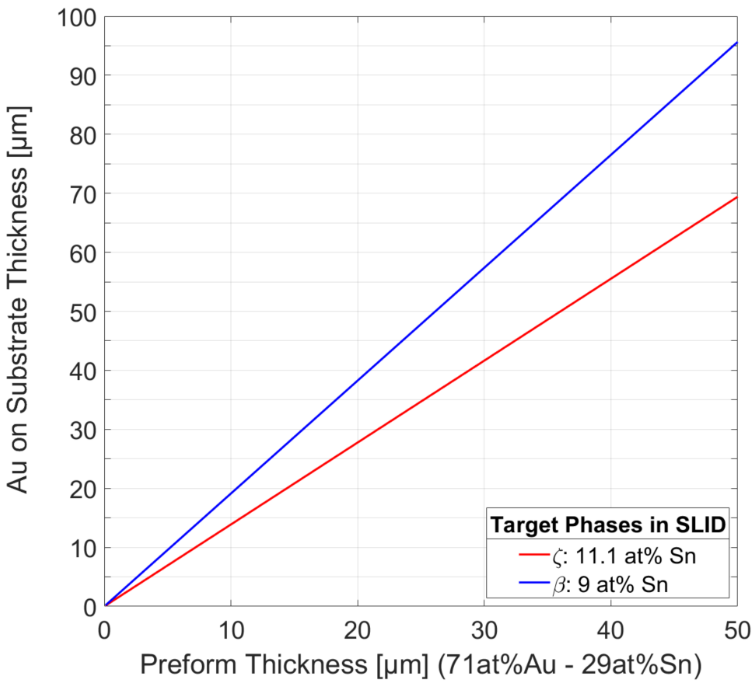


Figure 3-4 – Calculated Au thickness on substrate relative to preform thickness of composition 71 at% Au – 29 at% Sn.

3.5 Polymer Adhesives vs SLID vs Soldering

Table 3-2 – Summary of polymer adhesives, SLID and soldering.

	Polymer Adhesives	SLID	Soldering
Acoustic Properties	The characteristic acoustic impedance (Z_a) of polymer adhesives is generally in the range of 2 MRayls to 4 MRayls and therefore not well matched to piezoelectric ceramics. Z_a may be tuned by incorporation of filler particles.	Z_{aco} of metals is generally high. Au - Sn (80 wt% - 20 wt%) intermetallics have Z_a of approximately 20 MRayls. This provides good acoustic matching to piezoelectric ceramics and relaxed constraints on bond line thickness and uniformity. Voids in the bonding layer will effectively reduce Z_a.	
Mechanical Properties and temperature stability	Mechanical properties are dependent on the polymer system used. Epoxies are generally sufficiently strong at room temperature but degrade at elevated temperatures.	Mechanical properties are generally quite strong and stable at high temperatures. Soldering bonds are reflowable, while SLID bonds offer greater temperature stability with melting temperatures above the processing temperature.	
Conductive Properties	Epoxies are inherently not electrically conductive. Incorporation of electrically conductive particles is possible. Thermal conductive properties are generally low but may be increased by incorporation of metal particles or boron nitride particles.	Metallurgical bonds are electrically and thermally conductive.	
Fabrication	Fabrication is easy and does not require advanced equipment. Some high-temperature polymer adhesives require long curing at elevated temperatures.	SLID bonding requires µm-thick layers of the high melting temperature metal, hence, electroplating may be necessary. This step is not needed for soldering. Bonding is often performed in vacuum. Annealing steps prior to- and post- bonding may improve bond strength.	
Cost	Generally low, however, high-temperature epoxies are often more expensive than standard epoxies.	The price is dependent on the metallurgical system used. Metallurgical preforms are more expensive than polymers. SLID is considered more expensive than soldering due to the required excess of T_{high}-metal.	

4 Ultrasound Transducers in Harsh Environments

4.1 High Temperature

Ultrasound transducers need to be designed with consideration of the operating environment. Temperature dependency is common for all materials, and the most commonly used material groups found in ultrasound transducers are discussed here.

All polymeric materials degrade to some extent by exposure to high temperatures. The degrading mechanisms can weaken the polymer both cohesively and adhesively. Long exposure to high temperatures can have the following effects on polymer materials [3: Ch.17]:

1. Chain scission: Splitting of polymer molecules causes the bond to become more brittle and reduces the cohesive strength of the bond.

2. Crosslinking of polymer molecules which were not crosslinked during curing. This may result in shrinkage and make the bonding layer more brittle.

3. Evaporation of plasticizer, which makes the polymer more brittle

4. Oxidation: If oxygen or metals are present, weak boundary layers may form as a result of oxidation, which reduces cohesive strength.

Mechanical and electrical properties of metals and metal alloys are also temperature dependent. Properties such as elastic moduli, tensile strength and yield strength decrease with increasing temperature. Microstructural changes can occur in metals heated below their melting temperature. A good example of this is annealing, which is especially relevant for metallurgical bonding, since this process may relieve residual stresses. [31]

Piezoelectric materials are ferroelectric and have an associated Curie temperature at which the material phase changes from ferroelectric to paraelectric and the PZT material will be depolarized. Since elastic, piezoelectric and dielectric properties depend on the proximity to the Curie temperature, the maximum recommended operating temperature of piezoelectric materials is, as a rule of thumb, often set to half of the Curie temperature [8].

Differences in the coefficient of thermal expansion between layers result in residual stress during thermal expansion and contraction. Thermal conductivity will also influence residual stress due to uneven temperature distribution in the bonding layer. Making the bond thin can help uneven temperature distribution [3].

Voids, which are inclusions of gas or vacuum, may be an issue at low temperatures. The modulus of elasticity generally increases with decreasing temperature. Bonds which may readily relieve stress at room temperature, may no longer be able to relieve concentrated stresses at low temperatures due to increase in the modulus of elasticity [3].

4.2 High Pressure

The effects of pressure are generally dependent on the direction of stress relative to the direction of polarization. These effects are best described in the work by Berlincourt and Krueger [32] and Nishi and Brown [33], with a summary given here.

Application of directional compressive stress reorients the polar axes of domains to the direction perpendicular to the applied stress, as illustrated in Figure 4-1.

> Compressive stress parallel to the polarization direction reorients non-perfectly aligned polar axes perpendicular to the direction of compressive stress.
>
> Two-dimensional compressive stress perpendicular to the polarization direction reorients non-perfectly aligned polar axes perpendicular to the direction of compressive stress, i.e. parallel to the polarization direction.

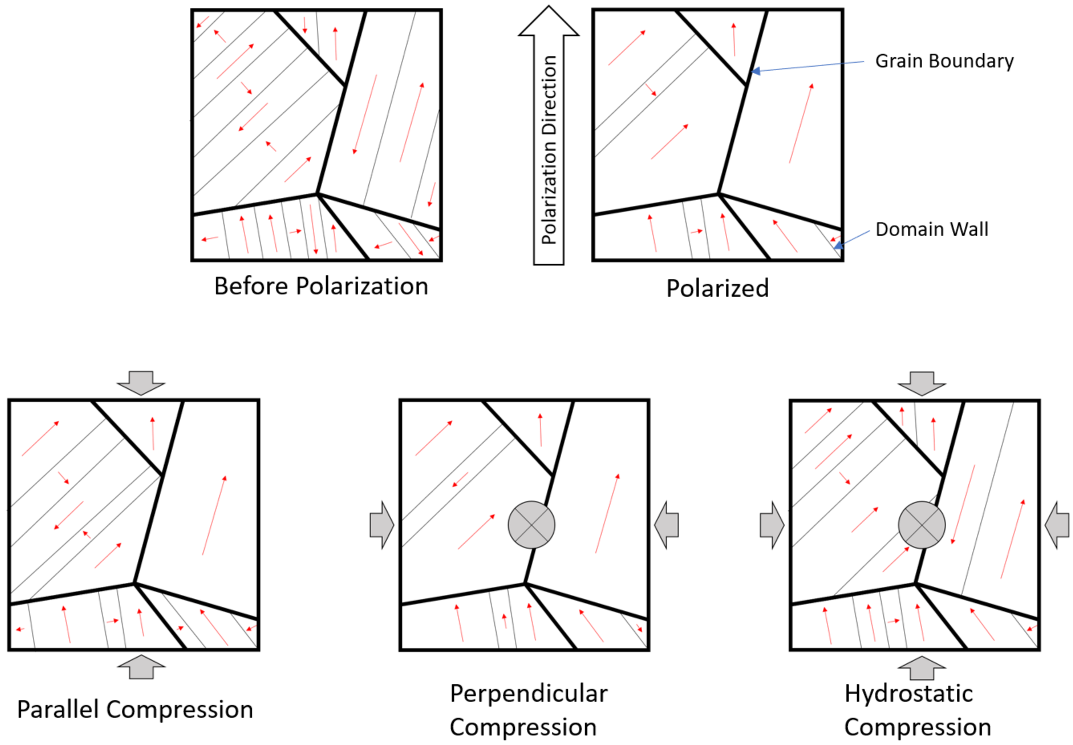


Figure 4-1 – Illustration of the domain orientation in a piezoelectric ceramic material before polarization (top left), after polarization (top right), under compression parallel to the polarization direction (bottom left), under two-dimensional compression perpendicular to the polarization direction (bottom middle), and under hydrostatic compression (bottom right).

In perovskites, the permittivity of domains oriented perpendicular to the direction of measurement is greater than the permittivity of domains oriented parallel to the measurement direction. Thus, permittivity increases due to compressive stress parallel to the polarization direction, and conversely, permittivity decreases due to compressive stress perpendicular to the polarization direction.

The piezoelectric effect is less affected for both scenarios of perpendicular and parallel compression. The reoriented domains due to two-dimensional perpendicular stress are parallel to the polarization direction, however, the orientation is random such that the net contribution to the piezoelectric effect is negligible. With applied stress parallel to the polarization direction, the reoriented domains will no longer contribute to the piezoelectric effect. However, domain reorientation induces an increase in domain walls, which contribute positively to the piezoelectric effect and thereby counters the mentioned reduction from domain reorientation. In addition, the increased number of domain walls increase permittivity further and increase compliance. Domain wall contributions are lossy, which reduces mechanical and electrical quality factors.

Since the stress from hydrostatic compression is isotropic, domain reorientation does not occur. However, the volumetric compression resulting from the hydrostatic compression results in an increase in domain walls. As mentioned, these domain walls contribute positively to the piezoelectric effect, increase permittivity and compliance, and reduce mechanical and electrical quality factors. These effects are mostly non-permanent, meaning that properties are restored to original values when the stress is released. In Paper 2, we observed evidence of hydrostatic pressure-induced depolarization after exposing a piezoelectric material (L-155N, TFT) to hydrostatic pressures up to 1000 bar [Paper 2]. This behaviour was also observed for PMN-PT (67/33) exposed to 4550 bar hydrostatic pressure [34].

Passive materials, such as metals and polymers, are also affected by pressure, yet not to the same extent. The yield strength and elastic moduli of metals and polymers increase under high hydrostatic pressure [35][36].

Birch [37] formulated an expression which states that the elastic modulus (E) of a material under hydrostatic pressure is a function of applied pressure (P) and the material's Poisson's ratio (ν) as

$$E = E(0) + (P * 2(5 - 4\nu)(1 - \nu)) \tag{4.1}$$

Where E(0) is the elastic modulus at atmospheric pressure. This shows that the change in elastic modulus is much more significant for materials with an initial low elastic modulus, e.g., polymers, as compared to metals and ceramics, which have a higher elastic modulus. It should be mentioned that some materials behave non-linearly at pressures greater than the range discussed in this thesis [36].

Influence from voids, cracks and delaminations may be reduced at high pressure as deformation of the surrounding material closes the flaws [38].

5 Estimation of Effective Material Parameters

5.1 Existing Models for Composite Materials

Analytical models for estimation of effective elastic parameters exist and can provide useful estimates. However, the analytical models in general rely on idealized assumptions which limit their accuracy for real composites.

As mentioned in Chapter 2.2, ultrasonic transducers commonly use ordered piezoelectric composites to achieve high electro-mechanical coupling, bandwidth, and sensitivity, with the additional benefit of reduced characteristic acoustic impedance relative to the load medium. Smith and Auld [39] presented expressions for effective medium parameters of ordered 1-3 connectivity piezoelectric composites. Qi and Cao [40] adopted the expressions by Smith and Auld to fit composite materials of ordered 2-2 connectivity. These expressions were formulated for piezoelectric materials but can be adapted to passive materials, as described by Manh et al. [10].

The simplest models for elastic properties of unordered 0-3 connectivity composites are the Reuss model, where constant stress in the solid is assumed, and the Voigt model, where constant strain is assumed [41]. Such composites are described as a random distribution of small particles in a matrix. The Reuss and Voigt models lead to extreme upper and lower bounds for the sound velocity in the composite. The variation between these two bounds can be large for dissimilar materials, limiting their usefulness, while more accurate predictions can be achieved by selecting materials with similar properties. Hashin and Shtrikman [42] presented another approach, providing upper and lower bounds that are within those formulated by Reuss and Voigt. Berryman [43] presented three approximations based on single scattering theory. Devaney and Levine [44] described an approach using a self-contained formulation of multiple scattering theory. The Reuss, Voigt and Hashin and Shtrikman models provide upper and lower boundaries, while the models presented by Berryman and Devaney and Levine provide estimates for effective values for the composite. All the models mentioned assume the inclusions to be spherical, of uniform size, and much smaller than the acoustic wavelength.

For low volume fractions of inclusions, all models behave similarly. However, for higher volume fraction of inclusions, the multiple scattering effects become dominant, making results from the Devaney model differ markedly from results from the other models as the volume fraction of particles increase. Of the mentioned models, the approach by Devaney and Levine has been found to be the most accurate model for estimating effective elastic properties of unordered composite materials [45][46]. In addition, effective medium theory for porous solids exists [47] which matches experimental data well.

A two-dimensional finite element modelling approach to estimate effective elastic properties of composite materials was presented by Gomez Alvarez-Arenas et al. [48]. This approach was found to provide tighter upper and lower bounds than those presented by Hashin and Shtrikman, and the resulting material parameters were found to match well to experimental results.

5.2 Background for New Technique

Reliable estimation of the influence from voids on the acoustic performance is important if metallurgical bonding is to be used in acoustic transducers. Characterization techniques presented in Chapter 2.6 are useful methods for the assessment of bonds. However, these techniques do not provide information about the elastic properties of the bonding layer material. Acoustic characterization techniques, such as pulse echo and through transmission, may be used to obtain elastic material properties. The frequency of the measurement system and the thickness of the sample must be considered in such measurement techniques. If the sample is too thin relative to the acoustic wavelength, the sample will be indistinguishable from the surrounding media.

The Ultrasound Lab at USN has a trough-transmission setup operating at 5 MHz. The SAM instrument in the USN lab is equipped with transducers up to 75 MHz and has a built-in pulse-echo characterization functionality. Calibration against a wide range of known samples is needed in order to provide accurate estimations, and measurement samples should preferably be single layered. Since the bonding layers produced in this work were thin (less than 30 µm) and sandwiched between other materials, the available characterization techniques were insufficient for reliable estimation of elastic properties.

The analytical models mentioned in Chapter 5.1 can provide useful estimates of effective elastic parameters. However, the analytical models in general rely on idealized assumptions which limits their accuracy for real composites. Additionally, these analytical models are limited as they cannot predict the spatial and geometric dependency of the inclusions in the matrix. The FEM study by Gómez Alvarez-Arenas et al. was performed in time domain and was limited to normally incident waves in 2D.

In Paper 1, we present an approach to estimate the elastic properties of composite materials using finite element method (FEM) modelling. The models can handle various composite configurations, including the different connectivity configurations such as 2-2, 1-3, 3-3 and 0-3. The described FEM approach is not limited to requiring a fine composite pitch and can handle dependence on inclusion sizes and orientations.

In addition to the FEM model, a graphical user interface was built for analysis of results and comparison to analytical models where relevant. This user interface is shown in Figure 5-1.

The random 0-3 configurations were created using the Application Builder feature available in COMSOL. The inclusions can be specified with dimensions as spherical or elliptical inclusions, which are either separated or overlapping within a layer. A block diagram of the code for generating a layer of a specified volume fraction of spherical inclusions which do not overlap is shown in Figure 5-2. Example geometries are shown in Figure 5-3, where the geometries are (A) a 2D layer with circular inclusions of random size which do not overlap, (B) a 2D layer elliptical inclusions of random size which do not overlap, and (C) a 3D layer of spherical inclusions of fixed size which do not overlap.

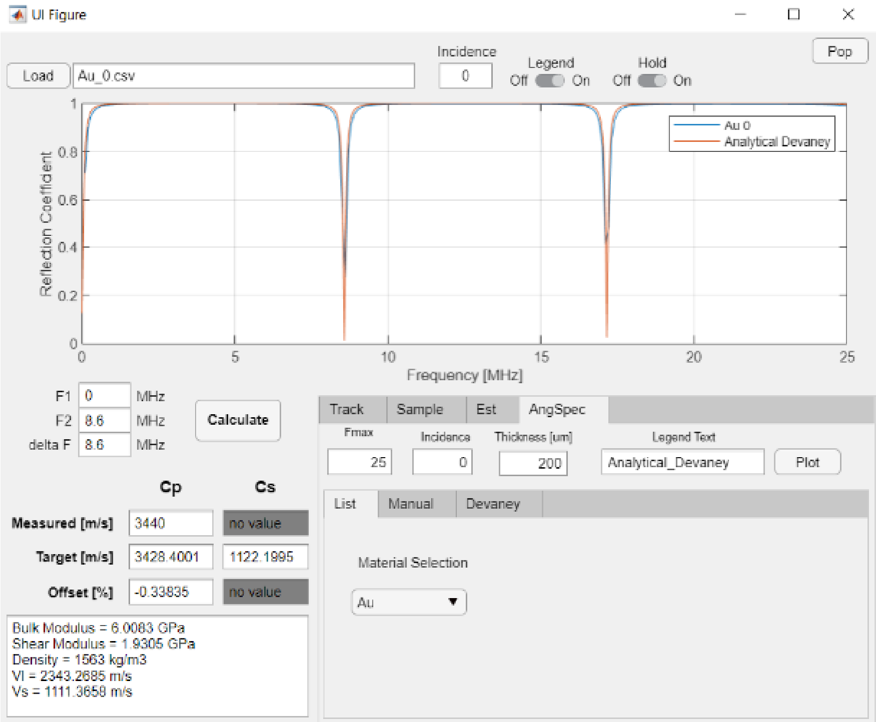


Figure 5-1 – Graphical user interface developed for processing of results.

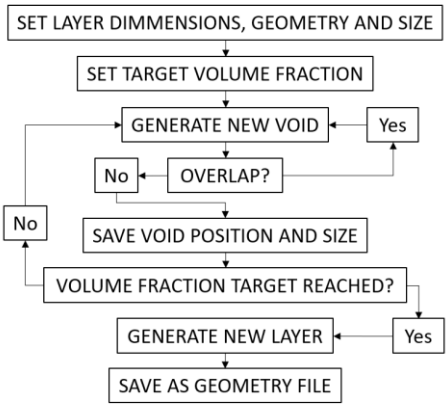
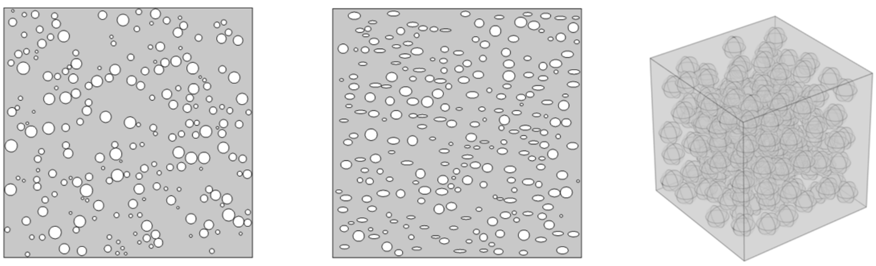


Figure 5-2 - A block diagram of the code for generating a layer of a specified volume fraction of inclusions.



A B C

Figure 5-3 - Example geometries generated for use in the FEM model. (A) A 2D layer with circular inclusions of random size which do not overlap. (B) A 2D layer elliptical inclusions of random size which do not overlap. (C) A 3D layer of spherical inclusions of fixed size which do not overlap.

6 Summary of the Thesis

This section summarizes the articles presented in the thesis and relates their content to the research questions. The research questions are:

1. Can metallurgical bonding be used to bond layers of the acoustic stack?
2. What are the limitations of developed metallurgical bonding techniques?
3. How will high pressure and high temperature influence transducer performance?

6.1 Paper 1

The background and motivation for the work in Paper 1 were discussed in Chapter 5. In Paper 1, we present an approach to estimate the elastic properties of composite materials using finite element method (FEM) modelling. The models can handle various composite configurations, including different connectivity configurations such as 2-2, 1-3, and 0-3. 2D and 3D implementations of the model were described, validated, and applied to estimate composites' compressional and shear wave velocities with different connectivity configurations. The described FEM approach is not limited to requiring a fine composite pitch and can handle dependence on inclusion sizes and orientations.

The main challenge with the metallurgical bonding techniques is the formation of voids in the bonding layer, and the extent of their influence on the acoustic properties was uncertain. This FEM-based modelling approach was developed to evaluate bonding layers containing voids where the influence of void size and orientation can be studied, addressing the second research question.

6.2 Paper 2

Paper 2 addresses all three research questions by evaluating polymer and metallurgical bonding techniques for piezoelectric transducers in harsh environments. The evaluation regards the fabrication processes, shear strength measurements at room temperature and high temperature, high temperature cycling and high hydrostatic pressure testing. Five different sample groups were fabricated and tested; two were polymer epoxies, and three were metallurgical bonds. Of the polymer epoxies, one was a standard epoxy, and the other was a high-temperature epoxy. From the metallurgical groups, two were made with different configurations of Au-Sn SLID and one was made using the Au-Sn pre-form soldering technique.

Metallurgical bonds were more resilient to high temperatures with higher shear strengths compared to the polymer-bonded samples. No significant changes were observed in any of the bonds after exposure to 1000 bar hydrostatic pressure.

6.3 Paper 3

In Paper 3, FEM modelling was used to evaluate and compare the performance of metallurgical Au-Sn SLID bonding and epoxy bonds. This was the initial evaluation where influences from bond thickness, voids and delamination of the bond were studied. This two-dimensional model isolates effects from thickness mode resonance through the application of roller boundary conditions. Despite the idealized boundary conditions applied to the structure, the observed influences from voids and delamination in a metallurgical bonding layer are consistent with succeeding experimental work and more realistic numerical evaluations. Furthermore, the results demonstrate the advantage of using metallurgical bonding in quarter wave resonators consisting of PZT coupled to a de-matching layer of very high acoustic impedance. Hence, research questions 1 and 2 are addressed in this work.

6.4 Paper 4

Elements of Paper 4 are covered by Paper 2; however, some elements are not covered. In this work, Si-SLID-Si samples and Si-SLID-PZT samples were exposed to 1000 bar hydrostatic pressure. The Si-SLID-Si samples were included to evaluate the influence of surface roughness on void formation in the bonding layer. One of the Si-SLID-Si samples showed signs of delamination after repeated exposures to 1000 bar pressure. This work was focused on addressing the first and third research questions.

6.5 Paper 5

In paper 5, an alternative metallurgical bonding technique, preform soldering, was introduced as a simpler alternative to SLID bonding. This bonding technique was used to couple two separate piezoelectric layers, creating one effective layer of reduced resonance frequency, thereby demonstrating alternative use cases for metallurgical bonding in ultrasound transducers. SAM measurements combined with electrical impedance measurements were used to study the influence of voids. This work addresses the first and second research questions.

6.6 Paper 6

Paper 6 covers the complete transducer production cycle of single element transducers, including design, fabrication, and characterization. The transducers discussed in Paper 6 were not designed for operation in harsh environments. However, the work illustrates the complete process of ultrasound transducer development as well as essential elements of a design process which were not covered elsewhere in the thesis, including the design of an electrical matching network for the transducer as well as characterization methods such as pulse-echo and hydrophone measurements.

7 Conclusion

Metallurgical bonding techniques for ultrasound transducers were explored in this thesis. Two different bonding processes were demonstrated: Au-Sn SLID bonding and Au-Sn preform-soldering.

Bond lines produced through metallurgical bonding processes may contain voids. A FEM modelling technique was developed to evaluate the elastic properties of inhomogeneous layers (Paper 1). Voids were shown to influence the acoustic properties of the layer by effectively reducing the characteristic acoustic impedance. FEM was used to model a complete transducer with metallurgical bond lines containing voids. It was demonstrated that the resonance frequency of the transducer would shift down in frequency for increasing concentrations of voids (Paper 3). These findings agreed with experimental measurements of the electrical impedance of two piezoelectric layers bonded together using the Au-Sn soldering technique (Paper 5).

Metallurgical bonding has been demonstrated as a viable solution to bonding layers of the acoustic stack. Bond lines formed through Au-Sn SLID and Au-Sn preform-soldering produce mechanically robust bonds with high-temperature stability and excellent electrical conductivity and thermal conductivity. Au-Sn SLID and Au-Sn preform-soldering bonds between PZT and Si did not indicate significant degradation after exposure to 180 °C at atmospheric pressure and 1000 bar hydrostatic pressure at atmospheric temperature (Paper 2).

8 List of Contributions

Accepted peer-reviewed papers included in the thesis:

1. P. K. Bolstad, M. E. Frijlink, T. Manh and L. Hoff, "Estimating Effective Material Parameters of Inhomogeneous Layers Using Finite Element Method," in *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, vol. 69, no. 12, pp. 3402-3410, Dec. 2022, doi: 10.1109/TUFFC.2022.3220371.
2. P. K. Bolstad, M. Frijlink, and L. Hoff, “Evaluation of bonding techniques for ultrasound transducers,” in *Microelectronics Reliability*, vol. 151, p. 115272, Dec. 2023, doi: 10.1016/j.microrel.2023.115272.

Published proceedings from international conferences:

3. P. K. Bolstad, D. Le-Anh, L. Hoff and T. Manh, "Intermetallic Bonding as an Alternative to Polymeric Adhesives in Ultrasound Transducers," in *2019 IEEE International Ultrasonics Symposium (IUS)*, Glasgow, UK, 2019.
4. P. K. Bolstad, S. L. Kuziora, H. -V. Nguyen, T. Manh, K. E. Aasmundtveit and L. Hoff, "Impact of High Pressures on Au-Sn Solid Liquid Interdiffusion (SLID) Bonds," in *2020 IEEE 8th Electronics System-Integration Technology Conference (ESTC)*, Tønsberg, Norway, 2020.
5. P. K. Bolstad, M. Frijlink and L. Hoff, "Metallurgical AuSn Bonding of Piezoelectric Layers," in *2022 IEEE International Ultrasonics Symposium (IUS)*, Venice, Italy, 2022.
6. P. K. Bolstad, L. Hoff, H. Torp and T. F. Johansen, "Design, Fabrication and Testing Highly Sensitive Single Element Doppler Transducers," in *2018 IEEE International Ultrasonics Symposium (IUS),* Kobe, Japan, 2018.

Additional contributions:

Published conference papers covered by peer-reviewed papers

7. P. K. Bolstad, T. Manh, N. Midtseter, M. Frijlink and L. Hoff, "Ultrasound Transducers for High Pressure Environments Up to 1000 Bar," *2020 IEEE International Ultrasonics Symposium (IUS)*, Las Vegas, NV, USA, 2020.

8. P. K. Bolstad, T. Manh, M. Frijlink and L. Hoff, "Acoustic Characterization of Inhomogenous Layers using Finite Element Method," *2021 IEEE International Ultrasonics Symposium (IUS),* Xi'an, China, 2021.

Co-authored conference papers

9. S. L. Kuziora, H. -V. Nguyen, P. K. Bolstad and K. E. Aasmundtveit, "Bi Behaviour in Au–(In–Bi) SLID Bonding," in *2020 IEEE 8th Electronics System-Integration Technology Conference (ESTC)*, Tønsberg, Norway, 2020.

Presentations at international scientific conferences

1. Poster Presentation: P. K. Bolstad, L. Hoff, H. Torp and T. F. Johansen, "Design, Fabrication and Testing Highly Sensitive Single Element Doppler Transducers," in *2018 IEEE International Ultrasonics Symposium (IUS),* Kobe, Japan, 2018.
2. Poster Presentation: P. K. Bolstad, D. Le-Anh, L. Hoff and T. Manh, "Intermetallic Bonding as an Alternative to Polymeric Adhesives in Ultrasound Transducers," in *2019 IEEE International Ultrasonics Symposium (IUS)*, Glasgow, UK, 2019.
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4. Oral Presentation: P. K. Bolstad, S. L. Kuziora, H. -V. Nguyen, T. Manh, K. E. Aasmundtveit and L. Hoff, "Impact of High Pressures on Au-Sn Solid Liquid Interdiffusion (SLID) Bonds," in *2020 IEEE 8th Electronics System-Integration Technology Conference (ESTC)*, Tønsberg, Norway, 2020.
5. Poster Presentation: P. K. Bolstad, T. Manh, M. Frijlink and L. Hoff, "Acoustic Characterization of Inhomogenous Layers using Finite Element Method," *2021 IEEE International Ultrasonics Symposium (IUS),* Xi'an, China, 2021.
6. Poster Presentation: P. K. Bolstad, M. Frijlink and L. Hoff, "Metallurgical AuSn Bonding of Piezoelectric Layers," in *2022 IEEE International Ultrasonics Symposium (IUS)*, Venice, Italy, 2022.

Presentations at national/regional conferences

1. 42nd Scandinavian Symposium on Physical Acoustics, Geilo, Norway, 2019, Oral Presentation.
2. 44th Scandinavian Symposium on Physical Acoustics, Geilo, Norway, 2021, Oral Presentation.
3. Centre for Innovative Ultrasound Solutions – Spring Conference 2019, Speed Update.
4. Centre for Innovative Ultrasound Solutions – Oil & Gas Fall Conference Online 2020, Oral Presentation.

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