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Silicon and Quartz Microengineering

Processing and Characterisation

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Abstract

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Microengineering has developed a broad range of production techniques to reduce size, increase throughput, and reduce cost of electrical and mechanical devices. The miniaturisation has also entailed entirely new opportunities.

In this work, a piezoresistive silicon sensor measuring mechanical deformation has been designed and fabricated with the help of microengineering. Due to the large variety of used processes, this device can serve as a survey of techniques in this field. Four basic process categories are recognised: additive, subtractive, modifying, and joining methods.

The last category, joining methods, has previously been the least investigated, especially when it comes to compatibility with the other categories. The adaptability of wet chemical etching to established silicon wafer bonding technique has been investigated. Further, phenomena related to oxygen plasma pre-treatment for direct bonding has been investigated by blister bond adhesion tests, X-ray photoelectron spectroscopy, and atomic force microscopy.

Wafer bonding has been adapted to monocrystalline quartz. For wet chemical pre-treatment, characteristics specific for quartz raise obstacles. Problems with limited allowable annealing temperature, low permeability of water released in the bond at annealing, and electrostatic bonding of particles to the quartz surface, have been studied and overcome. The influence of internal bond interfaces on resonators has been investigated.

Chemical polishing of quartz by ammonium bifluoride has been experimentally investigated at high temperatures and concentrations. Chemometrical methods were used to search for optimum conditions giving the lowest surface roughness. These extreme conditions showed no extra advantages.

Adhesion quantification methods for wafer bonding have been comprehensively reviewed, and augmentations have been suggested. The improved techniques' usefulness for three areas of use has been forecasted: general understanding, bonding scheme optimisation, and quality control. It was shown that the quality of measurements of all commonly used methods could be dramatically improved by small means.

Keywords: silicon, quartz, microengineering, microstructure, MEMS, wafer bonding, direct bonding, adhesion quantification

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Och Isagel bad mig ej sjunga.
Det måste jag ändå få göra
när jag av min härdande tunga
om asbest och kisel vill höra.

En visa ur hållfasthetsläran
jag sjöng för den hulkande bruden.
Jag sjöng om den avbrutna äran,
den oreparerbara guden.

Och Isagel slutade gråta
– om gråt nu är värre än annat –
Det var på det tjugonde året
på färden vårt hjärta förbannat.

Harry Martinson, Aniara, sång 81.

List of papers

This thesis for the degree of Doctor of Philosophy in Solid State Electronics comprises the following papers, which will be referred to in the summary by their Roman numerals:

I. High-temperature piezoresistive gauge fabricated on commercially available silicon-on-insulator wafers. Ö. Vallin and Y. Bäcklund. Journal of Micromechanics and Microengineering 10 (2000) 196–199.

II. Silicon fusion bond interfaces resilient to wet anisotropic etchants. J. Köhler, C. Strandman, Ö. Vallin, C. Hedlund and Y. Bäcklund. Journal of Micromechanics and Microengineering 11 (2001) 359-363

III. Radial variations in bond strength for plasma bonded oxidised silicon wafers, K. Jonsson, Ö. Vallin, M. Svedberg, and J. Köhler. In manuscript.

IV. Quartz-to-quartz direct bonding. P. Rangsten, Ö. Vallin, K. Hermansson and Y. Bäcklund. Journal of the Electrochemical Society 146 (1999) 1104-1105.

V. Direct bonded quartz resonators. Ö. Vallin, B. Einefors, C. Hedlund, and G. Thornell. In Proceedings of the 2001 IEEE International Frequency Control Symposium and PDA Exhibition, Seattle, WA, USA.

VI. Polishing of quartz by rapid etching in ammonium bifluoride. Ö. Vallin, U. Lindberg, R. Danielsson, and G. Thornell. Submitted to IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control.

VII. Adhesion quantification methods for wafer bonding. Ö. Vallin, K. Jonsson, and U. Lindberg. Invited and submitted to Materials Science and Engineering R: Reports.

The contribution by the author to the papers included in the thesis is as follows (see Figure 1 for the nomenclature used):

I. Major part of planning, all experimental work, evaluation, and writing.

II. Significant part of planning, experimental work, and evaluation.

III. Part of planning, evaluation and writing, significant part of experimental work

IV. Substantial part of planning, experimental work, evaluation, and writing.

V. Major part of planning and writing, all experimental work and evaluation.

VI. Substantial part of planning, evaluation, and writing, all experimental work.

VII. Substantial part of planning, evaluation and writing.

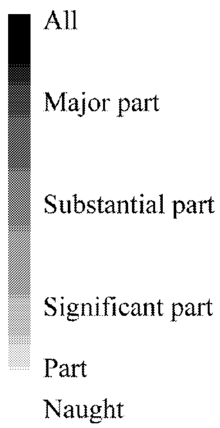


Figure 1. The author's approximate share of work performed.

Contents

Introduction

Microengineering, alternatively known as microstructure technology or microelectromechanical system technology, is quite young as an autonomous discipline within engineering science. In order to bring the research front quickly forward into *terra incognita*, it is tempting, and often necessary, to use a trial and error approach. Tempting, as the unknown new technique gives birth to dreams about solving old and new problems in ingenious ways. Necessary, as quick results are demanded from the financier. With numerous attempts, and a bit of luck, it is often possible to arrive at the vicinity of the pictured aim, and rich rewards and trophies can be brought back and exposed to each and everyone. However, upon inspection of the road to the target it is often found to be winding, bumpy and surrounded by unknown dangers. In order to secure captured land, the road must be properly paved stone by stone starting from known terrain. Not until then, these new results are suitable as starting bases for further exploration.

In the metaphor above, Papers I, II, IV, and V belong to the first type of works, while the other belong to the second type. Both types are important, but it is essential to recognise where they belong.

Starting from general silicon microengineering, the reader is invited to come along on a voyage that will go to the bottom with direct bonding and use it to chart new routes in quartz. During this passage, the surface properties of part of the quartz ocean will be addressed. At last, the final destination will be reached in safe port among solidly built piers of wafer bond adhesion quantification.

In the first sections, ideas and experiences will dominate, while details in the papers will have to wait until the summary of the papers at the end.

Silicon microengineering

Contrary to common machining, the workpiece in microengineering consist of many, maybe thousands of units that are worked on simultaneously. The process involves many steps of different kind. Here, they are categorised in four main classes: additive, subtractive, modifying, and joining methods. Paper I can serve as a survey of the methods used in silicon micro-engineering. For a detailed description of these processes, see for instance references [1,2].

Device manufacturing

In Paper I, the design and manufacturing of a piezoresistive deviation sensor is described. In short, the sensing piezoresistive structure is made from the thin top layer of a silicon-on-insulator (SOI) wafer. Such wafers are commonly employed for electronics, where devices are made in the top layer only. The buried oxide underneath the device layer serves as insulation. To facilitate handling, the SOI structure is supported by a thick substrate wafer. Here, deviation is felt by the bending of a beam fabricated in the substrate wafer. The tension at the surface of the beam's foundation is proportional to the deviation of its tip. In the piezoresistive structure, made from the device layer and placed on the beam foundation, the strain is converted to an electrical signal. To illustrate the variety of methods used in the fabrication, Figure 2, shows schematically a cross section of a device. The process steps used are given in Table I.

In the fabrication of this sensor, all classes of microengineering processes are used, although the joining had been performed in the starting SOI material beforehand. These wafers were manufactured by semiconductor wafer bonding (also known as direct bonding or fusion bonding), lapping and polishing. With semiconductor wafer bonding, two polished wafers can be joined without intermediate layers such as glue or solder [3,4]. The bonding is by no means limited to the semiconductors themselves. Prior to bonding, the wafers are often oxidised, or deposited with thin films of insulators or metals. The bonding process typically consists of a preparation step, where the surfaces are cleaned and made reactive. This is made with wet chemicals, gas plasmas, or atom beams. After that, the wafers are mated, whereupon they immediately stick together. Dispersion forces, or, if water or hydroxyl groups are present, hydrogen bonds, make the wafers adhere. By a thermal treatment, the annealing, the weak bonds originally holding the wafers together are replaced by stronger covalent bonds, over bridging the interface. In vacuum direct bonding, the annealing step sometimes can be omitted as high quality bonding occurs immediately at contacting.

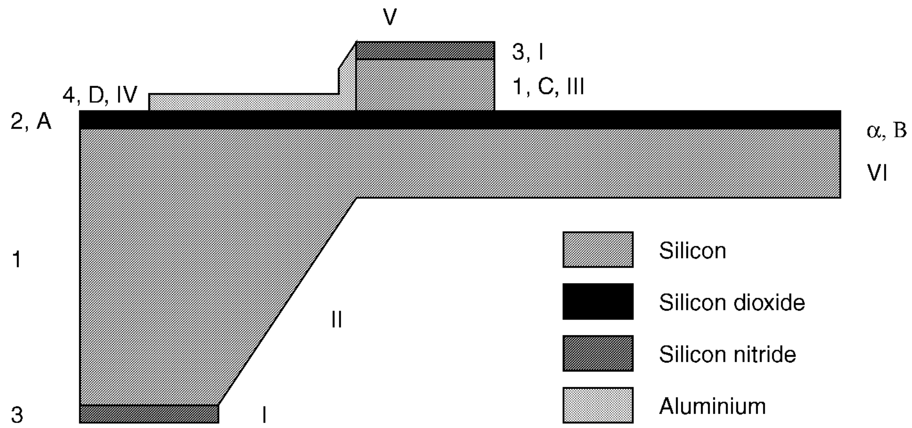


Figure 2. Schematic drawing of a piezoresistive sensor enlightening the variety of different processes used. Legend according to Table I.

Table I. The methods used in the fabrication of the piezoresistive sensor depicted in Figure 2.

Additive	Subtractive	Modifying	Joining
1 Starting material	I RIE CHF_3	A Thermal oxidation	α Direct bonding
2 Thermal oxidation	II KOH	B Bond annealing	
3 LPCVD	III RIE $SF_6 + O_2$	C Implantation	
4 Sputtering	IV Isotropic etching	D Post metal anneal	
	V Lift-off		
	VI RIE black silicon		

The additive techniques used in Paper I were thermal oxidation, low-pressure chemical vapour deposition of silicon nitride, and aluminium sputtering. While the silicon oxide became the buried insulator, and while silicon nitride worked as protective layers for underlying materials, as a cap preventing boron out-diffusion, and as electrical insulation, the aluminium was used as electrical conductor. These methods all add material to the whole surface.

Moreover, a number of subtractive methods were used for the same paper. Anisotropic etching of the substrate wafer was done locally to thin it to the desired beam thickness. Reactive ion etching was used to remove silicon nitride, the silicon device layer, and the silicon oxide. Isotropic etching combined with lift-off shaped the aluminium. To release the bending beams, the silicon was etched through by Twente MicroProducts, Enschede, The Nederlands [5] (black silicon method). In addition to the manufacturing methods, stress measurement structures in the device layer were released with isotropic vapour etching of the buried oxide [6].

The modifying methods used were bond annealing, boron implantation, and post metal annealing. Notwithstanding the fact that thermal oxidation already has been listed as an additive method, it is listed also here as the silicon in the silicon dioxide obtained originates from the wafer.

The additive methods work on the whole surface, and so do the subtractive ones, unless measures are taken to protect parts of the surface. By choosing a subtractive method that selectively removes one material and spares another, areas can be protected by the resilient material. Hence, structures are created that are integral parts of the components in fabrication. In our case, the predominant patterning method is photolithography. Again, addition, modification, and subtraction methods are used. In this technique, an applied photosensitive polymer exposed through a template protects the underlying materials in unexposed areas. As the entire unprotected surface is exposed in one step, all structures are defined simultaneously. The light modifies the structure of the polymer thereby changing its resistance to a wet etchant, *viz.* the developer. The result after a dip into the etchant is a protecting polymer pattern corresponding to the template. By choosing an etchant that selectively etches the unprotected areas but does not affect the protective layer, structures are defined all over the wafer. Afterwards, the protective layer can be selectively removed, making the surface ready for another additive layer. A clever use of additive, subtractive, modifying and joining methods, together with patterning, makes it possible to define structures of the components on the wafer.

Direct bonding

Direct bonding can cause problems when introduced in standard process chains. Although it might be possible to bond late in the production scheme, this would often require an alignment of two structured wafers, which entails difficulties in precision. In addition, the bond strengthening thermal treatment has to be limited to spare the structures previously made. If still endeavoured, the originally very smooth surfaces might be damaged, precluding bonding. On the other hand, if bonding is made early in the process, the bond must be resistant to later treatment. One such influence was investigated in Paper II.

A wet etch through a bond can be assumed to affect the result severely. Therefore, an investigation and characterisation on how an anisotropic wet etch affected the bond of silicon wafers was conducted. Prior to bonding, either, both, or none-of the wafers can be thermally oxidised. The bond can therefore be placed in four different locations with respect to the silicon wafers and the etching direction. The results showed that an oxide-oxide bond where the oxide was penetrated by plasma etching came off best.

While readymade bonded wafers were used in Paper I, and a standard bond's resilience against etching was investigated in Paper II, Paper III involves investigations of the bond process in itself. Preliminary results had shown an increase in bond strength at the edge of oxygen plasma pre-treated wafers. The objective was to get an understanding of the underlying mechanism responsible for this local improvement, in order to improve bond strength over the whole wafer area. The blister test, where sealed cavities are subjected to a gas of increasing pressure until fracturing, was chosen for the study. With this method, it was possible to measure bond strength at 24 locations over the wafer. In addition, the blister test is a good choice as it shows very good repeatability. The bond strength at different positions on the wafers were compared with x-ray photoelectron spectroscopy (XPS) investigations and atomic force microscopy measurements (AFM) at the same radial distance. The study revealed that the chemistry varied over the radius, while the roughness did not. By placing a fused silica plate between the wafer and the cathode in the plasma chamber, the radial variance disappeared.

Quartz microengineering

Quartz is a not only a semiprecious gemstone but also a material that surrounds us everywhere. It helps us to keep track of time in our wristwatches, it sequences the events in our computers, and it squeezes all our voices into the wireless world. Tiny quartz resonators make all this. The piezoelectrical property of quartz makes it possible to both create and detect vibrations electrically, so to speak to strike a tone, and listen to it. As in the case of musical instruments, it is sound velocity, mass, and shape of the vibrating member that decides its tone, *i.e.* its frequency. The ability to keep in tune, to keep the frequency stable, is essential. If the frequency is out of target, notes will be false on the piano, the clock will be gaining or loosing, and sender and receiver in communication systems will lose contact. In most cases, as those mentioned above, the inherent stability of the quartz resonator is good enough. For the most demanding applications, *e.g.* precision resonators used in telecommunication equipment, devices need to be developed to maximum stability.

The environment around a quartz resonator affects frequency by factors as temperature, acceleration, ionising radiation, electromagnetic fields, atmospheric pressure and humidity. Even if the device would be placed in an invariant surrounding, there would still be disturbances built-in in the quartz, in the electrodes, and in the encapsulation. Over periods of seconds, short time instabilities arise from mechanical stress, phonon scattering by defects and quantum fluctuations, and random vibrations and thermal fluctuations. Over periods of years, ageing will result from mass transfer, stress relief, out-gassing, diffusion, and chemical reactions. Of course, the stability will be affected by a combination of these influences.

Several of these stability problems originate from the encapsulation. All resonators have to be packed in capsules to shield them from the ambient as even individual molecules sticking to the surface will alter its mass, thereby altering its frequency. Unfortunately, the encapsulation is a source of contaminants in itself. Many ingenious solutions have been tested and some of them with success. An ultimate method would be to directly join quartz without any intermediate layer in such a way that the interface between the quartz is airtight but does not influence the mechanical properties of the device. Since the encapsulation is of the same material as the resonator there would be no thermal mismatch.

Given that such joining exists, it could also be used to tailor multi-layer resonators to have different crystallographic directions in different parts of the device. Due to its non-trivial structure, many properties of quartz are anisotropic, and so are their influences on the device frequency. By joining clever combinations of quartz cut in directions with antagonistic frequency response, the influence from the ambient, such as acceleration, temperature, and pressure, could be avoided.

For these reasons, a quest for such a joining method could be worthwhile. Where should one look?

Silicon microengineering presents itself immediately as silicon and quartz share characteristics. From a handling point of view, both materials come as clean, single crystalline, brittle slices. Silicon industry and quartz resonator industry produce the largest series of products in the world – electronic devices. Both industries make good use of complex and refined processing methods, but as they have different background, the methods differ. In silicon technology, wafers are large and can contain several thousand individual components. Most manufacturing equipment is adapted to wafers and work on all components simultaneously. In contrast, most quartz processing equipment handles one blank, *i.e.* one unworked component, at the time. However, there is one notable exception; the quartz tuning fork present in almost every wristwatch is processed in a similar way as the silicon components. In 1973, Jürgen Staudte presented a manufacturing method based on thin film deposition, lithography, and etching [7]. This was an extremely successful technology transfer from the silicon to the quartz world. Could there be more from where that came from?

The benefits of photolithography for quartz

As mentioned above, a successful technology transfer of silicon microengineering to the quartz business was made in the early seventies. Today, more than two billion quartz tuning forks are manufactured each year with a technology borrowed from there [8]. In this process, quartz crystals are cut into wafers and polished. With lithography, a deposited gold layer is patterned for masking, and the tuning fork is formed by anisotropic etching. Introducing this procedure has two distinct advantages. First, it is possible to miniaturise the tuning forks due to the precision of the lithography. Secondly, and more important, is the possibility to define many components on each wafer at once, and to etch many wafers simultaneously. This greatly reduces process time compared with the single component handling used previously.

The expectations on direct bonding for quartz

The quartz tuning forks mentioned in the previous section are made in a cost-effective chain of batch processes, but the chain is broken when it comes to packaging. Today every resonator is mounted individually. Depending on the choice of encapsulation, the package itself will introduce more or less severe instability problems. When adhesives or solders are used to hermetically seal the package, atoms and molecules from the sealing will eventually end up on the resonator. Thermal mismatch between the package, the sealing, and the resonator will introduce tensions in the resonator. Such disturbances will affect the stability of the resonator. Excluding other materials than quartz, and the inevitable electrical connections from the package as well as from the seal gives the best conditions for long-time stability. By borrowing the bonding technology from silicon microengineering, this can be done. In addition, bonding allows for batch processing.

Intuitively the transfer of bonding technology from oxidised silicon to quartz should be straightforward since quartz and silicon oxide are polymorphs *i.e.* has the same stoichiometric composition. The silicon bonding process based on surface termination with hydroxyl groups, and sequentially joining and thermal annealing simply should do the trick.

Indeed, it is possible to bond quartz with the same protocol as for silicon, see Paper IV, but for different reasons, success is far from granted.

Material quality and characteristics specific to quartz

The demand on the integrated circuit technology has forced the suppliers of silicon wafers to bring the polishing of silicon close to perfection. The surface roughness root mean square (rms) value is typically in the order of one Ångström. Quartz wafers intended for bulk acoustic resonators do not share these requirements, on the contrary they can be 80 times rougher (own unpublished work). Only with quartz wafers with a surface roughness less than 25 Å rms, we found it possible to bond.

The material characteristics of quartz also place obstacles in the way. In silicon technology, the bonded wafers are annealed, sometimes at temperatures over 800°C, to maximise bond strength. The temperature of transition from piezoelectric alpha-quartz to beta-quartz is 573°C. As quartz is mated to quartz, the change in density and other material characteristics is not critical but as the temperature is reduced to below the transition temperature the transition can lead to sudden and local twin formation or multi-crystal formation. Therefore, there is a maximum allowable temperature in quartz processing which obviously limits the annealing temperature and the bond strength.

Another problem arising has its origin in the difference between the amorphous silicon dioxide on the silicon surface and the crystalline silicon dioxide of quartz. In the wet chemical bonding process, water is attracted and adsorbed to the hydrolysed surfaces and abridges them due to hydrogen forces. At moderate annealing temperatures above 110°C, the water is rearranged between the wafers forming energetically favourable structures raising the bond strength. At higher temperatures, siloxane covalent bonds start to polymerise, creating and squeezing the water molecules at the surfaces. These water molecules have to move somewhere, implying that the permeability of water in silicon dioxide or quartz will matter. The permeability of helium in vitreous silica, *i.e.* high purity silicon dioxide glass, is seven orders of magnitude higher than in crystalline quartz [9]. It seems reasonable to assume that the permeability of water also differs significantly. If so, the only way for water to disappear in bonded quartz wafers is along the interface, out in the open air. If a water molecule is trapped, it becomes a defect and a nucleation site where more water molecules can gather and locally prevent bonding.

One more characteristic of quartz is that it is an insulator and therefore easily becomes charged. It will then attract particles worn off its edges to the surface, making the quartz wafer its own contamination source.

Quartz direct bonding adaptation

The differences in material quality and material characteristics hinder a straightforward transfer of silicon bonding technology to the quartz world. The process has to be adapted to overcome the difficulties encountered.

Furthermore, the surface roughness of the quartz wafers is a matter of wafer specification. The quartz polishing methods are often trade secrets. It will be time consuming to try to improve the polishing methods although a demand for smoother surfaces would probably result in a supply of such wafers. In fact, quartz wafers intended for surface acoustic wave (SAW) devices have smoother surfaces since the surface is the active part of the device and these wafers are therefore better but not excellent for bonding.

The transition temperature is a natural limitation that the quartz world has to live with. It could very well be that the a carefully conducted annealing above the transition temperature could lead to the same monocrystalline alpha quartz that was the starting material, but the risk that it will not does not justify the effort, especially since theory suggests that time might compensate temperature, at least in silicon bonding.

As mentioned, unbonded areas emerge due to the limited permeability of water in quartz. Creating diffusion channels that allow the water to escape was found to reduce this problem, Paper V. Another way to avoid the problem was to use alternative surface-activation methods such as plasma and argon bombardment methods [10].

Particle contamination can be almost completely avoided with special cleaning procedures specially developed for particle removal [11].

Direct bonded quartz applications

Bonded quartz structures have unlimited potential for tailoring the piezoelectric characteristics of the device. Probably of which only a small fraction has any commercial value.

The commonly employed AT cut crystal is cut at an angle that is thermally insensitive in a certain range. By laminating two or more wafers of carefully selected cuts, it should be possible to make the device stable in a wider temperature range. In the same manner, other frequency influencing quantities can be compensated for, as long as the bonded wafers act antagonistically.

A direct example is the acceleration dependence of the frequency in resonators. Quartz exists in two configurations, left- and right-handed, that are mirror images of each other, and so is the acceleration dependence. By bonding two equally thick wafers of left- and right-handed quartz, acceleration compensation can be achieved. As the quartz community has made up its mind to only use right-handed quartz in order to simplify handling, left-handed quartz is not easily accessible. Nevertheless, a far and wide search resulted in a few samples. Unfortunately, they seem to be of an unsuitable surface roughness for direct bonding.

However, to show the possibility of making a bonded resonator, equally thick wafers were bonded. Resonators were patterned and cut out of the bimorph wafer. For comparison, resonators were made out of a monolithic wafer in the same manner. The quality of both types of resonators were tested with frequency measurements; Paper V.

As mentioned in the beginning, an all-quartz package would increase the stability of quartz resonators. In 1977, Raymond Besson presented a clamped all-quartz resonator with exceptional good short-term frequency stability [12]. A replacement of the metal clamps holding this resonator pack together with bonding holds the potential of improving the ageing of the resonator further. In work not included in this thesis, two examples of all-quartz bonded packaging of devices were made by bonding quartz wafers [13]. These were SAW devices and web-moated resonators; *i.e.* the resonator was mechanically connected to the surrounding by a few narrow bridges made from the same wafer. These devices did not only show the feasibility of bonding quartz and the bonding compatibility with standard processing steps as metallisation and etching. They also showed the possibility of batch processing – to simultaneously process several components on the same quartz wafer, just as in silicon microengineering.

Quartz polishing

An oral presentation at the IEEE Frequency Control Symposium in Seattle 2001 arose the audience's interest. It was claimed that a polishing etchant for the non-crystallographically and industrial extremely important AT-cut wafers had been found [14]. The etchant was claimed to bring a roughly lapped quartz surface to atomic smoothness. Repeating these experiments indeed seemed as a both interesting and important task; Paper VI.

The conference paper was hardly assessable, as it was written in quite poor English, and what was more, it did not reveal any process details, although the importance of an effective cleaning was stressed. Correspondence with the author was almost fruitless; a patent application presenting process settings was attained, though. As it turned out, the etchant could not be brought to one of the temperature-concentration states given as it exceeded the liquids solutions boiling point. Furthermore, as no evident polishing effect could be seen in initial attempts, special precautions had to be taken in order to trustworthy refute this assertion. Ammonium bifluoride solutions had generally been used for quartz surface polishing, a literature survey showed, but never before at these extreme conditions. Experiences from surface cleaning for wafer bonding came in handy as the requirements for bonding surpasses those of most other uses. To maintain the desired concentration, a special set-up including a tight water-cooled lid was constructed. Stylus profilometry was supplemented with AFM for surface roughness measurements. Chemometrical methods were used to search for optimum conditions giving the lowest surface roughness. Hence, instead of generally used experimental design, where data are analysed by varying one parameter at a time, the experiments were factorial designed. By this, all combinations of the factorial levels are investigated. Here, obviously, the factors were temperature and concentration. This design is more efficient considering the influence of experimental errors on systematic variations and does reveal possible interaction between the factors. Then, the experiments are conducted and the desired variables are measured. Here the variables were etching rate and surface roughness. These data were used to calculate the relation between factors and variables. This relation can be represented as a curved surface in the (multidimensional) space that is filled out by factors and variables. This surface is used to find optimum conditions.

By this, it was evident that etching under these extreme conditions offered no extra advantages. In fact, initially polished samples became rougher. Non-spectacular as this finding certainly is, it might not render the paper the above-mentioned attention but it will definitely come as a relief for the quartz community.

Wafer bond adhesion quantification

From the very beginning of the development of wafer bonding, the bond strength has been considered one of the most important characteristics to determine about a bond. Adhesion measurements have contributed to the understanding of chemical reactions behind the bonding process, *e.g.* the transformation of weak hydrogen bonds, initially pulling hydrophilic silicon wafers together, into strong covalent siloxane bonds upon treatment at elevated temperature. Now, an increased interest in bond adhesion quantification can be anticipated when wafer bonding is optimised for complex microelectromechanical systems. In order to integrate circuitry into micromechanical systems, the bond strengthening thermal treatment has to be limited. This limitation also holds for bonding of dissimilar material with differences in thermal expansion. In the same way, the chemical surface pre-treatment must be selected to comply with all parts of the system. Further, at later steps of a fabrication process, the accumulated value of the previous steps is at stake. Merging of costly pre-processed subsystems into larger systems by wafer bonding therefore require high process yield. These severe requirements demand good quantification methods to develop reliable bonds and wafer bonding processes, and to provide the understanding necessary to facilitate the work.

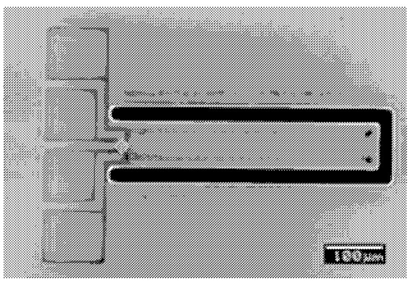
The report, Paper VII, encloses a comprehensive review of present adhesion quantification methods used for evaluation of the strength of wafer bonds, together with suggested augmentations. The most commonly used methods, namely the double cantilever beam, tensile, chevron and blister test methods, have been thoroughly treated by solid mechanics and brittle fracture theory. The improved techniques usefulness for three areas of use: general understanding of bonding mechanisms, bonding scheme optimisation, and quality control, has been forecasted.

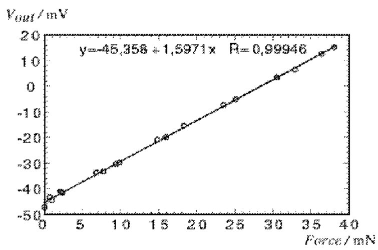
The main conclusion of this review is that adhesion quantification of wafer bonds can be dramatically improved by small means. The basic ways and means can be found in models and experiences made in neighbouring research fields. Both for the characterisation of brittle materials, and adhesives and solders, standard procedures have been developed. Adaptation of these procedures to wafer bonds would entail a time and resource saving shortcut. The crux of the matter is, however, that such adaptation demands basic knowledge in solid mechanics and fracture behaviour of brittle materials.

Summary of papers

Summary of Paper I: High-temperature piezoresistive gauge fabricated on commercially available silicon-on-insulator wafers

This paper presents a micromachined, four-terminal, piezoresistive gauge intended for use at high temperatures, Fig. 3a. Originally, the project was launched by Sandvik Coromant to solve the problem of when to exchange the cutting tool in metal-turning lathes. If the cutting edge is allowed to completely wear out, the tool breaks and causes damages on the working piece, or on the lathe. The company had an indication of the force on the tool's edge increasing immediately before breakage, but they could not measure the force accurately with existing techniques. The task, therefore, was to integrate a force sensor in the cutting tool holder, immediately below the replaceable cutting edge.





a) b)

Figure 3: a) Scanning electron microscope image of a gauge. The contact pads on the left connect to the piezoresistive mesa. The mesa is placed on the base of the cantilever. b) Output voltage versus load for a gauge at 188°C. The equation is the linear fit to the measurement points.

The idea was to measure the compression of the tool holder by monitoring the deflection of the supporting surface. Besides the limited size, a couple of cubic millimetres, the environment is harsh with high temperatures and mechanical vibrations, putting severe demands on the solution.

The gauge is manufactured out of the 2 μm thick device layer of a silicon-on-insulator wafer and a cantilever beam is defined in the handle wafer. The buried oxide layer serves as electrical insulation. The deviation is transferred to the bending cantilever, 600 μm in length, and the mechanical strain of its upper surface is measured by a single-element strain gauge, making good use of the shear modulus of silicon, Fig. 3a.

The device proved to work at least up to 400°C. *In situ* scanning electron microscope measurements showed good linearity for each examined temperature, Fig. 3b. Unfortunately, the off-set of calibrated measurements varied unpredictably with temperature, probably due to thermal strain in the chip's mechanical fixture.

Summary of Paper II: Silicon fusion bond interfaces resilient to wet anisotropic etchants

In order to spare the sensitive surfaces, direct bonding is most often performed as early as possible in a process. Consequently, to be able to choose the following processes freely, the bond must be compatible with subsequent machining.

In an effort to find the best way of penetrating the bond of direct bonded silicon wafers by wet etching, different configurations of thermally oxidised or chemically oxidised silicon, as well as hydrophobic pairs, were bonded. The behaviour of the anisotropic etchants potassium hydroxide (KOH), and tetramethyl ammonium hydroxide (TMAH) were investigated. Where necessary, the bond was penetrated by hydrofluoric acid or trifluoromethane plasma etching.

It was discovered that the bond interfaces were resilient to the process when two thermally oxidised wafers were bonded and the bond was penetrated by plasma etching, Fig. 4.

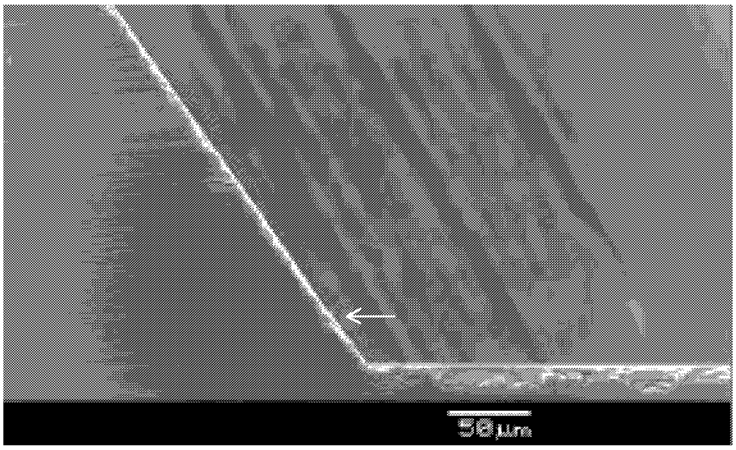


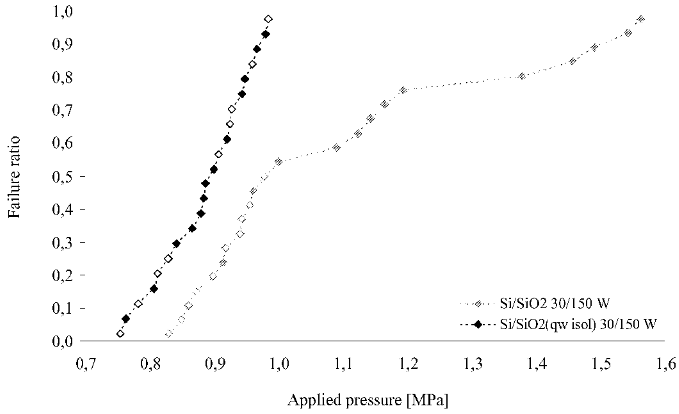
Figure 4: Etch profile of oxidised silicon bonded to oxidised silicon. The location of the bond is shown by an arrow. The desired sidewall profile is perfectly maintained.

Summary of Paper III: Radial variations in bond strength for plasma bonded oxidised silicon wafers

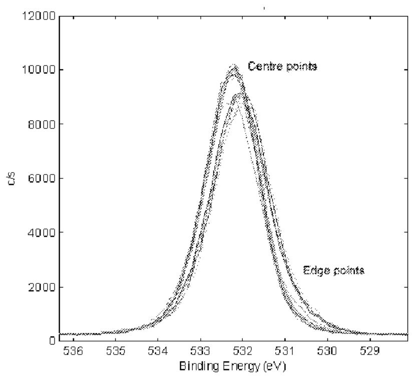
Previous studies had shown that the bond strength was higher at the edges than in centre positions of wafers bonded after plasma pre-treatment. To improve bonding over the whole wafer area, a study was performed in order to get an understanding of the underlying mechanism responsible for the local bond strength enhancement.

By using the blister test, see Paper VII, it was possible to map the bond strength of 100 mm diameter wafers at 24 points. The bond strength variation with radial location was related to XPS and AFM measurements.

Figure 5 (a) shows that the edge effect can be eliminated by using an isolating cathode. The use of an isolating cathode gives less scatter and diminishes edge defects at the expense of bond strength. The increased strength of edge specimens agrees to a shift in oxygen binding energy at the surface, as measured by XPS, Figure 5 (b).



(a)



(b)

Figure 5. Applied pressure at failure vs. failure ratio for Si/SiO_2 pairs bonded with and without isolating cathode during plasma surface pre-treatment (a). The markers are filled for edge specimens. XPS spectra for a wafer identical to that at the right in (a) obtained at centre and edge locations show a shift in surface oxygen bonding energy at the edge (b).

Summary of Paper IV: Quartz-to-quartz direct bonding

As oxidised silicon is readily bondable, a quest to bond crystalline quartz as well was undertaken. Quartz bonding would entail possibilities in making extremely stable quartz packages for quartz resonators, and to give new degrees of freedom in resonator design.

Silicon wafer bond schemes were used to bond quartz wafers of two commonly employed types.

The quest was prosperous; quartz-to-quartz direct bonding was scientifically presented for the first time. It was shown that it was possible to direct bond quartz with wet chemical treatment previously used for silicon as long as the surface roughness was low enough. The surface energy of the bond increased drastically with increasing annealing temperature, Fig. 6.

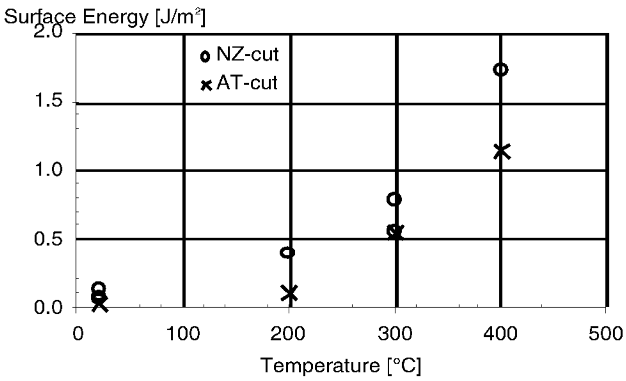


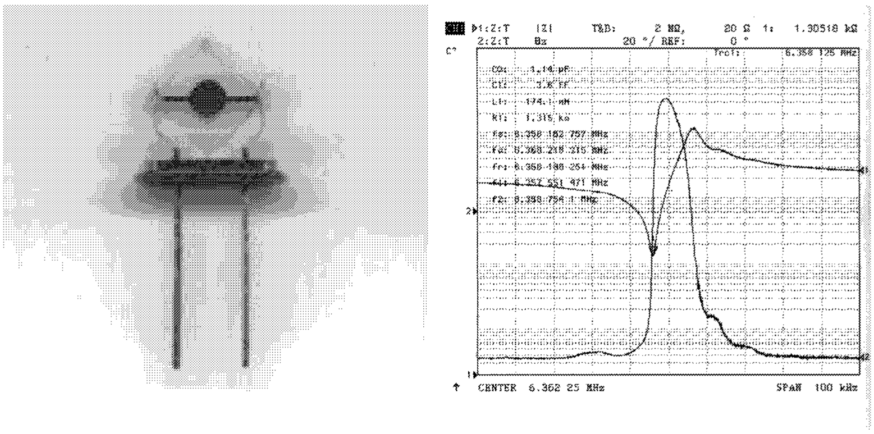
Fig. 6. Measured surface energies vs. annealing temperature for AT-cut and X+1°50'-cut quartz. Annealing time was 1 hour.

Summary of Paper V: Direct bonded quartz resonators

The influence on a resonator by a direct bond interface was investigated with two objectives in mind – to facilitate bonding in resonator design, and to investigate the possibilities of using frequency measurements for adhesion testing.

Here, the resonator design was adapted to silicon microengineering, and by no means an optimised structure, Fig. 7. Still, valuable measurements could be performed as monolithic resonators of the same design were characterised for comparison.

Bonded resonators had Q-values about one order of magnitude lower then monolithic resonators, mainly due to high series resistance. It was speculated that trapped water was responsible for the high resistance.



a) b)

Figure 7. A bonded resonator (a), and the resonance behaviour for one of the bonded resonators (b). The curves shown are for absolute impedance and phase.

Summary of Paper VI: Polishing of quartz by rapid etching in ammonium bifluoride

It was desirable to investigate the effect of high concentration and high temperature etching of quartz, as it had been clamed elsewhere that a polishing effect could be seen for a non-crystallographic wafer cut.

A thorough survey of relevant literature showed that polishing had not been performed at these conditions before. As no evidence of a polishing effect could be seen in initial experiments, great experimental care and multivariate data analysis were employed. The experiences from surface cleaning for direct bonding came in handy, as these demands probably surpass ordinarily employed techniques. The polishing was conducted with good control of temperature and concentration. Topography measurements by commonly employed cantilever stylus were supplemented with AFM measurements.

The investigation made it possible to claim with confidence that no polishing effect occurred, Figure 8.

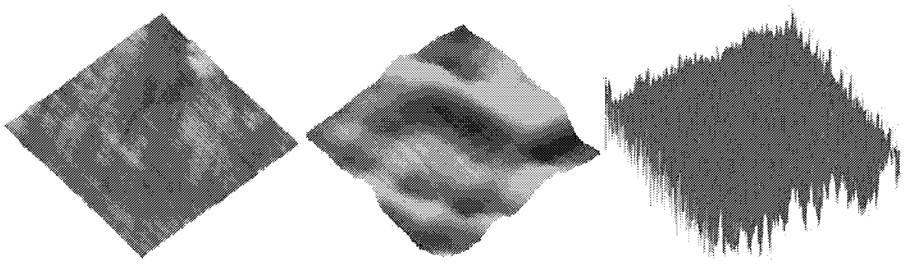


Figure 8. Atomic force microscopy scans for just ultrasonically cleaned (a), ultrasonically cleaned and HNO_3 cleaned (b), and cleaned as in (b) plus etched for 5 min in 90°C in 80 wt-% ammonium bifluoride (c). All profiles are 1 by 1 μm laterally and enlarged by an extra factor of 40 in the vertical direction.

Summary of Paper VII: Adhesion quantification methods for wafer bonding

The amount of adhesion has been considered one of the most important characteristics of a bond since the advent of direct bonding. Yet, the methods have not been developed since introduced, although isolated problems have been addressed. To improve the used methods, an overall view is necessary, where all obstacles are dealt with simultaneously.

This report offers a comprehensive review of present adhesion quantification methods used for evaluation of the strength of wafer bonds, together with suggested augmentations. The most commonly used methods, namely the double cantilever beam, tensile, chevron and blister test methods, have been thoroughly treated by solid mechanics and brittle fracture theory. The improved techniques usefulness for three areas of use: general understanding of bonding mechanisms, bonding scheme optimisation, and quality control, has been forecasted, Table II.

The major finding was that the quality of all commonly used methods could be dramatically improved by small means.

Table II. Forecast of the usefulness of the four main techniques reviewed in relation to the area of application – to understand, improve and check bond adhesion.

	Understand	Improve	Check
DCB	😐	☺	😐
Tensile	😐	😐	😐
Chevron	☺	😐	😐
Blister	☺	☺	😐

☺ = most useful 😐 = could be used ☹ = cannot be used

Acknowledgement

Almost twelve years ago, I sat down in Ylva's old sofa, making my first contact with the micromechanical world. (No, I didn't sit on any samples). During the following years I gradually and almost unnoticed made a journey from hang-around to present state. During this trip I met a number of persons to whom I am indebted. Addressed in somewhat chronological order you are:

Ylva Bäcklund, the spirit of the Micromechanics group was never as joyful as when you led it. It was a time when everything thinkable was doable. Many enterprises were challenging, and our banner was always kept flying.

Pelle Rangsten, it was during under-graduate courses we became friends, and you brought me to Ylva's sofa. You taught me everything you knew about ingenious experimentalism, and how to keep an enthusiastic approach. However, I could never mimic your endurance.

Bertil Hök, for me, you have always been the anchor firmly holding the micro-ship in place, preventing it from drifting away. Although we haven't always agreed on the means and ways, I believe we have always shared the objectives.

The Micro Mechanics group, all former and present members, your company has always been pleasant; we have shared many joyful moments. You are my main affiliation.

Sören Berg, due to the strong independence of the groups forming the Solid State Electronics division, you were almost anonymous to me for a long time. During the lengthy finish of my work you have however commendable come to my rescue with practical solutions and financing.

Solid State Electronics, the casual attitude amongst the people at this division has always appealed to me. It has made me feel at home. This special attitude is manifested through the Friday beer. I am sorry that I have been absent in recent years.

Herman Norde, for me you represent the Electronics in Solid State Electronics. You have always been able to provide practical solutions or explain the electro-physical background of any question asked. In addition, I have great respect for your values and way of thinking.

The administrative personnel, as well as the computer support, you have always been easy to get in touch with, and have always relieved my concerns. I have enjoyed your company over biscuits and cakes for numerous Wednesdays.

The research engineers, former and present, I have always admired your knowledge, and have always found it readily accessible.

Greger Thornell, my dear friend, hobby supervisor, and mentor. Although never formally appointed, you have been my best and most supervisor. With unflagging interest you have encouraged my ideas, criticised my mistakes, and never lost faith in the completion of my work.

Carolina Ribbing, sharing office with you was great. With professionalism, empathy, and a wonderful sense of humour, no one could wish for a better roommate.

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Kerstin Jonsson, working with you has been very pleasant, and has brought me back to firm ground at times when I almost lost foothold. You made me retrieve the true foundations of science – to go gradually without shortcuts, to work hard, and to have a humble attitude towards own results. No wonder that review took two years to complete.

Co-authors, you always made your part, and never just rode along. Thank you for good collaboration.

Hideki Takagi and Toshio Ishizaki, my Japanese friends. Although we have met at few and swift occasions, these encounters have made deep impressions on me. Thank you for your openness and hospitality.

The people involved in projects that were never fulfilled: Jun Lu preparing and transmission electron microscoping quartz in search for the Beilby layer, and, in quest of acceleration immunity, David Salt for the unique left-handed quartz blanks, Tianhua Wu for the matching right-handed blanks, and Michael Ottosson for good help in orienting the blanks with x-ray diffraction. Thank you for your kind assistance. I sincerely hope that you don't feel sent on fools' errands.

The Almost Royal Swedish Academy of Bitterness (Svenska Bitterhets-akademien) has been a forum where the rest of the world could be blamed for almost everything, whilst we were discussing how Good Research should be performed. I am grateful for the rise and existence of this distinguished safety valve.

The duty of the Work-Police has been to continuously monitor the members' efforts at work. As the end is near, I now can reveal that buying biscuits to be consumed at next inquisition is not an effective punishment for arrears of promised work. Nevertheless, the meetings we had were always pleasant.

The Ångström Library. This wannabe librarian expresses thanks for all the help, loans, and journal articles bestowed on me, and for providing a completely calm and quiet hideout, *viz.* the archive.

Uppsala, April 2005, sitting in the very same old sofa

Örjan Vallin

PS

Even though I never have understood the habit of dedicating one's work to somebody, I have at least realised that this is a good place to state feelings and affections not frequently expressed.

To my wife and children, to my family and family in law, and to my Brothers under the full moon: I love you all. You are the true reason for keeping going.

Sammanfattning på svenska
Summary in Swedish

I svensk översättning är titeln på denna doktorsavhandling "Mikroingenjörskonst tillämpad på kisel och kvarts – framställning och karaktärisering". Mikroingenjörskonst, även kallad mikrostrukturteknik eller mikroelektromekanisk systemteknik, betecknar en teknik som utvecklats ur mikroelektroniken. Med samma metoder som används för att göra elektroniska komponenter skapas mekaniska strukturer. Dessa strukturer kan fungera som avkännare, motorer eller resonatorer. Avkännarna, sensorerna, kopplar en mekanisk signal, t ex tryck, till en signal som kan avläsas elektroniskt. Motorerna, eller aktuatorerna som de ofta kallas, omvandlar tvärtom en elektrisk signal till kraft eller rörelse. Resonatorer kan sägas vara en hybrid där vibrationer både kan skapas och avläsas elektroniskt. Resonatorns geometri bestämmer med vilken frekvens den skall jobba och fungerar på samma sätt som en stämgaffel. Resonatorn som styr tiden i kvartsur är formad just som en sådan. Medan kisel är det vanligast utgångsmaterialet i sensorer och aktuatorer, så används kvarts ofta för resonatorer. Gemensamt för materialen är att arbetsstycket utgörs av en skiva.

Den första artikeln i avhandlingen kan fungera som exempel för mikroingenjörskonsten eftersom många olika framställningstekniker har används. De använda metoderna kan indelas i fyra grupper: additiva, subtraktiva, modifierande och sammanfogande. Medan de additiva metoderna används för att lägga till material, så används de subtraktiva för att ta bort. Material kan läggas till genom att låta gaser reagera och bilda ett tunt skikt på ytan, eller genom att förånga ett material i närheten och låta det kondensera på ytan. Material kan tas bort genom att använda kemiska etsvätskor, eller genom att bombardera ytan med atomjoner. De modifierande teknikerna utförs för att förändra materialen, t ex för att få dem att sitta fast bättre, eller för att ändra deras ledningsförmåga. Här är värmebehandling vanlig. Sammanfogande tekniker används för att sätta ihop större delar, ofta två skivor, till skillnad från de additiva metoderna där materialet byggs upp succesivt. Ingen av alla dessa tekniker skulle vara till mycken nytta om det inte gick att välja var på arbetsstycket man vill lägga till eller ta bort material. Man måste också kunna mönstra skivan. Detta görs övervägande med hjälp av fotolitografi. En ljuskänslig lack appliceras och exponeras med ljus som passerar genom en glasskiva där det önskade mönstret finns utritat i svart. För en positiv lack kommer de exponerade områdena att förändras så att de tas bort vid den efterföljande framkallningen. Nu kan en subtraktiv metod användas på önskade områden eftersom resten skyddas av lacken.

I huvuddelen av de arbeten som presenteras här fokuseras forskningen på sammanfogning av skivor. Två plana och släta ytor som gjorts reaktiva kan fås att fastna ihop utan mellanliggande skikt. Fogen blir så tunn att den kan försummas i förhållande till skivtjockleken och precisionen blir mycket god. Frånvaron av lim eller lod gör att man undviker föroreningar. Ytorna kan t ex göras reaktiva genom att tvätta skivorna i en syra, eller genom att utsätta dem för ett syrgasplasma i en vakuumkammare. När skivorna efteråt doppas i vatten så täcks ytan med hydroxylgrupper (OH) och några få lager med vattenmolekyler. Då två sådana ytor läggs samman attraheras de till varandra av vätebindningar. En efterföljande värmebehandling driver bort överflödigt vatten. Vid höga temperaturer ersätts vätebindningarna av starkare bindningar.

I följande stycken sammanfattas delarbetena i denna sammanläggningsavhandling.

I artikel I (*Paper I*) beskrivs tillverkning och karaktärisering av en sensor avsedd att mäta kraften på ett svarvverktyg. Avsikten var att mäta sammantryckningen av materialet omedelbart under svarvskäret för att få en uppfattning om kontaktkraften mellan verktyg och arbetsstycke. En halvmillimeterlång balk infäst i sin ena ände utsätts för en förskjutning orsakad av svarvskärshållarens sammanpressning. Då balken böjs sträcks dess ovansida; allra mest vid infästningen. Då sträcks också en liten kiselplatta som placerats där. Plattans kantlängd är 1/50 mm och dess tjocklek 1/500 mm. Om en ström leds genom kiselmaterialet kan en elektrisk spänning avläsas vinkelrätt mot strömriktningen. Den elektriska spänningen är ett mått på balkens nedböjning. Sådana sensorer konstruerades, utformades och utvärderades. De visade sig fungera åtminstone upp till temperaturer om 400°C, något som är en förutsättning för att de skall kunna användas i närheten av en metallbearbetande egg.

I artikel II undersöks vad som händer då man försöker att våtkemiskt etsa sig genom två kiselskivor som är direkt sammanfogade. Etsvätskan angriper fogen aggressivt, men genom att skydda båda skivorna med ett lager kiseldioxid före sammanfogningen minimeras problemet.

Artikel III behandlar plasmabehandling av kiselskivor som skall sammanfogas utan mellanliggande skikt. För att mäta hur starkt skivorna sitter samman görs gropar och inlopp i skivorna före sammanfogningen. De slutna hålrum som bildas trycksätts därefter med gas och brottrycket blir ett mått på bindningsstyrkan. Många teststrukturer kan göras på ett skivpar, vilket gör det möjligt att kartlägga variationer i bindningsstyrka över ytan. Den uppmätta bindningsstyrkan relaterades här till ytornas kemiska sammansättning och ytjämnhet. Den kemiska sammansättningen undersöktes med elektronspektroskopi och ytjämnheten med atomkraftsmikroskopi. Det visade sig att bindningsstyrkan och den kemiska sammansättningen varierar mot kanten av skivan, men inte ytjämnheten. Genom att lägga en isolerande platta under kiselskivan vid plasmabehandlingen försvinner variationerna, men fogen blir också något svagare.

I artikel IV presenteras för första gången direkt sammanfogning av kvartsskivor. Genom att de använda skivorna hade tillräckligt jämn yta kunde de sammanfogas på samma sätt som kisel vanligtvis görs. Det finns dock tre viktiga skillnader mellan kisel och kvarts. Kvarts genomgår en fastransformation med stor åtföljande materialutvidgning vid 573°C vilket begränsar den högsta tillåtna värmebehandlingstemperaturen. Kristallin kvarts kan inte heller absorbera och transportera överflödigt vatten så som oxiderat kisel; öar av instängt vatten kan förhindra sammanfogningen lokalt. Vidare är kvarts en isolator, vilket medför att partiklar – ofta från materialet självt – blir ett större problem eftersom de gärna fastnar elektrostatiskt.

Artikel V jämför kvartsresonatorer gjorda av sammanfogade skivor med resonatorer utan fog. Syftet var att se vilken störning fogen utgör. Sammanfogningen optimerades för den önskade skivstorleken. Trots detta visade det sig att kvaliteten blev tio gånger sämre med fog än utan, mätt med det så kallade Q-värdet. Om en fog ändå kan tillåtas skulle sammanfogningsmetoden kunna användas för att konstruera helt nya typer av resonatorer. Ett exempel är resonatorer som är okänsliga för acceleration.

Vid en internationell konferens presenterade häpnadsväckande resultat: man hade lyckats att våtkemiskt polera skivor sågade i en riktning som inte sammanfaller med ett kristallplan. Artikel VI vederlägger dessa resultat. För att göra detta på ett övertygande sätt används noggrann planering och genomförande av experiment samt så kallade kemometrisk analys av mätdata. Med kemometri tittar man på sambanden mellan flera ingående parametrar samtidigt, i motsats till konventionell metodik där en parameter varieras medan allt annat hålls konstant.

Artikel VII är en översikt av arbeten som behandlar eller använder utvärderingsmetoder för vidhäftningsförmågan mellan direkt sammanfogade skivor. Genom att föra in resultat från angränsande forskningsområden, t ex limning och utvärdering av styrkan hos spröda material, visas att stora förbättringar i mätmetoderna kan åstadkommas med små medel.

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