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Adhesion Evaluation of the Heat Resistant Pressure Sensitive Adhesives at Elevated Temperatures for MEMS Gyroscope Testing

This thesis is submitted in partial fulfillment of the requirements for the degree of Master of Science in Technology.

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Aalto University School of Electrical Engineering		ABSTRACT OF THE MASTER'S THESIS	
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<p>High-throughput functional testing of MEMS gyroscope can be performed on wafer level while the wafers are mounted on a conventional back-grinding tape used in integrated circuits manufacturing. However, the functional characterization should also be carried out at elevated temperatures as high as 150 °C. Exposure to elevated temperatures during the entire test procedure, which can take up to 90 minutes, set special requirements for the tapes. The adhesion properties of tapes must be adequate and stable at temperatures all temperatures and throughout the test.</p> <p>In this work the applicability of five different commercial wafer back-grinding (A-C) and dicing tapes (D and E) were evaluated. Tapes consisted of a base film, thin adhesive layer and protective film. The total thickness of tapes varied from 71 to 166 µm. Ethylene vinyl acetate (EVA) (Tape A), PET (B-D) and polyolefin (E) were used as base film materials. Tapes D and E were UV tapes. Adhesive in all five tapes was modified acrylic adhesive with different compositions of additives. Short- and long-time thermal stability, adhesion strength and detachment and adhesive residues were used as evaluation criteria. Adhesion was evaluated between typical MEMS package materials (silicon, polymer, and brushed steel). Examinations covered the following temperature and time frames: 25, 80, 100, 125 and 150 °C and 0, 30, 60 and 90 minutes.</p> <p>EVA tape deformed rapidly even at the moderate test temperature 100 °C, and was eliminated from the adhesion strength and residue evaluation. Other four tapes passed the thermal stability test without noticeable changes in their physical appearance.</p> <p>The adhesion strength and strain energy, or detachment work, were measured with the package pull-off test with the help of a tensile tester. Statistical analysis was used to analyse the results. Analysis of variance showed that all main test parameters and their interactions were significant ($\alpha=5\%$). Bonferroni all-pairwise comparison test was used to compare the means values. Following conclusion were made from the mean comparisons: i) surface material affects significantly to adhesion, ii) tapes' D and B adhesion varied the least among different treatments for the all three package materials, iii) adhesion strength increased up to 100-125 °C but at higher temperatures tapes lost their adhesion, iv) adhesion remains stable for the duration of about 60 minutes after which it starts decreasing abruptly. Additionally, tape C had very weak adhesion strength, and it can be especially too weak for small polymer packages with porous surface. The adhesion strength of 0.01 MPa may be considered as the limit below which the component won't attach properly to the tape. Tapes D and E had higher adhesion strength but they detach slowly from the tape due to glue fibrillation, in other words the detachment work is high.</p> <p>Detachment work can be used to predict the amount of residues on the package surface. UV-dicing tapes turned out to stain the components extensively, while Tapes B and C left few or no residues. Amount of residues were rated at scale of 1-5.</p>			
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<p>Puolijohdeteollisuudessa kiekkojen sahauskassa käytettäviä teippejä voidaan hyödyntää MEMS gyroskooppien funktionaalisessa testauksessa. Gyroskooppien toiminnallisuutta testataan korotetuissa lämpötiloissa, mikä asettaa erityisvaatimuksia teippien lämmönkestävyydelle. Teippien adheesion tulee säilyä stabiilina läpi testin 80-150 °C:ssa noin 1-2 h.</p> <p>Tässä työssä tutkittiin kaupallisten puolijohdeteollisuudessa piikiekkojen ohentamisessa ja sahaamisessa käytettävien teippien soveltuvuutta komponenttitestaukseen korotetuissa lämpötiloissa. Teipit koostuvat pohjateipistä ja ohuesta liimakerroksesta, jonka suojana on PET-kalvo. Teippien kokonaispaksuus vaihtelee 71-166 µm välillä. Pohjateipin materiaaleina on käytetty etyleeni vinyyli asetaattia (EVA) (teippi A), PET (B-D) ja polyolefiinia (E); Teipit D ja E ovat UV-teippejä. Kaikissa teipeissä on akrylipohjainen liima. Arviointikriteereinä käytettiin teippien stabiilisuutta lyhyt- ja pitkäkestoisessa lämmityksessä, adheesivoimaa ja irrotustyötä yleisempien MEMS kotelointimateriaalien (pii, polymeeri, teräs) ja teippien välillä sekä liimajäämien määrää komponenttien pinnalla. Testilämpötiloina käytettiin 25, 80, 100, 125 ja 150 °C ja lämmitysaikoina 30, 60 ja 90 minuuttia.</p> <p>EVA teippi jätettiin jatkotutkimuksien ulkopuolelle sen alkaessa käpristyä ja aaltoilla jo matalimmassa testilämpötilassa 100 °C 30 minuutin lämmityksen jälkeen. Muissa neljässä teipissä ei esiintynyt fyysisiä muutoksia lyhytkestoisissa lämmityksissä.</p> <p>Adheesivoima ja irrotustyö mitattiin vetokokeilla. Tulosten tulkinnassa käytettiin tilastollisen analyysin menetelmiä. Varianssianalyysin perusteella kaikki päätestiparametrit ja niiden vuorovaikutukset keskenään olivat merkittäviä ($\alpha=5\%$). Eri parametrien keskiarvojen vertailussa käytettiin Bonferroni-testiä, jonka perusteella tehtiin seuraavat johtopäätökset: i) kotelointimateriaali vaikuttaa merkittävästi adheesioon ii) teipit D ja B säilyttivät adheesionsa parhaiten eri lämpökäsittelyissä iii) adheesiolujuus kasvaa 100-125 °C:een asti, minkä jälkeen se alkaa laskea iv) teippien adheesio pysyy stabiilina noin 60 minuuttia. B teipin adheesiossa säilyy parhaiten eri lämpötiloissa ja lämmitysajoilla ja sen adheesio on riittävän suuri kaikille kolmelle kotelomateriaalille. C teipin adheesio on erittäin heikko ja erityisesti polymeerikotelo kiinnittyy huonosti siihen. 0.01 MPa voidaan pitää tämän perusteella minimiarvona adheesivoimalle. UV-teippien adheesio on vahva, mutta komponentit irrotettaessa muodostuu liimasäikeitä, jotka jättävät jäänteitä komponenttien pinnalle. Tämä näkyy suurena irrotustyönä.</p> <p>Irrotustyötä voidaankin käyttää ennustamaan liimajäänteiden määrää. UV-teipit jättivät eniten liimajäänteitä peittäen usein komponentin pinnan täysin. B ja C teipeistä ei irronnut liimajäänteitä kuin satunnaisesti. Komponenttien pinnanpuhtaus arvioitiin mikroskooppikuvien perusteella asteikolla 1-5.</p>			
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Preface

This Master of Science thesis work was carried out in the Laboratory of Electronics Production Technology as a contract study for the MEMS testing device company Afore. I am very grateful to supervisor of my thesis, Professor Mervi Paulasta-Kröckel for her support and time.

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Abbreviations

ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
AUC	Area Under Curve
BGA	Ball Grid Array
BS	British Standards
CSP	Chip Scale Package
DoF	Degrees of Freedom
EDL	Electric Double Layer
FINAT	Fédération Internationale des fabricants et transformateurs d'Adhésifs et Thermocollants sur papiers et autres supports
ISO	Organization for Standardization
LCP	Liquid Crystal Polymer
MEMS	Microelectromechanical system
PET	Polyethylene terephthalate
PSA	Pressure sensitive tape
SMT	Surface Mount Technology
TOTA	Test on Tape
QFN	Quad Flat No lead
WLP	Wafer Level Pack

1 Introduction

During the last two decades microelectromechanical systems (MEMS) have found their way from laboratories to consumer electronics and automotive industry. MEMS are usually defined as 1 to 100 micrometers in size mechanical structures fabricated with IC processing on silicon wafers. Also other materials such as glass and quartz wafers can be used. Their functionality is based on micromechanical sensor and actuator structures, such as bars, mirrors, resonators and pumps that react to a physical input and transform that into electrical signal. Optical, acoustic and fluidic phenomena have been also applied. MEMS actuators and sensors are used for example in inkjet printers, as memory read/write heads, and in vehicles as accelerometers, pressure sensors and gyroscopes.

Besides sharing common manufacturing methods with integrated circuits, MEMS devices are also dealing with similar reliability issues as ICs. However, the essential difference between the IC wafer level and MEMS wafer level testing is that whereas ICs need only electrical input and output, MEMS require in addition a physical input which set special demands for testing equipment.

High-throughput testing with low yield loss is important for the MEMS manufacturers. Beside general reliability testing at the R&D stage, functionality of the manufactured sensors has to be tested properly at different use temperatures. In case of MEMS gyroscopes testing, a test setup must also induce positive and negative angular velocities to the sensor elements. Capability to test various package types and wafers with the same test set-up increases efficiency remarkably.

One solution to enhance the performance of gyroscope testing is a Test-on-Tape method, in which the same test configuration is used for the wafer level and package level components. MEMS sensors are placed on the adhesive tape to the optimum pattern and no separate carriage is needed. However, the high temperature affects the adhesion of the pressure sensitive adhesives and can lead to adhesive failure between the tape and the MEMS gyroscope during the test.

In this thesis the adhesion durability of the Test-on-Tape adhesives will be evaluated at high temperatures and analysed, whether it can cause problems in the MEMS gyroscope testing process. Chapter 2 introduces briefly MEMS gyroscope and its testing. In chapter 3, adhesion and its failure mechanisms, pressure sensitive adhesives and the main adhesion strength test methods are presented and compared. In chapter 4, the objective of the thesis is specified. The most important results are concluded in chapter 6.

2 Microelectromechanical Gyroscope

A gyroscope is a device for measuring an object's turning velocity. The operation is based on conservation of angular momentum. The gyroscope was invented in 1852 by the French experimental physicist Leon Foucault during his investigation of the rotation of the earth [1]. However, there exist also earlier descriptions of similar devices by Bohnenberger (1817) and Johnson (1832) [2,3], but Foucault gave the original name and became known as the inventor. Foucault's gyroscope was a rapidly rotating disk with a heavy rim, mounted in low-friction gimbals or rings. The device is shown in Figure 2.1.

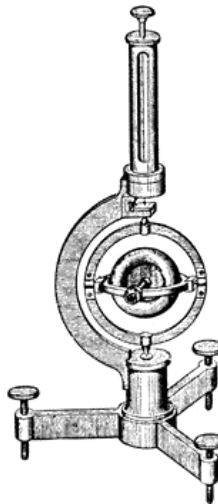


Fig. 2.1 *Foucault's gyroscope*

The gyroscope was a significant invention for the navigation and flying of the early 20th century [4]. Gyrocompasses and gyrostabilizers were used in ships and aeroplanes and they made auto-piloting possible. Today gyroscopes have become a common part of cars, aeroplanes, consumer electronics, and robotics [5]. In modern gyroscopes the rotating wheel is replaced by fiber optics, ring laser, or microelectromechanical systems (MEMS) [6]. Optical gyroscopes are an excellent choice for precision applications since they are the most accurate angular velocity sensors, but the size and price make them impractical for other applications. Mobile devices, wireless game controllers and stability control systems have

created a huge demand for small low/medium-price gyroscopes with medium accuracy. Microelectromechanical sensor technology has been the answer to that.

MEMS gyroscopes use vibrating mechanical elements to sense rotation [7]. Sensing is based on Coriolis acceleration which causes the transfer of energy between two vibration modes of a structure [8]. The Coriolis force (Figure 2.2) occurs when a mass m is moving in the x -direction \vec{v} and angular rotation velocity $\vec{\Omega}_z$ is applied around the z -axis. The mass will experience a force in the direction of the yellow arrow and will be physically displaced.

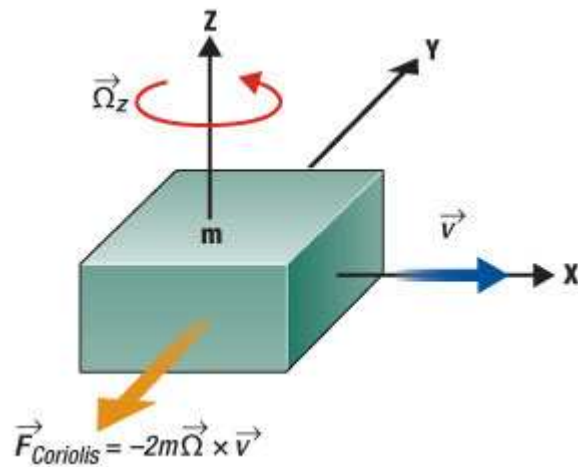


Figure 2.2: *The Coriolis effect*

A tuning fork [8] is the most used configuration in MEMS gyroscopes, but there are also other vibrating structures like vibrating shells and rings [7]. In the tuning fork system there are two masses oscillating and moving in opposite directions (Figure 2.3). When angular velocity is applied, the Coriolis force on each mass acts also in opposite directions, resulting in a capacitive change, which is proportional to the angular velocity $\vec{\Omega}_z$. If both masses move in the same direction, there is linear acceleration, no capacitance difference will be detected. The Coriolis change can be measured also by piezoresistive or piezoelectric mechanism [7].

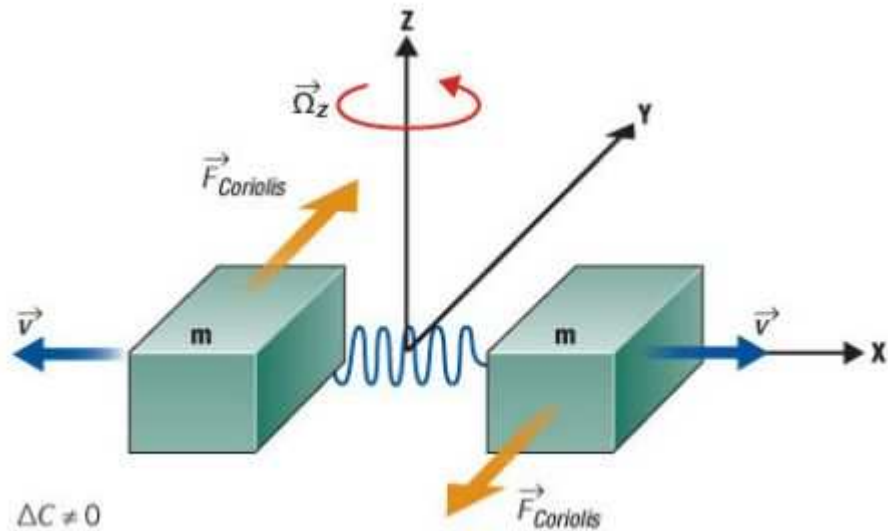


Figure 2.3 Schematic of a tuning fork configuration

In Figure 2.4 is presented VTI's new CMR 3000 gyroscope which is currently the smallest 3-axis gyroscope with the lowest power consumption [9]. Some of the advantages of MEMS sensor technology are miniaturisation, mass production, and reduced price per unit [7]. In addition, the long time reliability of the sensor increases greatly because the amount of moving parts is minimized and vibrating structures make only small movement around their axis. Nevertheless, the long-time reliability of manufactured sensors has to ensure by comprehensive product testing. These testing methods will be reviewed in the next chapter.



Figure 2.4 CMR 3000 gyroscope

2.1 MEMS Packaging

General electronics packaging trends have strived to increase interconnection densities of components. High I/O density is also important in MEMS components but microelectromechanical systems set in addition a lot of special demands for packaging, because of miniaturization, free standing microstructures, hermetic sealing, vacuum encapsulation and temperature sensitive microelectronics and materials [10]. A hermetic seal prevents the entering of moisture and contaminants into the package that could cause corrosion and mechanical damage for the sensor element. Due to these special requirements, packaging has become one of the most expensive processes in MEMS manufacturing.

The most important functions of MEMS packaging are:

- 1) Reduce electromagnetic interference
- 2) Dissipate heat
- 3) Minimize coefficient of thermal expansion
- 4) Deliver required power
- 5) Protect the component from environment and contamination

A MEMS component consists of a chip carrier, sensor element and ASIC circuit (Fig 2.5). The sensor element and ASIC circuit are attached by die bonding to the blank substrate on the chip carrier and electrical connection between the sensor element and the ASIC circuit is created by wire bonding [11]. Finally the package is filled with silicone based gel. Then the package is enclosed by hot moulding or left open in some cases.

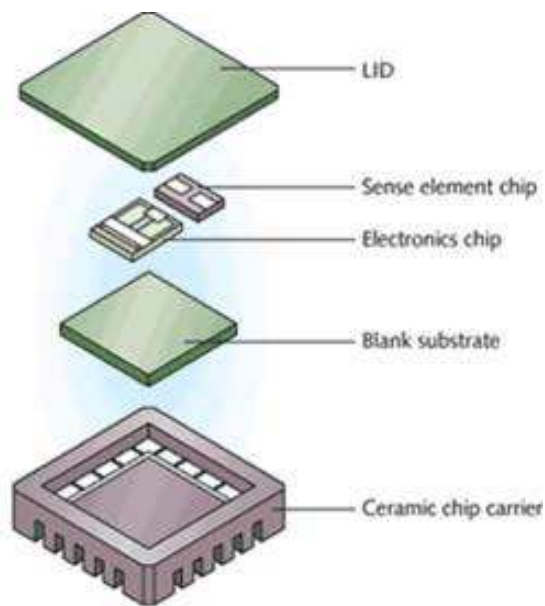


Figure 2.5: *Structure of a MEMS component*

Size of the package is important factor for the MEMS technology. As it can be noticed in Figure 2.5 the size of the package can be much larger than the sensor element and ASIC chip. Nowadays all IC and MEMS packages are surface mounted. SMT (Surface Mounted Technology) can be considered as one of the major innovation in the electronic assembly. Chip Scale Packages (CSP) [12] are bare chip packages with dimensions smaller than or equal to 1.2 times the dimensions of die. A Quad Flat No Leads (QFN) package is nearly chip scale package used by many MEMS manufacturers (Fig. 2.6) [12]. Wafer Level Package (WLP or WL-CSP) [12] is even more miniaturised version of CSP. Its size is equal to that of the die. WLP technology can be considered as an ultimate packaging solution: the device become the package.

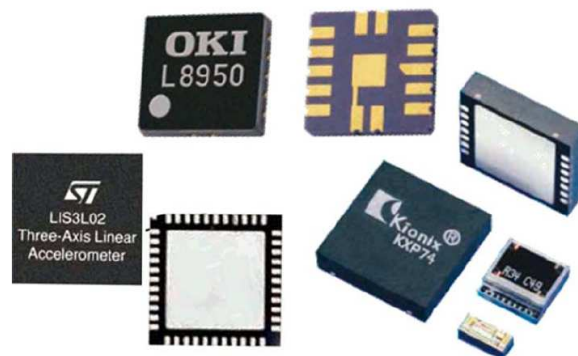


Figure 2.6: *QFN packages*

Silicon, ceramic, steel and polymers like Liquid Crystal Polymer (LCP) are the most common packaging materials in MEMS industry [13]. Plastic non-hermetic packaging materials are inexpensive and moulding techniques are simple. The QFN has eliminated secondary operations like solder ball attachments and simplified the chip carrier [12]. Ceramic packages have high Young's Modulus, and they can be hermetically sealed. Flip Chip or wirebonding is used to connect the chip electrically to the package. Metallic packages can be also sealed and their assembling is easy. However, the pin count is usually lower.

2.2 MEMS Gyroscope's Functional Testing

Testing of MEMS components has a big effect on overall manufacturing costs; therefore it must be fast and efficient. MEMS gyroscope testing includes testing during the product's life-time, validation of the sensor, production testing, calibration and end testing [11].

Reliability testing for the components at the R&D stage can be done in relatively small scale. Reliability testing helps to evaluate failure modes and expected life-time for the product. The goal is to prevent customers getting faulty products. On the other hand, analyzing faulty products can be useful for the process development point of view. Even in consumer electronics a product can be exposed to harsh environments and sudden impacts. Especially temperature and humidity have to be taken into consideration when predicting life-time of an electronic product and its limits of use. Temperature and humidity are usually tested in environmental cabinets. The impact durability is evaluated by mechanical impact testing. [14]

All manufactured gyroscopes go through functional testing before ending up to customers. During the test, proper functionality in specific use environments is evaluated. AEC-Q100 standard [15], which is used by the car-industry, specifies that the operational temperature range of MEMS gyroscopes is -40 °C...125 °C.

End-product testing requires test equipment that can rotate tested wafers or packages around every three axis separately or simultaneously, and monitor the output of the sensors during the test. Most MEMS testing companies use custom made machines because there is no standard equipment for testing.

As opposed to the R&D stage testing and validation, the volume of the tested components is much higher in the end-product testing. Efficient high-throughput testing is an important factor in MEMS test equipment design. Test-on-Tape (TOTA) test method is developed especially for this need [16]. In the TOTA the same test configuration can be used for wafer level packages and single components (Figure 2.7). Wafers or components are placed on the wafer back-grinding tape to optimum pattern and the tape frame is attached to the heater unit which is used to heat the components to a specific temperature. There is no need for complex carriers or test socket boards, thus tested products can be changed easily. Only requirement for TOTA tested product is that the component top is flat so it can be placed easily on the tape. Thanks to increased test throughput, the cost per test is extremely low.



Figure 2.7 *Afore Kronos handler for testing motion sensors in wafers, in diced wafers, or single components placed on tape*

Testing at elevated temperatures creates special requirements for the TOTA tapes, however. The tape has to maintain its adhesion in up to 150 °C. Adhesion between the tape and the silicon wafer or component is a critical issue. In motion testing this is emphasized even more due to induced G-forces. Adhesion strength should be high enough to prevent shifting of the wafer or components but, on the other hand, the devices has to be easily removed after the test, and the adhesive should not leave any residue on the surface of the wafer or packages. Thermal stability and good heat conduction are also important requirements for the tapes.

For evaluating adhesion durability during MEMS sensor testing we must at first understand the complex nature of adhesion. This will be discussed in the next chapter.

3 Adhesion of Surfaces and Adhesive Tapes

The interaction that binds two materials together is called adhesion. Adhesion is widely used in all kinds of applications in households and industry: tapes, paints, glues, bandages, thin film coatings. Liquid glue material, usually polymer based, is called adhesive, and the solid substrate is adherend. The interaction between these two is achieved through three main attractive forces: (i) physical intermolecular interactions, (ii) chemical bonds, and (iii) electric double layer which is created by mobile charges interdiffusing through the bond because of the initial difference of electrochemical potential between adhesive and adherend. Interdiffusion of the macromolecules between two polymers can be considered as a fourth attractive force. [22]

Free surface energy determines how well the adhesive can wet, or spread on, the substrate. Good wetting is required to achieve a sufficient adhesive bond between adhesive and substrate. Surface energy cannot be measured directly but there are indirect methods to do this. Contact angle measurement [17] is one of the most used methods to evaluate hydrophilicity (*hydrophilic* = attraction to water) of the surface. [17]

In a contact angle measurement, a drop of liquid is placed on a solid surface, and the angle formed between three phases, solid (S), liquid (L) and gas (G), is measured optically. The setting is illustrated in Figure 3.1. Components of interfacial tension acts at the the triple interface, but in equilibrium these tensions are in balance. The contact angle (θ) is the angle between the solid surface and the tangent to the liquid surface at equilibrium, which is described by Young's equation:

$$\varphi_{SG} = \varphi_{SL} - \varphi_{LG}\cos\theta. \quad (3.1)$$

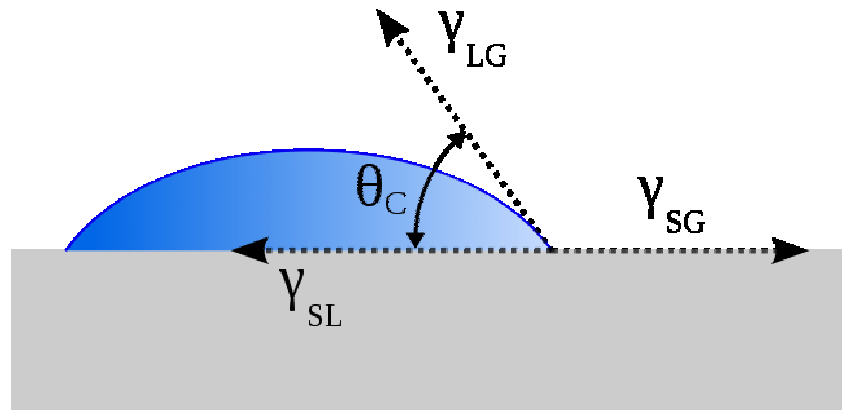


Fig 3.1 *Contact angle*

The liquid is said to wet the surface perfectly, if $\theta = 0$. Wetting does not occur if $\theta > 90^\circ$.

i) Physical intermolecular interactions

Physical intermolecular interactions include weak Van der Waahls forces and mechanical crosslinking with a rough and porous surface. Van der Waahls attraction is the result of momentary dipoles between the surfaces. Mechanical crosslinking can be enhanced by etching and sand blast.

Mechanical interlocking is based on the assumption that roughness of a substrate increases adhesion strength [20]. An adhesive penetrates into the small pours and binds physically to the substrate. If the pours are too small or the adhesive does not wet the surface well mechanical interlocking is not occurring. There is also possibility that the adhesive leaves uncoated voids and therefore joint strength is decreased. Thin and low viscosity adhesive layer has also less contact area on the rough and porous surface that can have a negative effect on adhesion. Especially for materials such as wood, cloth, paper and etched metals and polymers interlocking theory has great importance.

ii) Chemical bonds

Chemical attractive forces are based on chemical bonding through covalent, ionic, or hydrogen bonds [20]. Molecules share valence electrons or there are weak secondary bonds, like in hydrogen bonds. Chemical bonds require that there are reactive functional groups so that electron transfer is possible. Hydrogen bonds need polar hydrogen groups (N-H, S-H-, O-H-).

Organic functionalities, such as isocyanates, carboxyls, amides, amines, hydroxyls, and epoxides, react at the interface and enhance adhesion. Also coupling agents such as chrome complexes, silanes and titanates have similar effect. They form a covalent bond between an inorganic substrate and an organic overcoat. However, adhesion starts to decrease if the concentration of functional groups is too high. [18]

iii) Electric double layer

Two materials with different electronegativities may form an electrical double layer (EDL) that resembles a capacitor [19]. In this electrical double layer electrons transfer from the lower electronegativity material to that of higher electronegativity.

Oppositely charged atoms or molecules attract each other while two particles with the same charge are repulsive. A very simple example of electrostatic adhesion is a rubber balloon that acquires a surface charge when rubbed and sticks to non-conductive surfaces. Electrostatic adhesion is used widely in the coating industry by charging polymer particles negatively while the substrate to be coated is positively charged.

iv) Diffusion theory

Diffusion theory is usually used to explain polymer-polymer adhesion [20]. It claims that the best adhesion is obtained when both adhesive and adherend have mutual solubility. Adhesion is the result of interdiffusion of the macromolecules

between two compatible polymers, and this interdiffusion has been observed to increase joint strength between certain high polymers with time. However, the theory is not applicable for adhesion between incompatible polymers, polymer-metal, metal-metal or metal-glass interfaces.

3.1 Failure Mechanisms of Adhesive Bonding

There are three principal mechanisms of failure [21]: structural, adhesive, and cohesive failure. These three failure mechanisms are shown in Figure 3.2. Structural failure takes place in a substrate material in a region close to the joint. Adhesive failure is interfacial failure in which one of the substrates is separated from the adhesive layer. Internal failure of the adhesive layer is called cohesive failure. Parts of the adhesive remain on the substrate.

Structural and cohesive failures of the pressure sensitive tape are especially disadvantageous in MEMS testing. Adhesive residues on the component can make component handling much more difficult during the testing process. Another problem is ruptured pieces of tape backing that can cover the component or part of it. Components are identified by using machine vision and a dense residue layer can hide the markings completely. This causes easily a fault situation in the automated process.

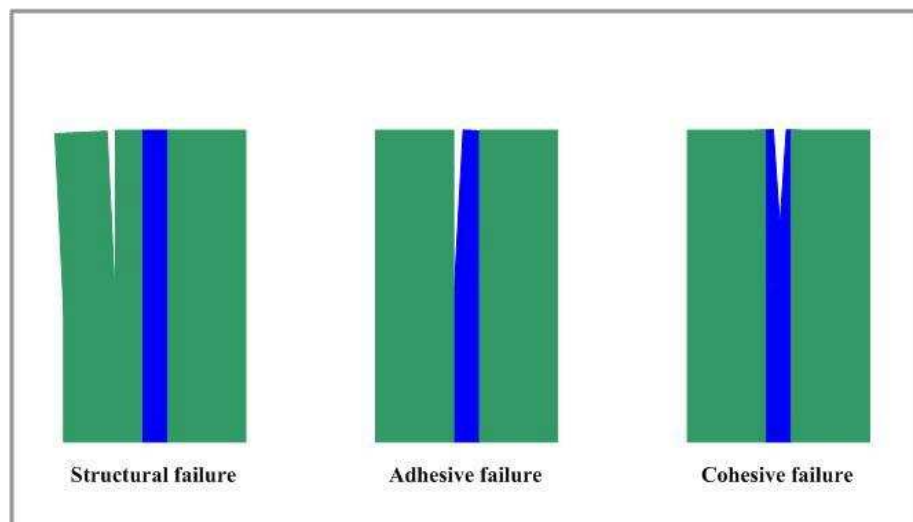


Figure 3.2 Failure mechanisms of adhesive bonding

3.2 Pressure Sensitive Adhesives

Pressure sensitive adhesives (PSA), that are used in the TOTA testing, are commercial wafer back-grinding and dicing tapes that exhibit viscoelastic properties, maintain strong and permanent tack, and have enough cohesive strength to be adhesively removed from a substrate without leaving a noticeable residue [22]. The adhesion is based mainly on the van der Waahls attraction, and no/minimal chemical reaction or physical change occurs. The adhesion strength of the PSA is relatively insensitive to applied pressure. The debonding mechanism, shown in Figure 3.3, involves cavitation at the interface, fibril formation within the PSA and backing deformation [23]. PSAs are classified according to the chemical composition or to the physical form [22].

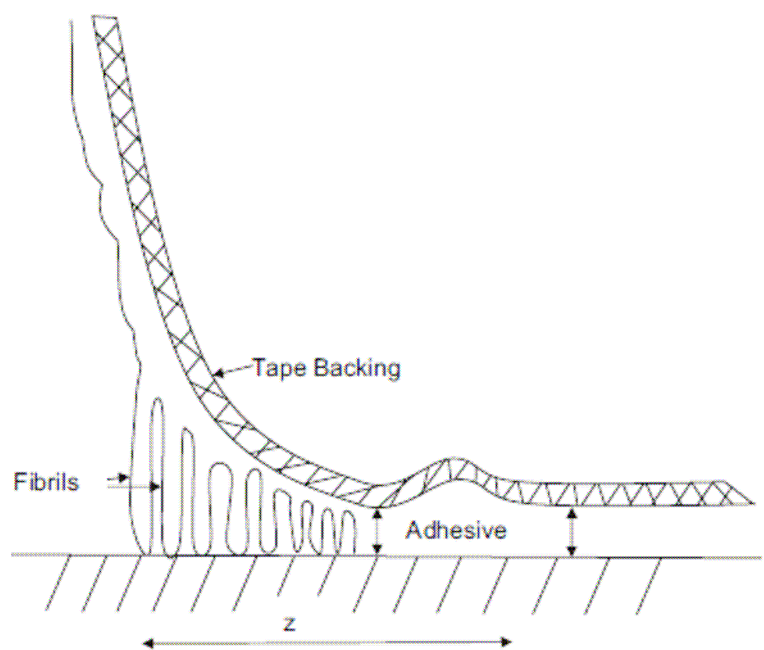


Figure 3.3 Schematic diagram of the peel front of a PSA tape

3.2.1 Classification by Chemical Composition

The classification by chemical composition is based on the main monomer of adhesive [22]. There are three main chemical composition groups in adhesives: rubber adhesives, acrylic adhesives, vinyl ethyl polymers and silicone adhesives. Applications and use environment define which type of adhesive should be used.

Rubber adhesives

The earliest pressure sensitive adhesive applications were mainly based on **natural rubber** adhesives [24]. It is the main elastomer type used in the manufacture of adhesives.

Natural rubber is a polymer of isoprene and it has naturally a very low tack and adhesion properties. Adhesion can be improved by adding tackifying resins, antioxidants, plasticizers or other elastomers to rubber.

The second group of rubber adhesives are **thermoplastic rubbers** [25]. They are composed of A-B-A block copolymers, where A is a thermoplastic polystyrene endblock and B is a rubber midblock of polyisoprene, polybutadiene or poly(ethylene/butylene). The adhesive properties of thermoplastic rubber depend on the styrene concentration and midblock rubber type. The typical styrene concentration range between 10 wt-% and 35 wt-%. Thermoplastic rubbers have low viscosity at elevated temperatures, so their hot-melt processability is very good.

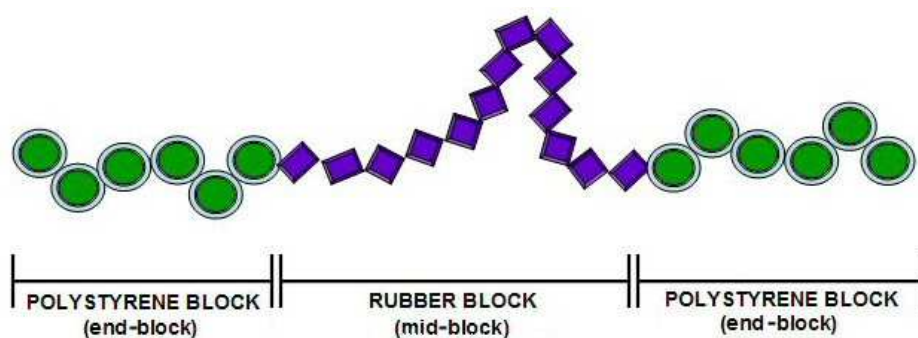


Figure 3.4 Schematic drawing of a thermoplastic rubber, A-B-A block copolymer

The third rubber adhesive group are **butyl rubber and polyisobutylene** [26]. Both are used in PSAs as primary elastomers, tackifiers and modifiers. Butyl rubber is a copolymer of isobutylene with a small content of isoprene. Polyisobutylene is a homopolymer, so it contains only one type of repeat unit.

Acrylic adhesives

Acrylic adhesives [22] have many advantages that make them favourable in PSA applications. Contrary to rubber adhesives, acrylic adhesives can be inherently pressure sensitive and there is no need for additional compounding. They offer a wide range of molecular weights, good tack, low glass transition temperature (T_g), transparency and good resistance to oxidation. These features make acrylic adhesives useful in applications that require clarity, stability on outdoor exposure or stability at high temperatures. Most wafer back-grinding tapes use acrylic adhesive which is modified to improve its properties.

The general pressure sensitive acrylic polymer composition is following:

Main monomer 50-90 w-%

Modifying monomer 10-40 w-%

Monomer with functional groups 2-20 w-%

2-ethylhexyl acrylate (2-EHA) and butyl acrylate (BA) are the main components of acrylic adhesives due to their low glass transition temperature ($T_g < -20$ C) and high flexibility at room temperature. Low glass transition temperature adhesives have better tack. Copolymerization with functional carboxyl groups are used to achieve high adhesive strength and crosslinking with other functional monomers.

Vinyl ethyl polymers

Copolymers of vinyl ethers and acrylates are used as a raw material for producing PSA compounds. Vinyl ether is produced by adding alcohols to acetylene, which is the only method of preparation in industrial-scale production:



The degree of polymerization determines appearance of polyvinyl ethers. They can be viscous oils, tacky soft resins or rubberlike substances. Vinyl ether polymers are usually mixed with other polymers. The adhesive properties are varied by mixing ratio of polymers. [27]

Silicon adhesives

Silicone [28] has two main components: a polymer and a tackifying resin. The polymer is usually polydimethylsiloxane or polydimethyldiphenylsiloxane, which has high molecular weight and contains residual silanol group (SiOH) on the ends of the polymer chains. The resin has a three-dimensional silicate structure which is encapped with trimethylsiloxy groups (OSiMe₃) and contains also some residual SiOH functionalities.

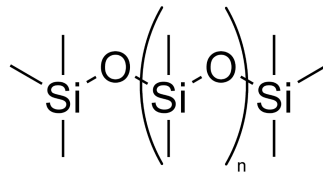


Figure 3.5 PDMS

Silicones have good thermal stability up to 250 °C and its glass transition temperature is low (-120 C). They can be modified in a various ways to achieve different tack, peel adhesion and cohesive strength properties.

3.2.2 Classification by Physical Form

Solution adhesives are replaced to some extent by aqueous and hot melt adhesives, but they still cover almost 50 % of the PSA market and 70 % of the tapes market. Their advantages are easy applying and versatile properties. The major disadvantages are the environmental hazards, high energy processing, lower coating speeds at high coat weights and poor aging resistance.

Hot melt adhesives are tacky and soft materials at room temperature. They are generally based on styrene-isoprene-styrene (SIS) and styrene-butadiene-styrene (SBS) block copolymers tackified with resins. With hot melt adhesives it is possible to achieve high coating speeds, but they lack aging and heat resistance because of the double bonds of the polymers.

Aqueous adhesive emulsions differ from solution adhesives with their much higher molecular weight which ($> 10^6$). Molecular weight does not have effect on viscosity in the aqueous adhesives either. The problem of these adhesives is the difficulty of coating and poor resistance to water.

Calenderable adhesives are fully solid compounded materials processable on calendaring equipment. They are composed of natural rubber or natural rubber/SBR blends, compounded with tackifying resins.

Radiation curable adhesives are 100 % solid reactive coatings, and consist of acrylic functionality containing oligomers and monomers. They have sufficiently low viscosity, so using conventional coating methods is possible. UV and EB radiation are most used for curing. Radiation curable adhesives are very useful in applications where the adhesive coating must be applied over a small area. [29]

3.2.3 Composition

Polymer composition of adhesives varies in terms of adhesive type. Natural rubber adhesives are usually heavily compounded with extra additives while acrylic adhesives are 100 % pure polymer.

Additives

Additives are used to modify properties of adhesives. By adding different amounts of tackifiers, plasticizers, fillers, cross-linking agents, surface active agents, thickeners and antioxidants, same type of adhesives can have substantially different physical and chemical properties.

i) Tackifiers

Tackifiers [30] produce typical pressure sensitive properties for the adhesive composition. Their molecular weight ranges between 500 and 2000, and molecular weight distributions are broad. The softening points vary from 50 °C to 150 °C. There are two main classes of tackifier resins: rosin derivatives and hydrocarbon resins. Rosin derivatives are primarily rosin esters formed by rosin acids and polyhydric alcohols. Hydrocarbons are polymers which are polymerized from monomers (C₅ and C₉) of petroleum, coal and wood.

A tackifier resin must be compatible with the base adhesive polymer. Its molecular weight should be very low and glass transition higher compared with the base polymer. Especially for the natural rubber adhesives tackifiers are important, because without them rubber adhesives would have very low tack. Acrylic adhesives do not require tackifiers so much due to their different flow properties and surface energy.

ii) Plasticizers

Additives that decrease the glass transition temperature and cohesive strength or increase elongation are considered as plasticizers [31]. The most common plasticizers in PSAs are hydrocarbon oils, liquid polybutenes or polyacrylates, phthalates and lanolin.

iii) Fillers

Fillers [31] are used to add special properties to pressure sensitive adhesive. For example CaCO_2 makes elastomer easier to handle and reinforcing fillers increase tensile strength and stiffness. By using pigments such as TiO_2 or ZnO , PSA's colour can be altered or its cost can be reduced. They can also improve the performance of the adhesive.

iv) Cross-linking agents

Polymer chains bind together through cross-linking during the polymerization or after, on heating or aging. Cross-linking agents [31] forms this bind and are thus very important additives in pressure sensitive adhesives. There are number of cross-linking agents: carboxylic, hydroxyl, epoxy, amide, amine and isocyanate. Also zirconium, zinc or titanium compounds, and peroxides are applicable for most elastomers.

v) Surface active agents

Surface active agents [31] stabilize the polymer during emulsion polymerization for stabilization against mechanical or chemical coagulation. These surfactants contain at least one hydrophilic and one hydrophobic group. Surfactants can be also charged (anionic, cationic, amphoteric). Anionic sulfates, sulfonates, carboxylates are the most common surface active agents.

vi) Thickeners

An adhesive coating must spread smoothly over the substrate. Viscosity can be increased by various thickeners [31], for example by polyacrylate and cellulosic thickeners or polyvinyl methyl ethers. Cellulosic thickeners and polyvinyl methyl ethers have less effect on adhesive properties.

vii) Antioxidants

Antioxidants [31] stabilize an adhesive against oxidation and heat and light degradation. Secondary amines and their derivatives, alkyl phenols, dithiocarbamates and compounds of quinoline are commonly used antioxidants.

3.2.4 High Temperature Resistant PSAs

Heat resistance of adhesive bonds is the property of maintaining the bond at high temperatures. The critical limit is considered as a temperature at which the bond loses this property. At this flow temperature failure of the bond occurs even without the effect of an external force. [32]

Polymers have inherently weak heat resistance. Most polymers decompose already at 90 °C to 120 °C temperatures, such as PVC, polyethylene, polycarbonate and polyester. Heat resistant adhesives have high fluorine or aromatic ring content, or high cross-linking density [33]. Fluorinated plastics with varying fluorination levels have a broad temperature performance range. For example polytetrafluoroethylene can be used over a temperature range of -200 to 260 °C.

Thermal resistance of PSAs can be improved also by cross-linking. Silicone adhesives have low cohesive strength above 150 °C, but through cross-linking they can resist temperatures above 180 °C. Crosslinked acrylate and natural rubber adhesives can be used at temperatures up to 155 °C.

Commonly cross-linking is made thermally through the employment of alkyl phenolic resins, chemically through peroxides or di- or tri-isocyanates, and physically by electron- or gamma-ray irradiation.

3.3 Adhesion Testing

The mechanical adhesion strength of adhesives is the information that is the primary concern for most users. One typically needs to know how large force is required to detach an adhesive from a substrate. There are various methods for the testing of adhesion strength: peel test, pull-off test, tack test and scratch test. The problem in many of these tests is that they give more qualitative than quantitative information, and comparing results between the tests is difficult, because of different standards and units of measurement between the countries.

Organization for Standardization (ISO), British Standards (BS) and the American Society for Testing and Materials (ASTM) are globally recognised organizations [34]. They develop and deliver international voluntary consensus standards, improve product quality, safety, and build consumer confidence by providing test methods, specifications, guides and practices that support industries and government worldwide.

There are also some more specialised organizations, such as Fédération Internationale des fabricants et transformateurs d'Adhésifs et Thermocollants sur papiers et autres supports (FINAT), which concentrates only on the self-adhesive testing [35].

The adhesion strength depends on many variables such as the test method, temperature, peel rate and angle, adhesive chemistry, adhesive thickness, aging, and stiffness and thickness of the adhesive packing [36]. In addition, the properties of the substrate have also effect on the adhesion.

Comparative tests between different adhesives should always be performed by using the same test set up and parameters. Combining different standards and test methods will greatly change the results.

3.3.1 Peel Test

Peel tests [37] are commonly used in the tape industry. The test setup is quite simple: an adhesive is peeled at constant peel rate and the detaching force is measured. The adhesion strength is the average peel force per unit width. The test set up can be altered by changing peel rate, temperature, peeling angle, and other parameters.

Different tests are commonly named after the used angle: T-peel, 90° peel and 180° peel test (Figure 3.6). In the T-peel the adhesive is placed between the two substrates which are pulled in opposite directions. In the 90° and 180° peel tests PSA tape is peeled at 90° and 180° angles at constant peel rate. The angle affects on the required peel force. The perpendicular peeling measures only the tensile force, but in the 180° peel test both shear and tensile stresses occur.

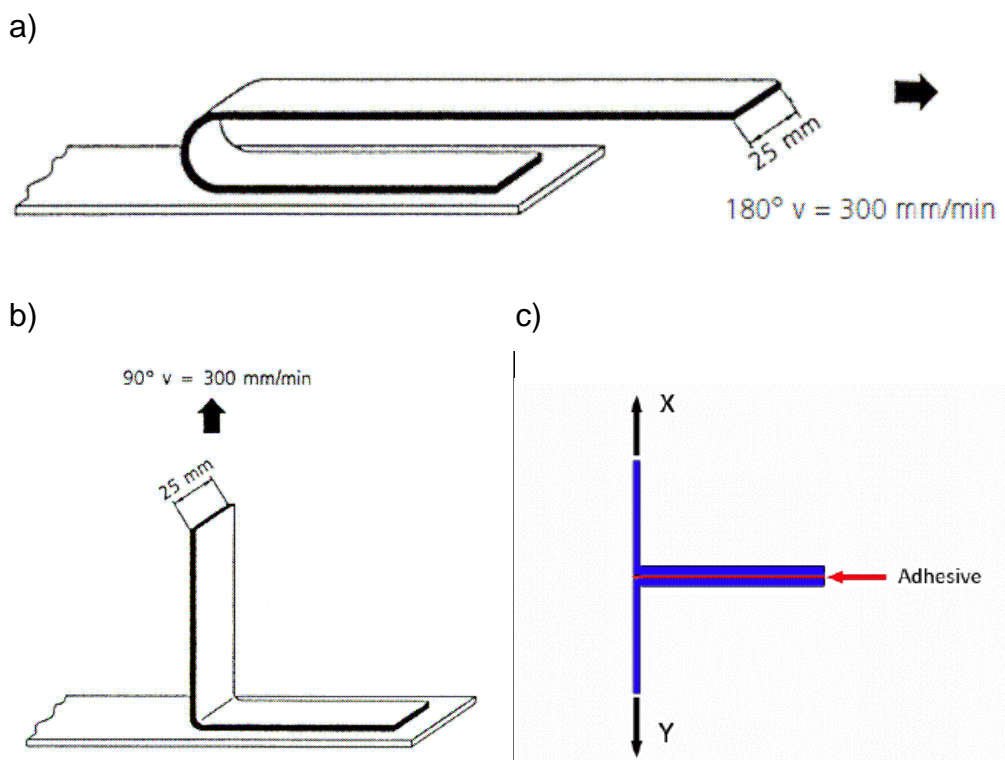


Figure 3.6 a) 180°- peel test b) 90°- peel test c) T-peel test. Peel rate 300 mm/min is based on the FINAT standards FTM1 and FTM2

3.3.2 Pull-off Test

Pull-off test [38] is used to determine either the perpendicular force that a surface area can bear before a plug of material is detached, or whether the surface remains intact at a predefined force. The test is developed initially for testing coatings of metal substrates, but practically it is applicable to all rigid surfaces. Test parameters have to be optimised for each tested materials. In flexible substrates the tensile stress will concentrate on the edges [39].

There are different kinds of pull-off test equipments, but the basic idea in most of them is similar to that shown in Figure 3.7. An adhesive sample is attached firmly on the stud, for example by epoxy, and the stud is pulled up by the force F . The force is applied by increasing the pressure in the gasket. The adhesion strength is the perpendicular force per area of the adhesion zone.

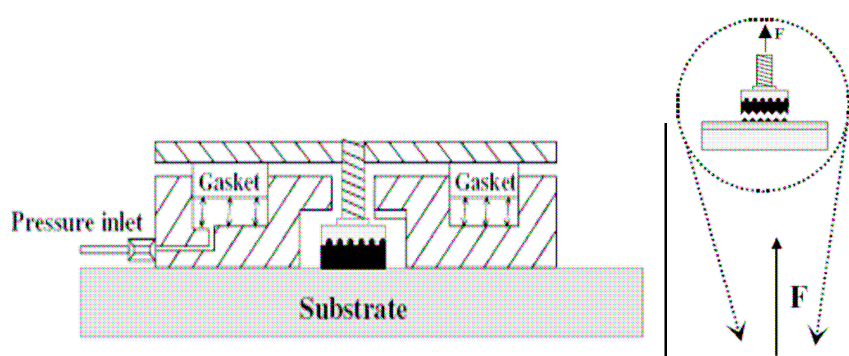


Figure 3.7 *Pull-off test*

3.3.3 Tack Test

The tack test [40] is very similar to the pull-off and peel tests, but it concentrates more on the failure process of the adhesion. The adhesive is separated slowly from the substrate and simultaneously the interface is monitored optically. At the same time the work required to detach the object is measured by the determination of the area under the load-displacement curve. Compared to the pure adhesion strength measurement, the detachment work contains more information about the failure mechanism of the adhesion. Schematic of the tack test is presented in Figure 3.8.

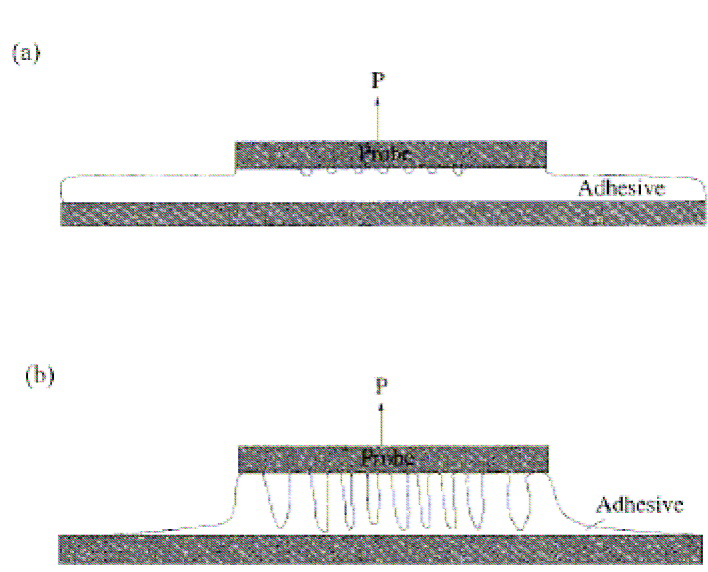


Figure 3.8 Schematic of a tack test, from a) cavitation to b) fibrillation

3.3.4 Tape test

The tape test [41] is a simply and widely used adhesion test for thin films and coatings. An adhesive tape is applied on the surface of the film and pulled off rapidly. The adhesion strength is determined only qualitatively. Adhesion is 'poor' if the film is completely removed, 'adequate' if the film is partly removed or 'good' if the film is not removed at all. This method is a fast and cheap first-hand test, but for scientific purposes far too simple.

3.4 Analysis/Comparison of the Adhesion Test Methods and Parameters

The test methods, introduced in Chapters 3.3.1-3.3.4, have their own advantages and disadvantages. The aim is to find an accurate and quantitative test method to determine the adhesion strength. In choosing the right method, it is also important to consider conditions of use of the adhesives. In this case, PSAs are used in the TOTA handler for MEMS sensors to attach the tested wafer or component on the frame. Applied forces are not especially high, and tested wafers of components are small and light, but during the thermal characterization temperature can rise up to 150 °C that will affect the adhesion strength.

Basically, there are two methods to test the effects of temperature on the adhesion durability. Pull-off test or peel test can be performed inside a temperature cabin, on a temperature plate or for pre-heated samples. An alternative method is to use a hot-air blower, but keeping the temperature stable is difficult and most probably hot blowing air would dry the adhesive fast.

Choosing the peel test angle is one essential parameter. As discussed before, 90° peel applies only tensile stress to the tape. Thus it is more comparable to the pull-off test. However, 90° angle requires a separate peel arm configuration; for example, the tape is attached on a freely rotating wheel. Although the wheel would have very low friction, it adds an extra component to the system and makes the strength analyses more complex. The 180° peel test is more straightforward to perform, but it has both a tensile and a shear stress component. Perpendicular angle results higher adhesion strength values than 180° angle.

The peel rate varies exceedingly among different standards. For example, ASTM's 180° peel test (D-903) uses peel rate of 152 mm/min (6 in./min) and FINAT's similar test uses 300 mm/min. A high and low peel rate causes a different mode of detachment, in which also temperature has a role [42]. At very low peel rate and high temperatures, a cohesive failure is more common; the adhesive experiences viscous flow and it is drawn out into long fibrils (see Figure 3.3). Eventually fibrils fail in tension and leave residue of the adhesive on the both

surfaces. At high peel rate or less elevated temperatures, the adhesion fails before the fibril formation, and the adhesive remains on the one of the surfaces. These modes of detachment and the peel force are stable as long as the peel velocity is maintained constant.

Stiffness of the tape can cause difficulties in the peel test. A thick tape does not bend easily and bending stresses the backing and, eventually, leads to structural failure. For the rigid PSA the pull-off test is a better option to determine the adhesion strength. It is also much easier to conduct for the small MEMS packages than the peel test which would require larger surface area.

The advantage of the pull-off test is that the adhesion strength is easy to evaluate by dividing the average peel force by the adhesion area. There is only tensile stress affecting the adhesion and the tape is firmly attached to the plate, so multiple stress components or bending are not complicating analyzing. However, it must be made sure that the test head to which the package material is attached must be perfectly perpendicular to the interface substrate. In case there is even a small tilt the surface does not attach evenly to the sample.

Tape test does not bring any substantial benefits compared to peel and pull-off tests. The tape test can not be considered in this case because it gives only qualitative results.

Due to extremely low forces between the MEMS components and tapes and small surface area of the packages, the pull-of test was chosen to the adhesion test method in this work. Another advantage is that the test set-up resembles very much the real component testing circumstance.

Some sacrifices must be done to keep the test throughput high, because there are an extensive amount of variables (tape, package, temperature and time) that means that there is a considerable number of different treatments which in addition have to be repeated at least three times to make statistical assumptions from the results. Consequently pre-annealing of the tape samples is the only

reasonable method. Changing the samples will be made easy by using high-strength double-sided tape to attach the sample to the test plate.

3.5 Statistical Analysis

Analysis of variance (ANOVA) is a useful statistical tool to study significance of different test factors and interaction between them [43]. The adhesion strength experiment to be discussed later on in this work was designed as a full factorial experiment, where the effect of four main factors (i.e. variables) and their interactions on the measured adhesion strength is investigated. These variables are surface material, tape, temperature and annealing time, each having a different amount of levels (3 x 4 x 5 x 3).

As a post hoc test for ANOVA, Bonferroni all-pairwise comparison test was used to compare means of different main factors. Although the test is quite simply, it is very powerful for analysing extensive experimental data when there are more than two factors with multiple levels affecting the measured output.

3.5.1 Analysis of Variance

Multiple factor ANOVA is an extension of two-factor analysis of variance. Multiple factors make computations more laborious while the general principle remains the same. In the analysis of variance the group means of the depended factor are compared. Means of different treatment combinations must be significantly different in order to reject the null hypothesis H_0 which claims that all means are the same:

The first null hypothesis is the null hypothesis of no interaction between the factors:

$$H_0 : (\alpha\beta)_{ij} = 0 \quad i = 1, 2, \dots, a; j = 1, 2, \dots, b;$$

If H_0 is not rejected, then the null hypothesis of no difference among levels of factor A is tested,

$$H_0^I = \mu_{1..} = \mu_{2..} = \dots = \mu_{a..}$$

$$H_0^I = \alpha_{1..} = \alpha_{2..} = \dots = \alpha_{a..},$$

and the null hypothesis of no difference among levels of factor B

$$H_0^{II} = \mu_{.1} = \mu_{.2} = \dots = \mu_{.b}$$

$$H_0^{II} = \alpha_{.1} = \alpha_{.2} = \dots = \alpha_{.b}$$

These tests are for main effects. Also levels of factor A for each level of factor B and vice versa must be compared. If the null hypothesis of any of these tests is rejected H_1 will take effect:

$$H_1 = \mu_i \neq \mu_j \text{ for some } i \text{ and } j.$$

In practice, the variance of depended factor is subdivided into components $SS_{\text{treatments}}$ and SS_{error} . The variance is quantified by the Sum of Squares (SS) identity. The total variation of the studied population is divided to those that are attributed to each one of the treatments ($SS_{\text{treatments}}$) and the part that remains unexplained (SS_{error}):

$$SS_{\text{TOT}} = SS_{\text{TR}} + SS_E \quad (3.2)$$

$$SS_{\text{tot}} = \sum_{i=1}^k \sum_{j=1}^{n_i} (Y_{ij} - \bar{Y} \dots)^2 \quad (3.3)$$

$$SS_{\text{TR}} = \sum_{i=1}^k n_i (\bar{Y}_i - \bar{Y} \dots)^2 \quad (3.4)$$

SS_{TR} measures variability in data attributable to the use of different levels of factor k.

$$SS_E = \sum_{i=1}^k \sum_{j=1}^{n_i} (Y_{ij} - \bar{Y}_i \dots)^2 \quad (3.5)$$

SS_E measures variability in data due to random or unexplained sources.

In case these variances differ from each other significantly, the null hypothesis is rejected. The F-test (Eq. 3.6) is used to compare the variances and determine the probability p at which H_0 can be rejected. The 5 % risk level or less is commonly used in engineering applications:

$$F = \frac{\text{variance between items}}{\text{variance within items}} = \frac{MS_{Tr}}{MS_E} . \quad (3.6)$$

The general two-way analysis of variance table is shown in Table 3.1 [43]. Source of variation column contains all experiment variables and their combinations. Degrees of freedom (DoF) is the number of values in the final calculation of a statistic that are free to vary. For example, the DoF of the mean of two values is one. Sum of squares is calculated with Equation 3.3. Furthermore one gets mean square (MS) by dividing SS by degrees of freedom.

Table 3.1 ANOVA table for the two-way classification design with fixed effect

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F	P
A	$a - 1$	SS_A	$\frac{SS_A}{a - 1}$	$\frac{MS_A}{MS_E}$	P_A
B	$b - 1$	SS_B	$\frac{SS_B}{b - 1}$	$\frac{MS_B}{MS_E}$	P_B
AB	$ab - 1$	SS_{AB}	$\frac{SS_{AB}}{(a - 1)(b - 1)}$	$\frac{MS_{AB}}{MS_E}$	P_{AB}
Error	$ab(n - 1)$	Subtraction	$\frac{SS_E}{ab(n - 1)}$		
Total	$abn - 1$	SS_{Tot}			

3.5.2 Bonferroni All-pairwise Comparison Test

In case analysis of variance results that H_0 is rejected we can conclude that at least two of the population means differ in value. However, it does not give information which of the population means can be regarded as being different. Bonferroni pairwise comparison test can be used to compare pair of means [44].

The number of possible pairs of means and conducted tests is $\binom{k}{2} = k(k-1)/2$:

$$H_0 = \mu_i = \mu_j$$

$$H_1 = \mu_i \neq \mu_j.$$

A pooled T-test is used to test the hypothesis:

$$T_{N-k} = \frac{\bar{Y}_i - \bar{Y}_j}{\sqrt{MS_E \left(\frac{1}{n_i} + \frac{1}{n_j} \right)}} \quad (3.7)$$

The critical point for two-tailed test at the α level of significance is

$$cp = t_{N-k, 1-\alpha/2} \sqrt{MS_E \left(\frac{1}{n_i} + \frac{1}{n_j} \right)}. \quad (3.8)$$

The null hypothesis is rejected whenever $|\bar{Y}_i - \bar{Y}_j|$ exceeds the critical point.

The results of the Bonferroni test are presented in a table, such as that shown in Table 3.2, that includes comparison tests for all main factors. Means of different factor levels are organized in descending order, and beside the mean column there are column which shows homogenous groups (A, B, etc.). In case the means of two or more factor levels are not significantly different (assuming 5 % risk level, which is denoted as $\alpha = 5\%$) they belong to the same group.

Table 3.2 *General way to present Bonferroni test results*

Bonferroni All-Pairwise Comparisons Test of depended factor x for factor k

Factor k	Mean	Homogenous groups
level 1	0.1010	A
level 2	0.0489	B
level n	0.0272	C

Alpha 0.05 Standard Error for Comparison VARIES
 Critical T Value 2.400 Critical Value for Comparison VARIES
 Error term used: Error, 692 DF
 All 3 means are significantly different from one another.

4 Purpose of the Thesis

This thesis is a contract study for a Finnish MEMS testing company Afore. Afore has developed a wafer-level Test-on-Tape method for evaluation MEMS motion sensors. Kronos is a TOTA handler for accelerometers and yaw rate sensors with infinite turn angle, all turning axes. In Kronos motion testing can be combined with thermal characterization. However, it is unclear how high temperatures affect the adhesion between the adhesive tape and the wafer or components.

The main object of this thesis is to evaluate the adhesion durability of different Test-on-Tape adhesives during thermal characterization, and to analyze their suitability for the wafer-level or package level functional characterization. Before the adhesion evaluation, thermal stability of the tapes will be studied by annealing them at high temperatures. This study aims to determine annealing temperatures and times for the adhesion testing and to eliminate poor quality tapes from the follow-up evaluation. Short- and long-time thermal stability, adhesion strength and detachment work between typical MEMS gyroscope package materials (silicon, polymer, and brushed steel), and residues are used as evaluation criteria.

Examinations covered the following temperature and time frames: 25, 80, 100, 125 and 150 °C and 30, 60 and 90 min. In addition, the investigation of adhesive residue will be carried out for the adhesives. Optical microscopy is used for the inspection of surface purity.

Experimental work has been carried out in the Aalto University School of Electrical Engineering, in the Electronics Integration and Reliability Unit.

5 Material and Methods

Chapters 5.1-5.2 present studied surface materials of different MEMS packages and wafer back-grinding tapes. Chapters 5.3-5.5 include descriptions of the used experimental methods in the thermal stability test, adhesion evaluation and residue analysis.

5.1 Surfaces Used to Represent Different MEMS Packages

Adhesion of the test tapes was studied on three different kinds of surfaces that we chosen to represent typical MEMS packaging materials including the silicon wafer. Studied MEMS packages are (A) a silicon wafer level package (WLP), (B) a polymer chip scale package and (C) a stainless steel lid (of a ceramic package). All three surfaces are presented on Figure 5.1

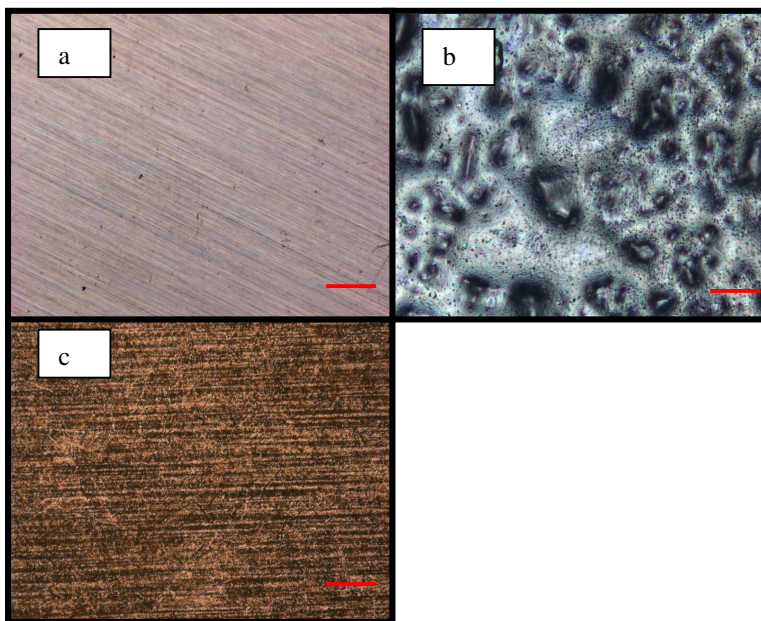


Fig. 5.1 Studied MEMS package surfaces a) silicon b) polymer c) brushed steel (scale bar 200 μm)

Silicon is metalloid and the most used component in semiconductor devices. In WLP form silicon is grey, smooth, reflective and brittle material, much similar to glass. Polymer and brushed steel has much rougher surface than silicon has. Polymer has pores which sizes vary between 20 and 200 μm . Steel's surface roughness is lower and lines produced by brushing go straight over the surface.

5.2 Heat Resistant Tapes

Studied PSAs were commercial surface protective tapes used in wafer back-grinding and dicing processes in integrated circuits manufacturing. Ethyl vinyl acetate (EVA) is used as a base film material in Tape A, polyethylene terephthalate (PET) in tapes B, C and D and polyolefin (PO) in Tape E, which is a prototype tape. PET is used as a protective film in all five tapes. Dicing tapes D and E are both UV curable tapes, thus they lose their tack when exposed to UV light. Wafer back-grinding tapes A, B and C are non-UV tapes.

Because manufacturers did not provide information about the type of the adhesive, it was determined by a Fourier transform infrared spectroscopy (FT-IR) spectroscopy. Butyl acrylate group, which is one of the main components of acrylic adhesives, was found from all adhesives. More specific analysis of the chemical composition was not carried out because it would have been out of scope of this work.

The thickness of the adhesive layer was 20 μm in the wafer back-grinding tapes and 5 μm in the dicing tapes. The total thickness of the tapes varied from 71 to 166 μm .

Table 5.1 Specifications of the tapes as given in their data sheets Adhesion strength is determined by the 180 degree peel test for a 25 mm wide tape strip

Tape	Protective film	Base film	Adhesive	Colour	Total thickness μm	Adhesive layer thickness μm	Adhesion N/25mm
Back-grinding tapes							
A	PET	EVA	Acrylic	Blue	166	20	1,76
B	PET	PET	Acrylic	Transparent	100	20	8,32
C	PET	PET	Acrylic	Transparent	71	20	0,29
UV-dicing tapes							
D	PET	PET	Acrylic	Transparent	105	5	5,796/ 0,036**
E*	PET	PO	Acrylic	Milky white	85	5	4,440/ 0,0864**

*prototype **before/ after UV

5.3 Thermal Stability of the Tapes

In order to set the values of time and temperature parameters in the following adhesion strength evaluation, the temperature tolerance limit as a function of time was investigated. Temperatures of 80, 100, 125 and 150 °C were used and annealing times were 30, 60 and 90 min. Tapes were cut in 2.5 x 2.5 cm pieces, cover film was removed and the samples were placed into the oven (Heraus). Every 30 minutes one test sample of each tape type was removed from the oven and the physical condition was qualitatively evaluated by visual inspection and using an optical microscope. Condition of the tapes was classified in three groups: Good, Moderate, and Poor.

The tapes that did not show any significant changes in the appearance after 2 h annealing at the temperature of 150 °C, were exposed for longer annealing times in order to find out if any changes would be expected soon after the time region of interest. The temperatures in the long annealing tests were the same. Annealing times of 4, 8, 12 and 24 hours were chosen. After the annealing the tape surface was investigated again by employing the optical microscopy to find out changes in the appearance.

5.4 Adhesion Strength Evaluation at Different Temperatures

Adhesion strength of the tapes on the three studied surfaces were evaluated by employing the pull-off test method. Annealing temperatures and times were:

0: RT as a reference	25 °C
1: Low temperature	80 °C
2: Moderately elevated temperature 1	100 °C
3: Moderately elevated temperature 2	125 °C
4: Highly elevated temperature	150 °C

Annealing times per temperature:

- | | |
|-------------|--------|
| 1: Short | 30 min |
| 2: Moderate | 60 min |
| 3: Long | 90 min |

Adhesion testing was carried out in the laboratory of polymer technology with the help of the Instron 33R tensile tester using a 100 N load cell. Measured adhesion strength of actual MEMS packages would have been too small with regard to the resolution of the instrument. For example the top surface area of the smallest MEMS packages (e.g. the Quad Flat Non-lead (QFN) package) are well below 10 mm². Thus the adhering surfaces of MEMS packages were replicated with larger pieces of the same materials or multiple packages attached side by side of a flat surface to form larger surface area. Instead of using a single WLP, six such packages were placed in an array of 2 x 3 packages to increase the total area to 73.6 mm². Particular attention was paid on the placement of the packages to gain a uniform alignment of the top surfaces. The QFN package was replaced with a 100 mm² BGA package with a similar polymer encapsulation and surface topography. The top area of the ceramic package was 25 mm² which was considered large enough and it was the only one that was used in the test “as such”. These packages were glued by Loctite Super Attak Control on a steel cylinder (height 21 mm, diameter 10 mm), which was designed to fit the tensile test adapter. Three test heads are presented in Figure 5.2

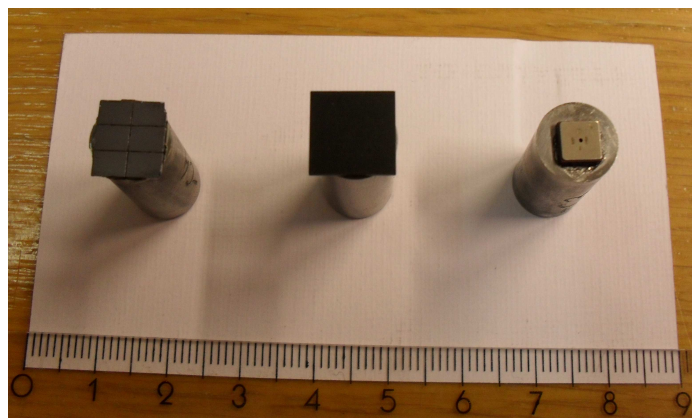


Figure 5.2 Package test heads for the adhesion testing. Silicon surface (left) has been built from six WLPs, small polymer package is replaced with a BGA and ceramic/stainless steel package is used as such

A 15x15 mm tape sample was attached on the stainless steel disc with the 0.21 mm thick and 19 mm wide Scotch 3M™ Double Coated Tape with 3M™ Adhesive 375, which adhesion strength is 16 N / 10 mm for stainless steel (180 degree peel test) [45]. The test setup is shown in Figure 5.3

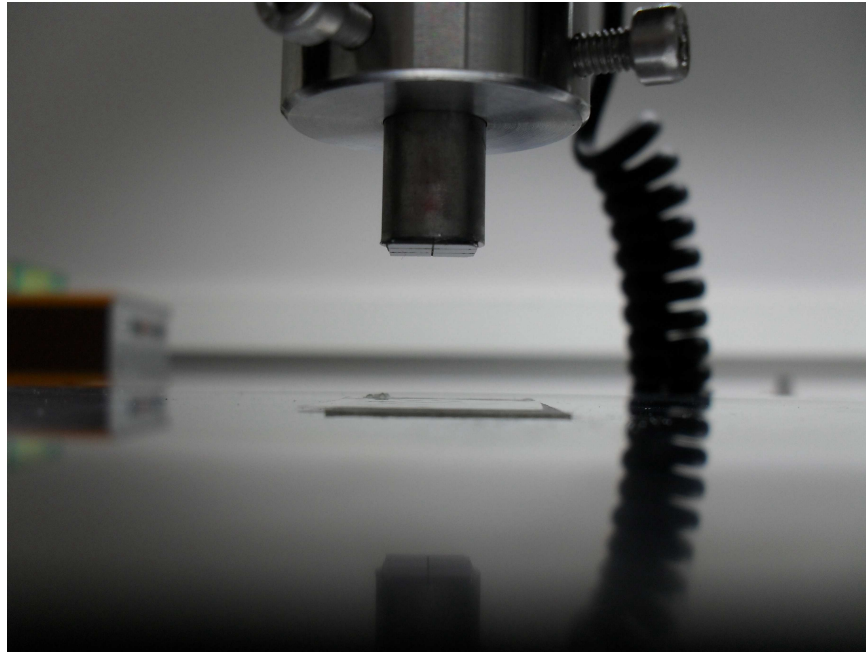


Figure 5.3 *Adhesion strength test setup*

At the first phase of the test the tensile tester was programmed to apply a compressive load of 5 N on the component at the speed of 0.05 kN / min. Then the package was pulled off at the constant speed of 0.1 mm / min and load and extension were measured during the test. The detachment of the package from the tape occurs at the peak of the load curve. Every test was repeated 5 times always with a new (equivalently pre-treated) specimen, and the surface of the specimen holder was purified with ethanol between the tests.

The measured data was post-processed with Microsoft Excel. The actual adhesion strength was evaluated by dividing the load by the total area of the specimen ($\text{N} / \text{mm}^2 = \text{MPa}$). Strain energy is considered as detachment work, based on the assumption that the energy required to remove the component from the tape is made mostly against the glue fibrils. The strain e is the ratio of deformation to the initial dimension of the adhesive layer:

$$e = (x - D)/D \quad (5.1)$$

where D is the thickness of the adhesive layer and x is the displacement.

The strain energy [Nm/mm³] was determined from the adhesion strength – strain curves by employing the Trapezoid Rule (Fig. 5.4), according to which the area under the curve (AUC) is divided into series of trapezoids and these areas are summed:

$$AUC = \sum h_{avg} * width \quad (5.2)$$

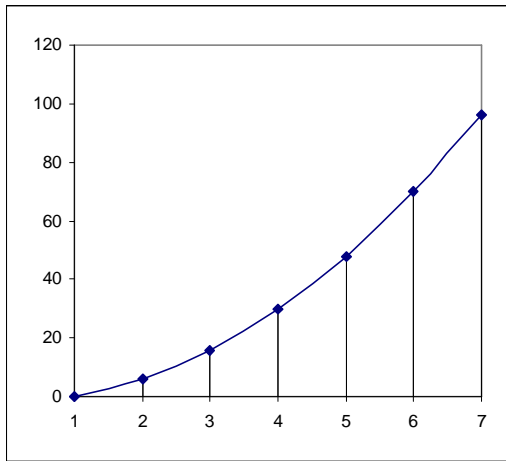


Figure 5.4 *Trapezoid Rule*

5.5 Residue Analysis

MEMS packages were laid on a tape that was attached to the frame (diameter 27.7 cm). Before laying packages on the tape their surfaces were purified with ethanol to avoid grease and other impurities to promote staining. After the release of the packages from the tape surface purity was inspected by employing the optical microscopy.

The attached packages and the tape were heated 30, 60 and 90 minutes at the temperatures of 80, 100, 125 and 150 °C. After heating the packages were

removed from the tape by lifting them up with tweezers and simultaneously pushing them from under the tape with tweezers. Surfaces were investigated by an optical microscope using magnification of 50x and 100x.

Tape residues on the packages were rated visually on a scale of one to five. Rate one indicates that there are no residues on the surface and rate five that the surface is nearly completely covered with residues. The references for all five residue levels are presented on Figure 5.5.

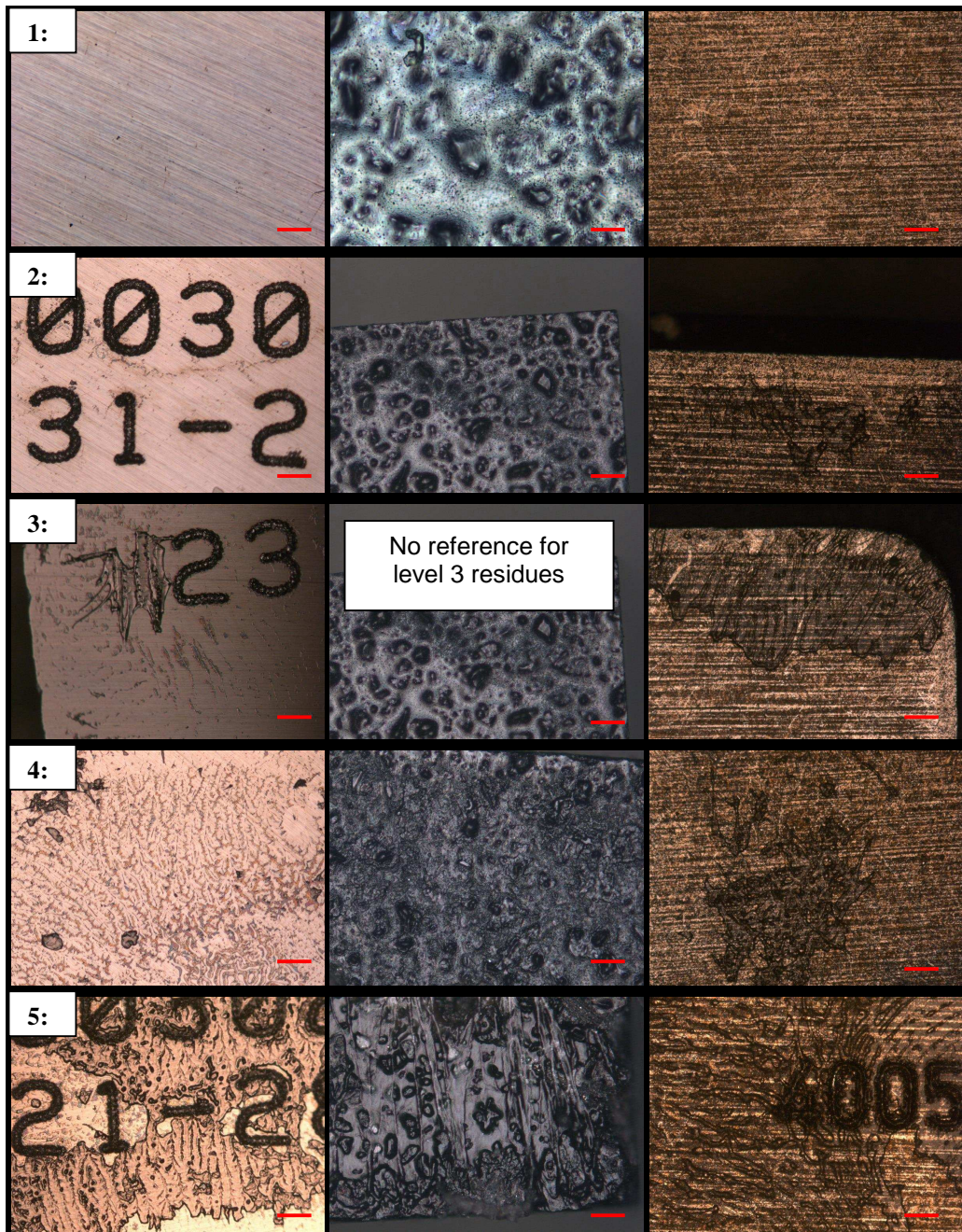


Figure 5.5 Silicon (left), polymer (middle), steel (right) (scale bar 200 μ m)

1: Clean surface

2: Few adhesive stains on the component

3: Lot of adhesive stains and/or few medium pieces of tape packing

4: Large areas of component are covered with adhesive and/or there are small pieces of tape backing, component markings are difficult to read

5: Surface of a component is covered almost completely with adhesive and large pieces of tape backing, component markings are unreadable

6 Results

The most important results of this work are concluded under this chapter. The results are analysed in Chapter 7.

6.1 Thermal Stability of the Tapes

The temperature tolerance at 80, 100, 125 and 150 °C was investigated as a function of time: 30, 60 and 90 min. The physical condition was qualitatively inspected visually and by employing optical microscopy, and classified into three categories according to the principles described in chapter 5.3. The condition is illustrated with colours:



Tape A

Table 6.1 Thermal stability of Tape A

Tape A	RT	80 °C	100 °C	125 °C	150 °C
30 min	Green	Yellow	Red	Red	Red
60 min	Green	Yellow	Red	Red	Red
90 min	Green	Yellow	Red	Red	Red

Figure 6.1 shows the reference surface of Tape A. Tape bends strongly when it is removed from the roll. When the tape is heated it bends even more. Due to bending the thermal stability at 80 °C was rated moderate

Evidence of changes in the adhesive of the tape were not found in optical microscopy investigations, but the EVA base film started to show signs of melting by warping excessively after about 30 minutes heating at 100 °C. “Wavy patterns” could be seen even with bare eyes. Longer heating time did not have essential difference on the deformation.

The behaviour of the EVA base film at temperatures above 100 °C indicates that Tape A is not suitable for MEMS component testing at elevated temperature. Based on the short thermal stability test, Tape A was not included in the further evaluations.

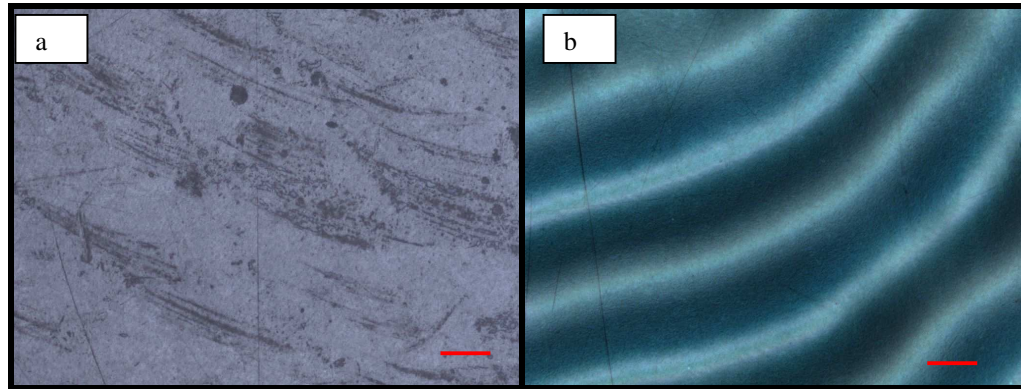


Figure 6.1 a) Tape A reference b) Deformed base film after 30 min heating at 100 °C (scale bar 200 μm)

Tape B

Table 6.2 Thermal stability of Tape B

Tape B	RT	80 °C	100 °C	125 °C	150 °C
30 min	Green	Green	Green	Green	Green
60 min	Green	Green	Green	Green	Green
90 min	Green	Green	Green	Green	Green
4 h	Green	Green	Green	Green	Green
8 h	Green	Green	Green	Green	Green
12 h	Green	Green	Green	Green	Green
24 h	Green	Red	Red	Red	Red

Tape B showed very good stability at every studied temperature. It did not show any signs of deformation and its form remained unchanged during and after heating. Due to the much thinner base film (100 μm) Tape B did not bend/deform like Tape A did.

Adhesive lines can be identified in the reference image of Tape B's surface (Fig. 6.2 a). Still after 12 hours annealing at 80 °C there was adhesive on the tape but after 24 hours all glue had vanished/evaporated from the surface of the base film.

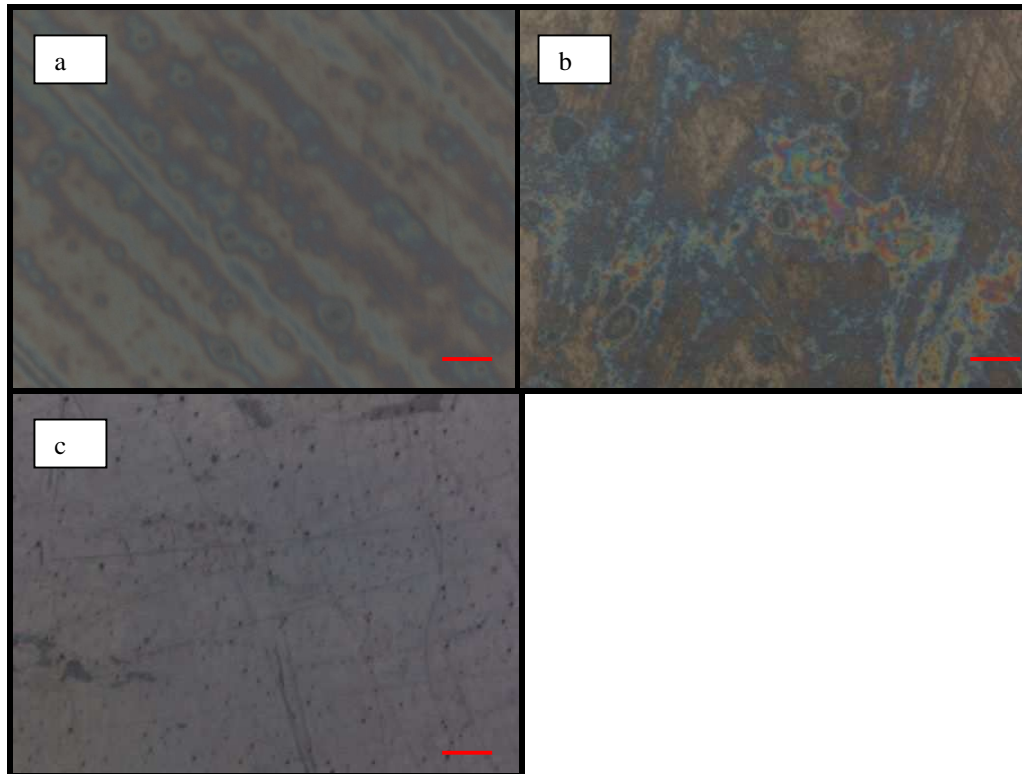


Figure 6.2 Tape B a) reference b) after 12 hours at 80 °C c) after 24 hours at 80 °C

Tape C

Table 6.3 Thermal stability of Tape C

Tape C	RT	80 °C	100 °C	125 °C	150 °C
30 min	Green	Green	Green	Green	Green
60 min	Green	Green	Green	Green	Green
90 min	Green	Green	Green	Green	Green
4 h	Green	Yellow	Yellow	Yellow	Yellow
8 h	Green	Red	Red	Red	Red
12 h	Green	Red	Red	Red	Red
24 h	Green	Red	Red	Red	Red

The adhesive layer of Tape C is formed from the small glue drops. There were no visual changes found in the appearance of the base film or the adhesive layer after short annealing times but the tape became dry after 4 hours annealing and the adhesive drops seen in Figure 6.3a vanished/evaporated after 8 hours annealing at 80 °C. Furthermore, the surface of the base films became porous after 24 hours annealing. Tape C was, nonetheless, included in the further evaluations.

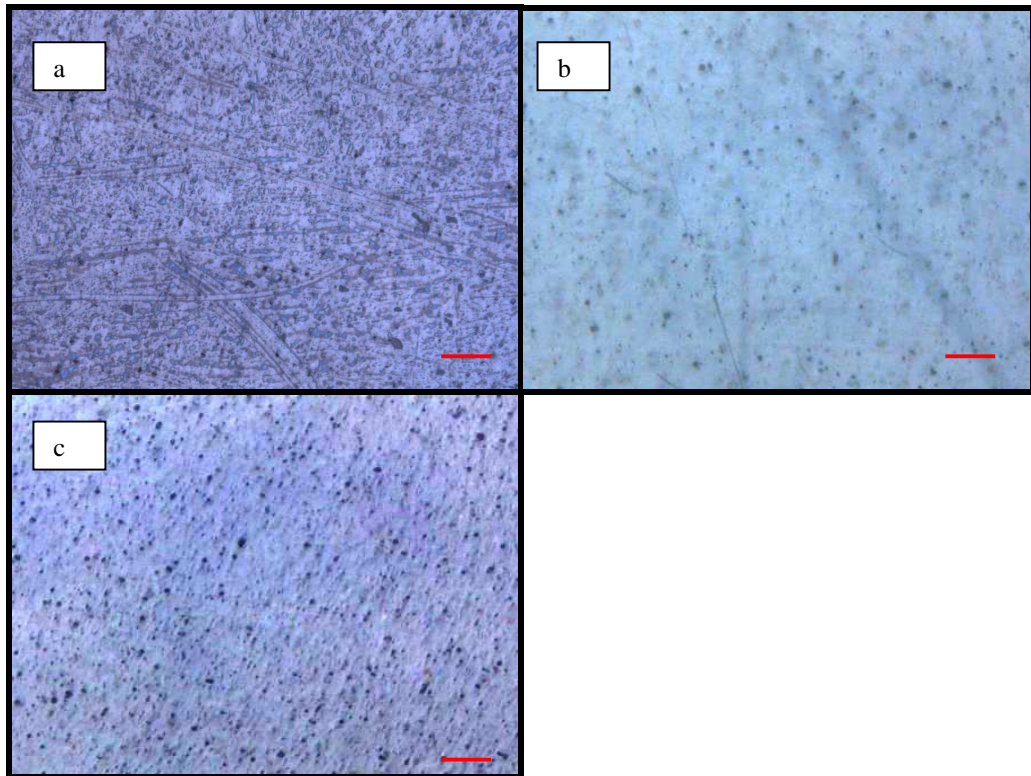


Figure 6.3 Tape C a) reference b) after 8 h at 80 °C c) after 24 h at 80 °C

Tape D

Table 6.4 Thermal stability of Tape D

Tape D	RT	80 °C	100 °C	125 °C	150 °C
30 min	Green	Green	Green	Green	Green
60 min	Green	Green	Green	Green	Green
90 min	Green	Green	Green	Green	Green
4 h	Green	Green	Green	Green	Green
8 h	Green	Green	Green	Green	Green
12 h	Green	Green	Green	Green	Green
24 h	Green	Red	Red	Red	Red

The reference surface of Tape D shows large glue areas on the base film (Fig. 6.4 a). Shorter annealing times didn't cause any remarkable changes on the tape or adhesive. After the 24 hours annealing adhesive areas had dried out and there were several dark “burned” areas on the base film.

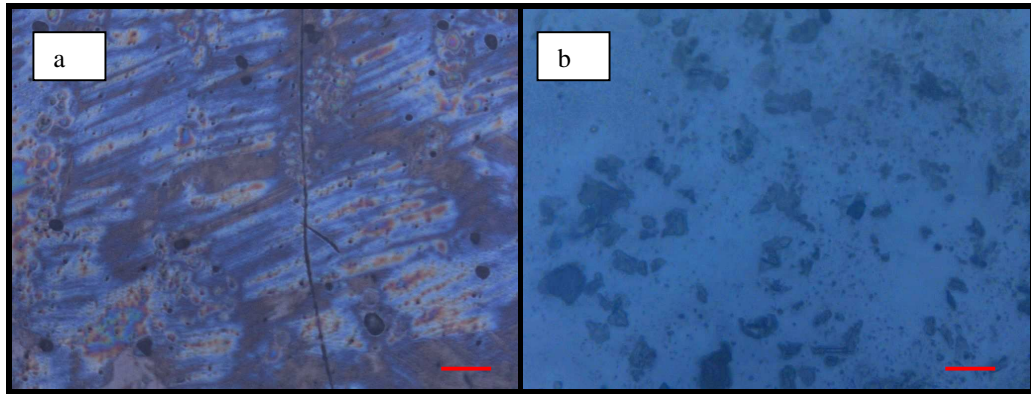


Figure 6.4 Tape D a) reference b) after 24 h at 100 °C

Tape E

Table 6.5 Thermal stability of Tape E

Tape E	RT	80 °C	100 °C	125 °C	150 °C
30 min	Green	Green	Yellow	Yellow	Yellow
60 min	Green	Green	Yellow	Yellow	Yellow
90 min	Green	Green	Yellow	Yellow	Yellow
4 h	Green	Green	Yellow	Yellow	Yellow
8 h	Green	Green	Yellow	Yellow	Yellow
12 h	Green	Green	Yellow	Yellow	Yellow
24 h	Green	Red	Red	Red	Red

The base film of Tape E is based on polyolefin. It is much softer and more elastic when compared to the EVA and PET films in this study. Microscopy investigation revealed that PO has exceptional crystal-like structure which can be seen in Figure 6.5 a. It is difficult to distinguish the adhesive layer from the base film.

The PO base film transformed into more elastic when heated at higher temperatures than 80 °C and was very difficult to handle and prone to tearing when removed from the surfaces. During the short annealing times there were no

signs of changes in the adhesive layer or the base film. After 24 hours annealing at 100 °C tape becomes less glossy which probably results from drying out.

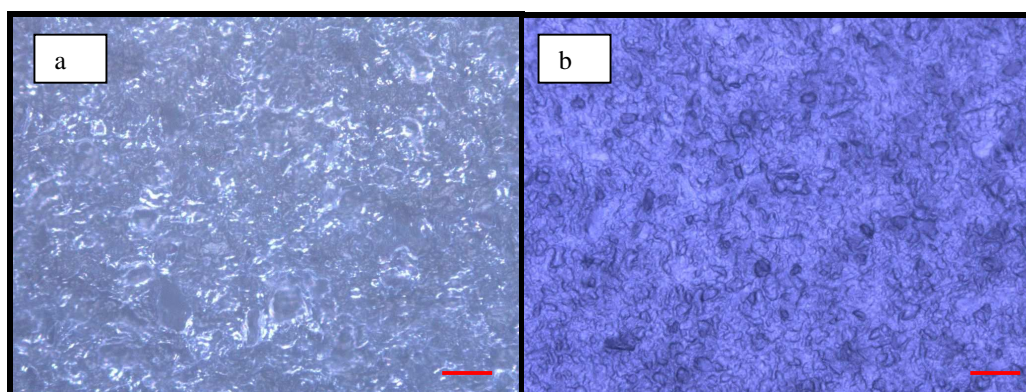


Figure 6.5 Tape E a) reference b) after 24 h at 100 °C

6.2 Evaluation of Adhesion at Different Temperatures

Adhesion strength was evaluated by determining maximum value of adhesion strength from individual measurements. All measurements were repeated five times and an average value is reported. In addition, the detachment work (strain energy) was evaluated from all measurements by employing the Trapezoid rule. The results are reported in the same manner by averaging five individual measurements. Maximum strength values and detachment work for the tapes B, C, D and E are presented on Figures 6.6-6.12. The standard deviation of the five repeats is included in every strength bar.

Figure 6.6 Adhesion strength of Tape B

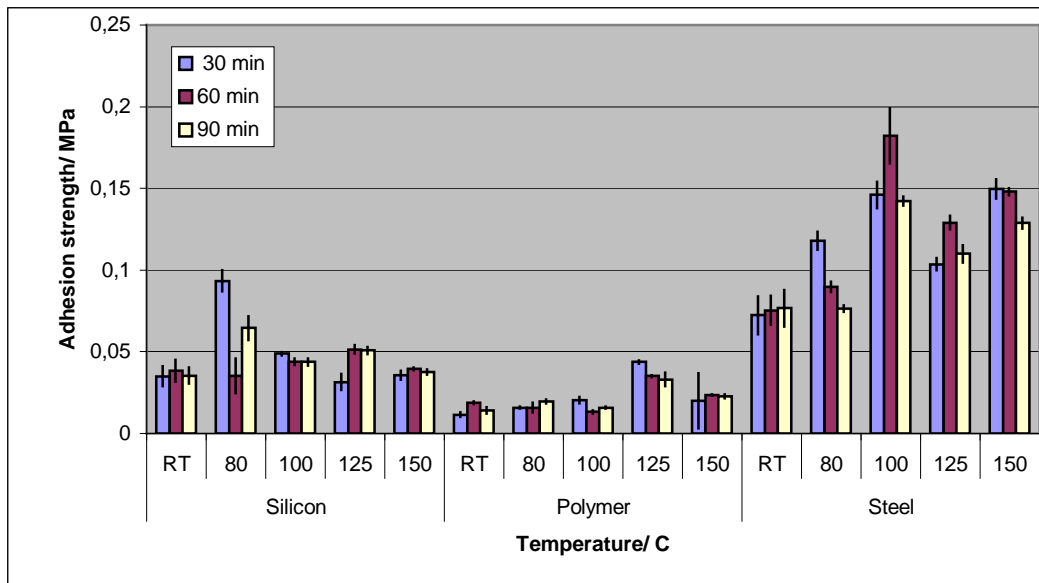


Figure 6.7 Detachment work of Tape B

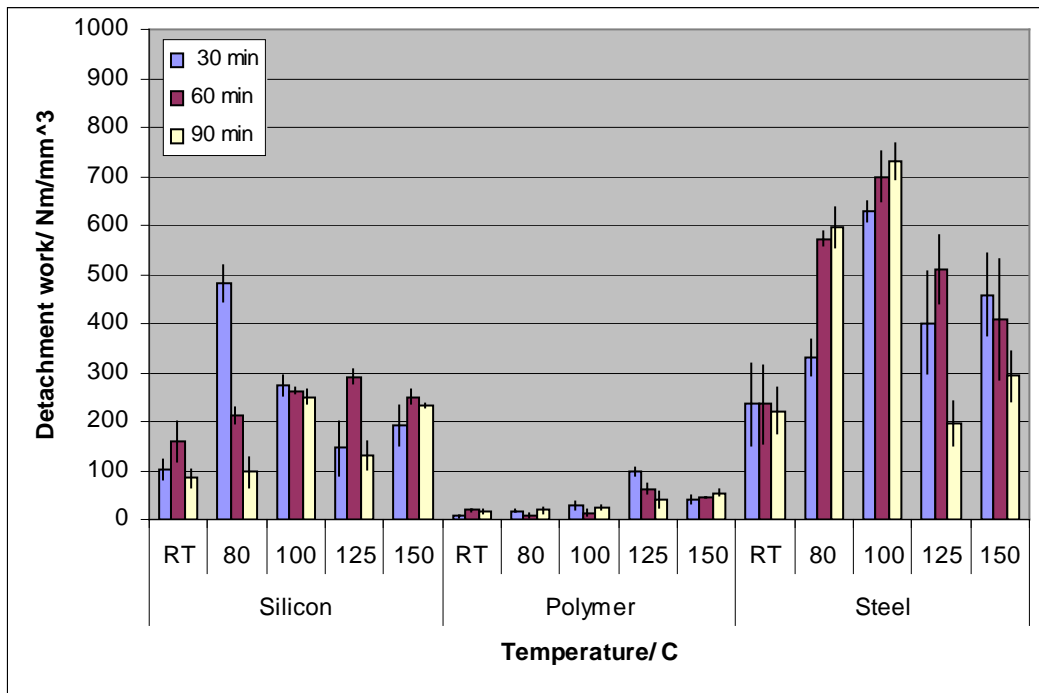


Figure 6.8 Adhesion strength of Tape C

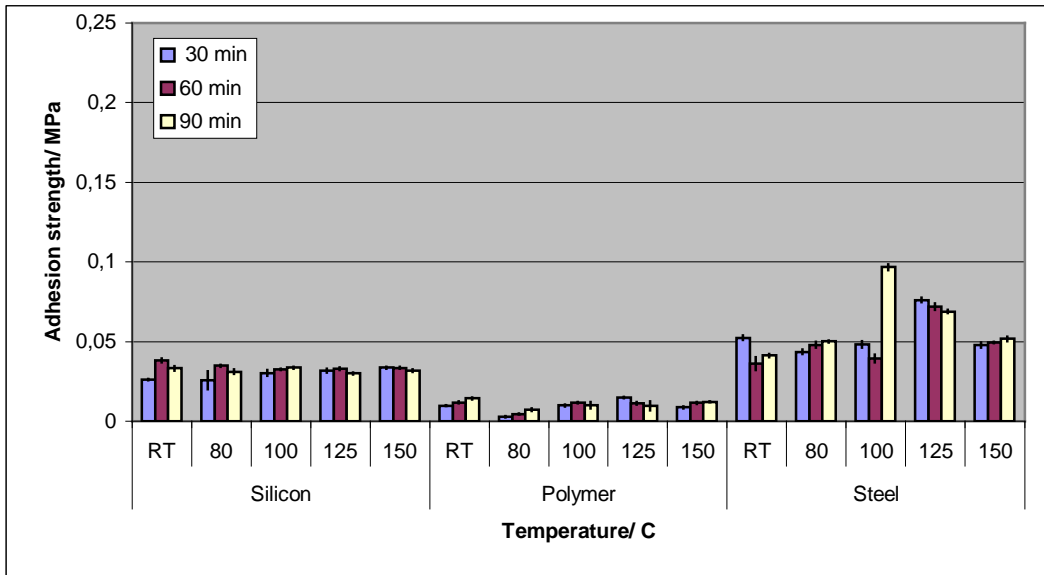


Figure 6.9 Detachment work of Tape C

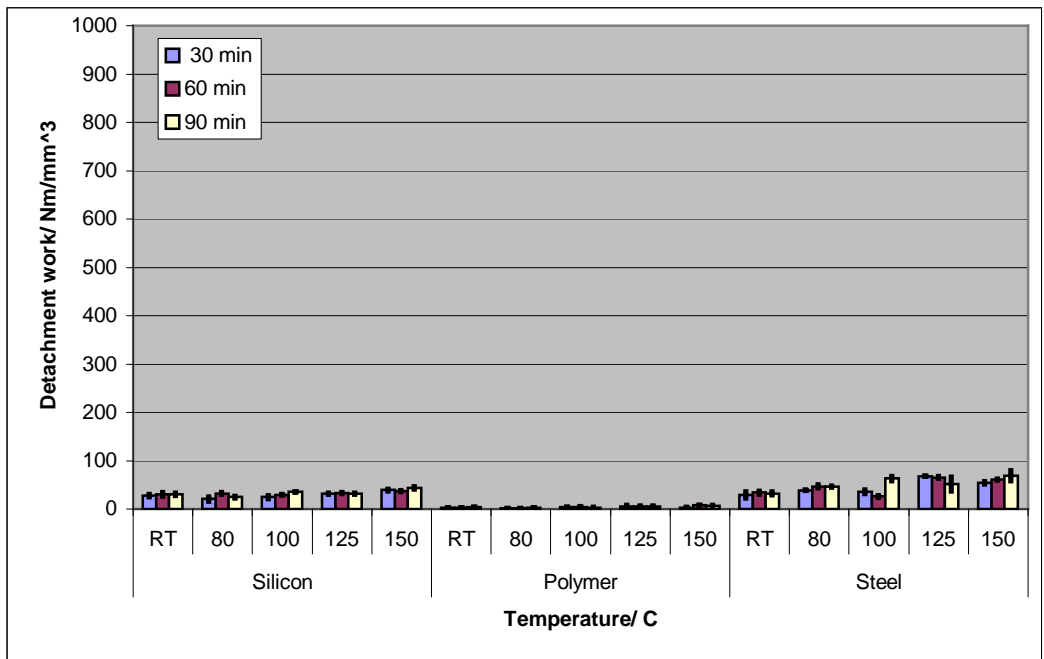


Figure 6.10 Adhesion strength of Tape D

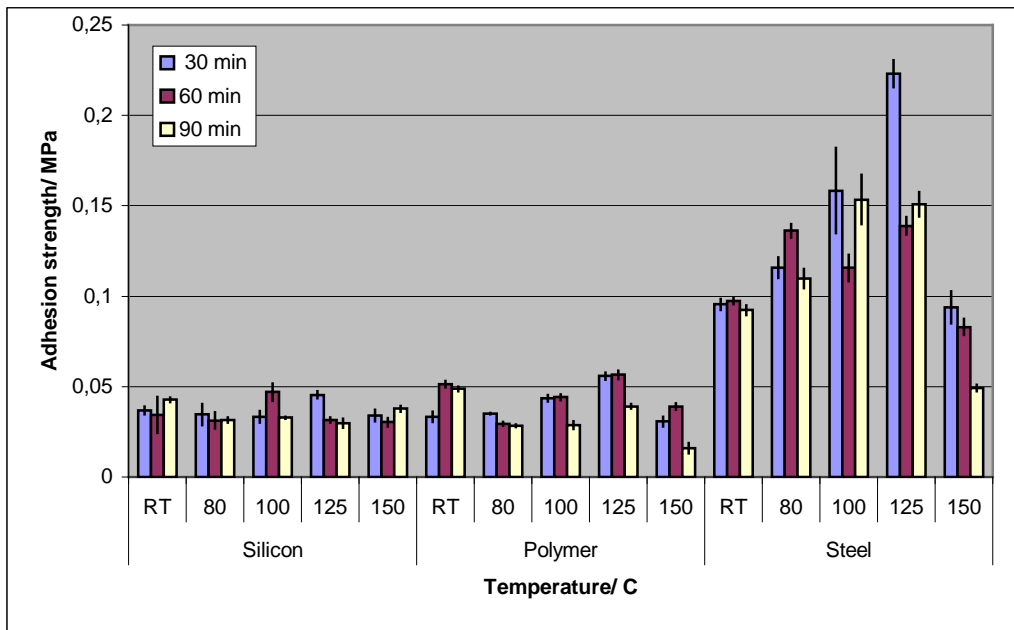


Figure 6.11 Detachment work of Tape D

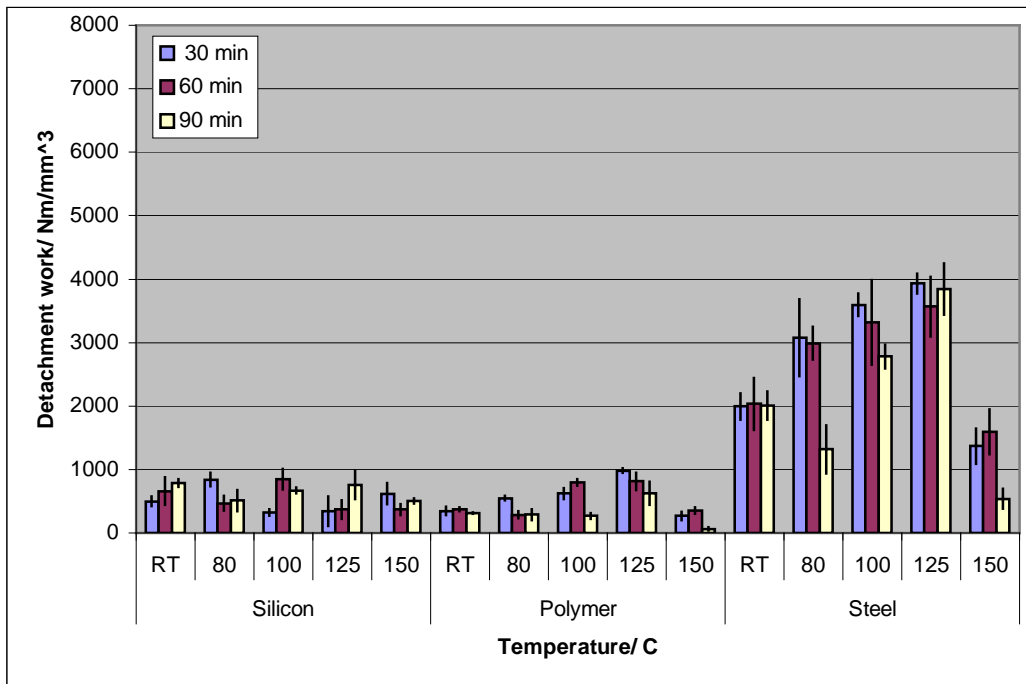
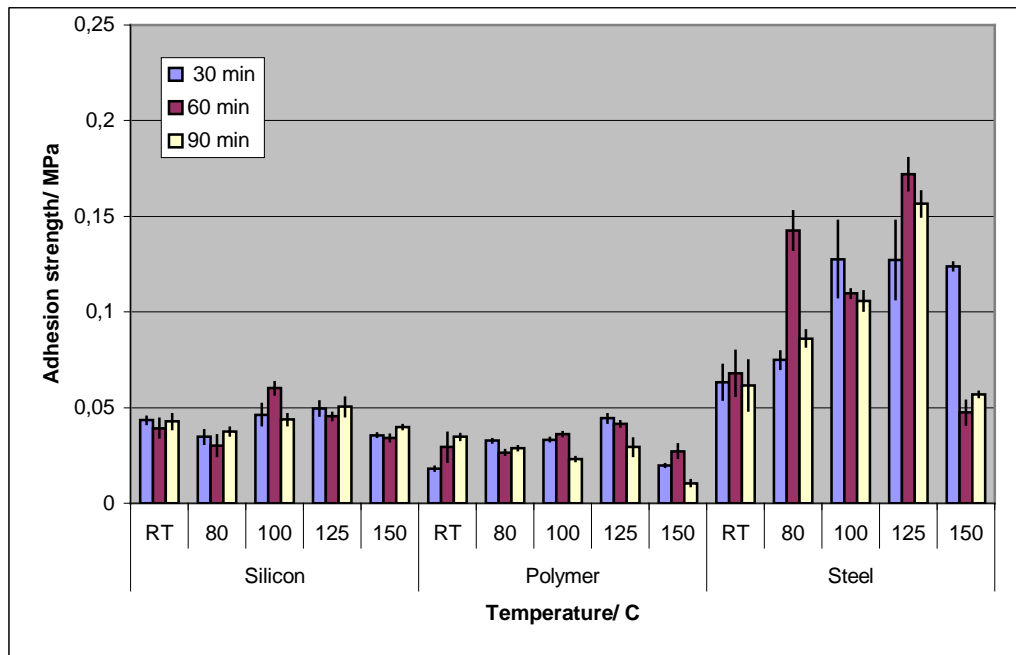
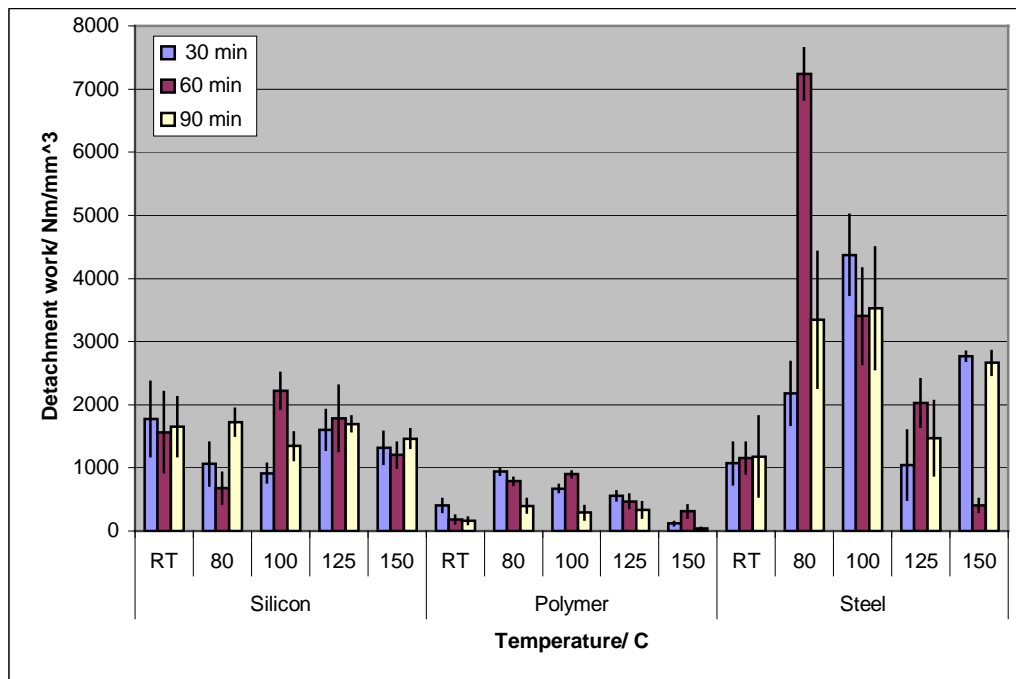


Figure 6.12 Adhesion strength of Tape E



Graph 6.13 Detachment work of Tape E



6.2.1 Statistical Analyses

The measured data was analyzed statistically by employing the full factorial analysis of variance. The results are shown in Table 6.6 for adhesion strength and Table 6.7 for adhesion work with the help of ANOVA tables. The effect of all main factors (i.e. tape, component, temperature and time) is significant at well below the 0.1 % risk level in the case of both adhesion strength and adhesion work. The main factors show also significant interaction as all interactions are also significant at well below 0.1 % risk level.

Table 6.6 Analysis of variance table for the adhesion strength

Depended Variable: Adhesion strength						
Source	Sum of Squares	df	MS	F	p	
Tape	0,15	3	0,05	330,935	0	
Component	0,889	2	0,445	2939,961	0	
Temperature	0,067	4	0,017	111,218	0	
Time	0,006	2	0,003	18,218	0	
Tape*Component	0,088	6	0,015	96,795	0	
Tape*Temperature	0,046	12	0,004	25,499	0	
Tape*Time	0,008	6	0,001	8,492	0	
Component*Temperature	0,081	8	0,01	66,596	0	
Component*Time	0,012	4	0,003	19,235	0	
Temperature*Time	0,008	8	0,001	6,8	0	
Tape*Component*Temperature	0,059	24	0,002	16,169	0	
Tape*Component*Time	0,012	12	0,001	6,794	0	
Tape*Temperature*Time	0,027	24	0,001	7,369	0	
Component*Temperature*Time	0,024	16	0,002	10,069	0	
Tape*Component*Temperature*Time	0,054	48	0,001	7,471	0	
Error	0,106	702	0			
Total	4,27	882				

Table 6.7 Analysis of variance table for the detachment work

Depended Variable: Detachment work					
Source	Sum of Squares Type III	df	MS	F	p
Tape	3,42E+08	3	1,14E+08	554,045	0
Component	2,09E+08	2	1,05E+08	507,281	0
Temperature	2,82E+07	4	7057500	34,259	0
Time	2,34E+06	2	1,17E+06	5,667	0,004
Tape*Component	1,61E+08	6	26800000	130,097	0
Tape*Temperature	4,82E+07	12	4,02E+06	19,494	0
Tape*Time	3,92E+06	6	6,54E+05	3,172	0,004
Component*Temperature	3,57E+07	8	4464250	21,671	0
Component*Time	2,63E+07	4	6,57E+06	31,881	0,013
Temperature*Time	5,89E+06	8	7,37E+05	3,575	0
Tape*Component*Temperature	7,94E+07	24	3,31E+06	16,051	0
Tape*Component*Time	5,77E+06	12	4,81E+05	2,333	0,006
Tape*Temperature*Time	2,26E+07	24	9,40E+05	4,565	0
Component*Temperature*Time	2,31E+07	16	1,44E+06	7,011	0
Tape*Component*Temperature*Time	5,60E+07	48	1,17E+06	5,664	0
Error	1,48E+08	720	2,06E+05		
Total	1,66E+09	900	0		

Table 6.8 shows the results of the Bonferroni pair wise comparison test for pairs of means for the adhesion strength (MPa) and detachment work (strain energy) (Nm/mm³). The risk level for rejection is 5 %. Homogenous groups indicate which means are not significantly different.

Table 6.8 *Bonferroni pairwise comparison test for pairs of means*

I ADHESION TO STUDIED SURFACES

Adhesion strength			Detachment work		
Surface	Mean	Homogenous groups	Surface	Mean	Homogenous groups
Steel	0.0984	A	Steel	1384,04	A
Silicon	0.0393	B	Silicon	576,93	B
Polymer	0.0251	C	Polymer	234,5	C

II ADHESION OF THE TAPES

Adhesion strength			Detachment work		
Tape	Mean	Homogenous groups	Tape	Mean	Homogenous groups
D	0.0662	A	E	1475.90	A
B	0.0611	B	D	1197,5	B
E	0.0576	C	B	226,08	C
C	0.0322	D	C	27,82	D

III EFFECTS OF TEMPERATURE EXPOSURE ON ADHESION

Adhesion strength			Detachment work		
Temp	Mean	Homogenous groups	Temp	Mean	Homogenous groups
125	0.0673	A	100	944.60	A
100	0.0614	B	80	867.35	B
150	0.0510	C	125	802.191	B
80	0.0482	C	25	539.73	C
25	0.0435	D	150	505.24	C

IV EFFECT OF EXPOSURE TIME

Adhesion strength			Detachment work		
Time	Mean	Homogenous groups	Time	Mean	Homogenous groups
60	0.0561	A	60	790.68	A
30	0.0560	A	30	738.36	A
90	0.0507	B	90	666.43	B

6.3 Residue Analysis

Figures 6.14-6.16 show typical residue types on the components. Figures 6.17-6.20 shows residue rating for the packages being held in contact with the tapes as a function of time and temperature.

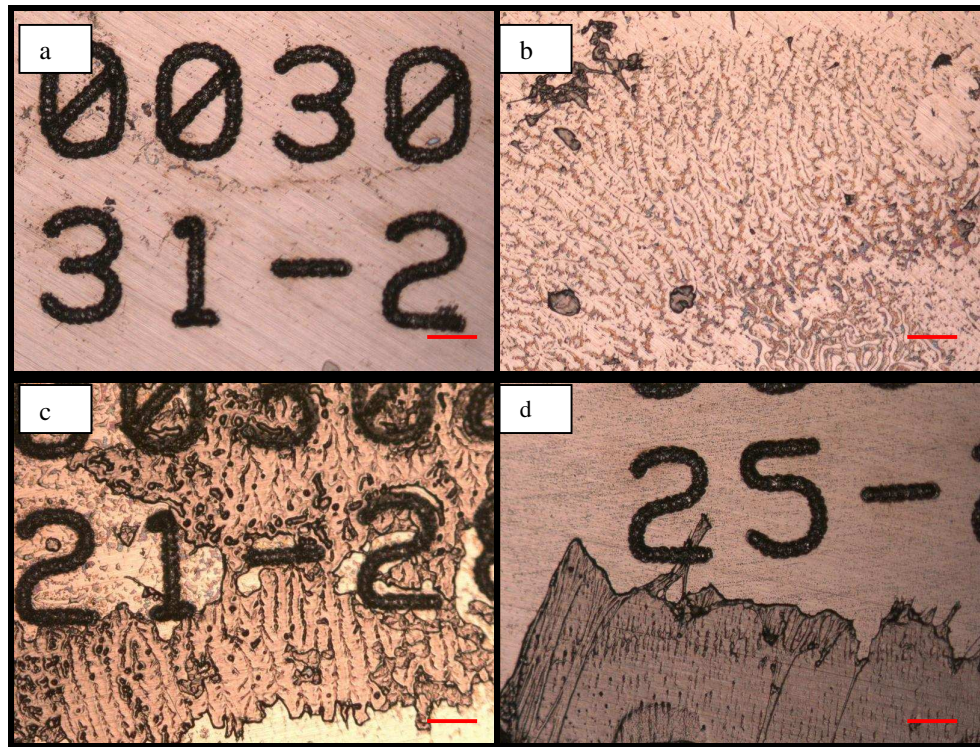


Figure 6.14 Silicon a) Tape C at 80 °C 60 min b) Tape D at 125 °C 30 min c) Tape D at 150 °C 90 min d) Tape E at 125 °C 30 min. Residue level is 2 in A, 4 in B and 5 in C and D

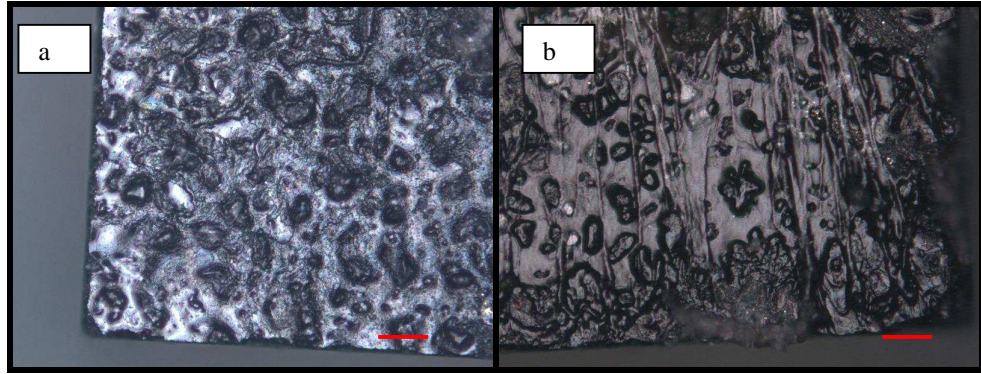


Figure 6.15 Polymer a) Tape B at 100 °C 30 min b) Tape E at 80 °C 60 min. Residue level is 2 in A and 5 in B.

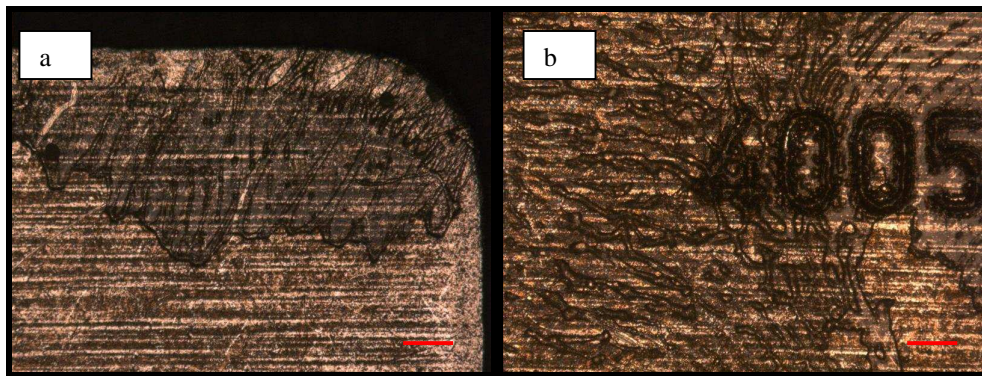


Figure 6.16 Brushed steel a) Tape E at 80 °C 30 min b) Tape E at 150 °C 90 min. Residue level is 3 in A and 5 in B

Figure 6.17 *Residue rates of Tape B*

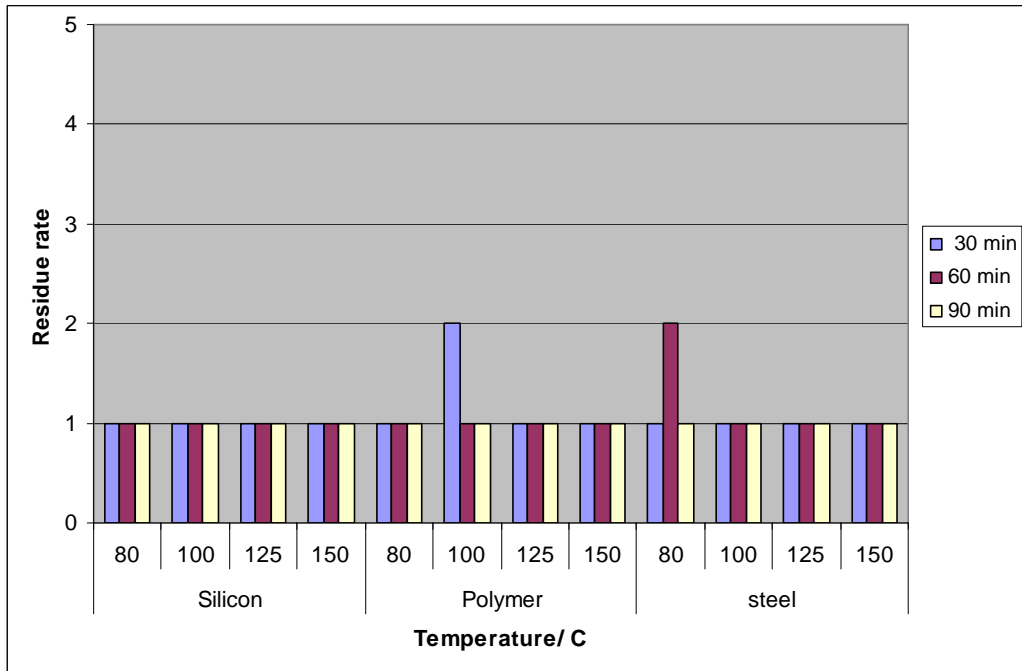


Figure 6.18 *Residue rates of Tape C*

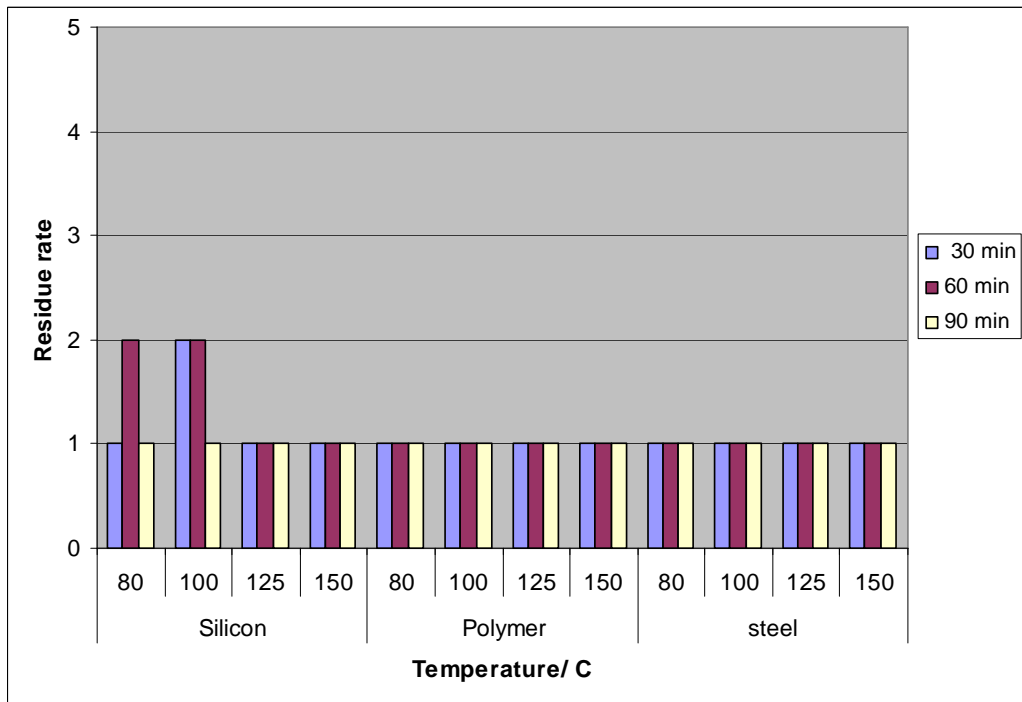


Figure 6.19 Residue rates of Tape D

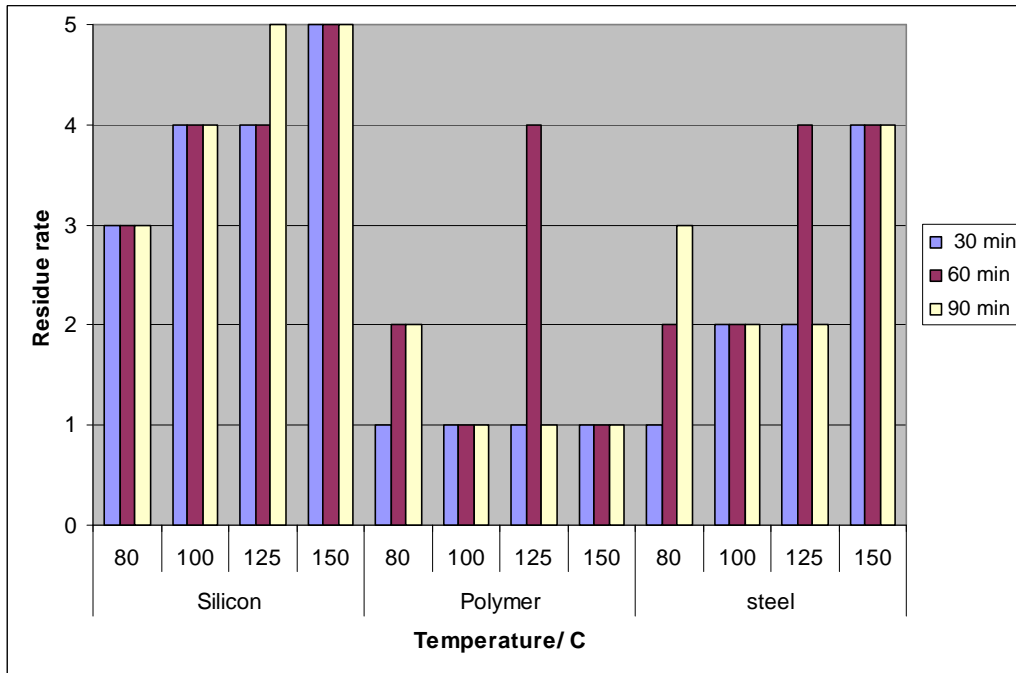
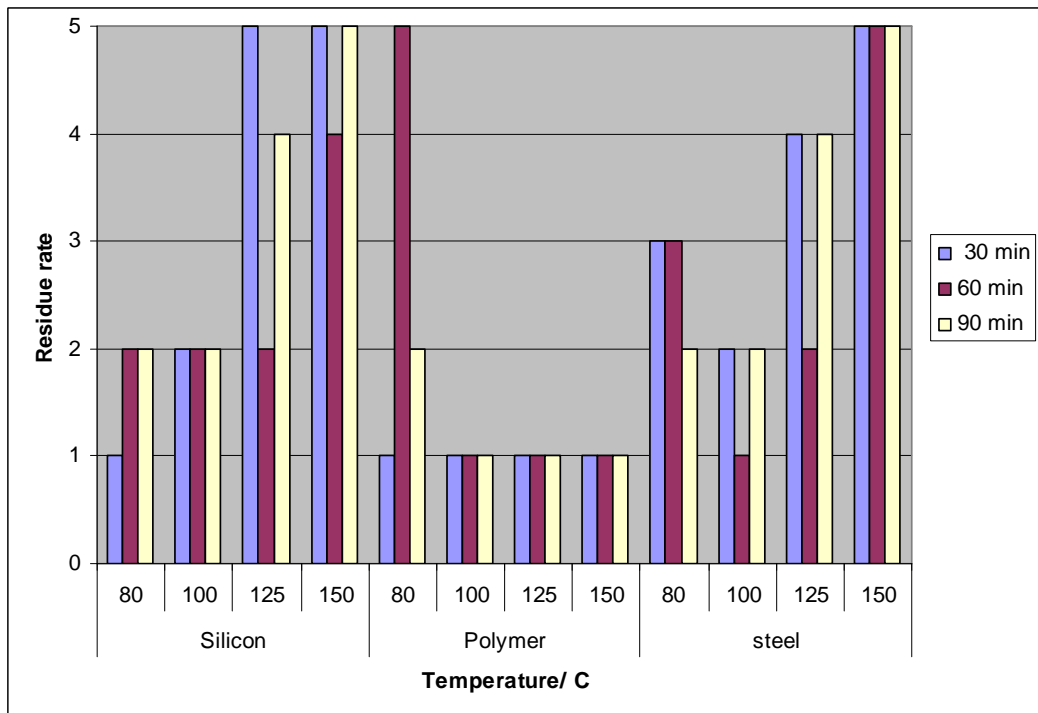


Figure 6.20 Residue rates of Tape E



7 Examination of the Results

The results of the thermal stability test, adhesion evaluation and residue analysis are discussed and analysed under this chapter.

7.1 Thermal Stability of the Tapes

Except for Tapes A and E, all tapes are stable at elevated temperatures up to 150 °C. There were no noticeable deformation occurring in short-term annealing and longer annealing times 4 - 24 hours only dried out the adhesive. However, we can leave drying out of account because the component testing will not take more than 90 minutes.

Tape A was not included in further evaluations because of the weak heat resistance of the EVA base film. The base film of the tape started to show signs of melting at 100 °C by warping excessively after about 30 minutes heating at 100 °C. The melting point of EVA is 98 °C. Longer heating time did not have essential difference on the deformation. At 80 °C the thermal stability was rated moderate due to bending of the tape. The reason for the bending is most probably the thick and stiff base film. Tape A is the thickest of all studied tapes with the total thickness of 166 µm. Evidence of changes in the adhesive of the tape was not found in optical microscopy investigations.

Thermal stability of Tape E was rated moderate at temperatures above 80 °C because its polyolefin base film turned into more elastic and softer when heated. This makes handling and removing Tape E difficult and prone to tearing but is not considered as a reason to eliminate the tape.

7.2 Adhesion Evaluation at Elevated Temperatures

There is a large difference between the adhesion strength of the studied surfaces. This is the result of different surfaces of the components. WLP's adhesion interface is formed between silicon and adhesive layer. Polymer and brushed steel have a rougher surface. The polymer package has a very porous surface as it can

be seen in Figure 5.1 b. Roughness of the substrate is an important factor in adhesion. If an adhesive penetrates into the pores, it forms a strong mechanical interlock. However, pores can have a negative effect on adhesion if they are too large, because then the contact area is decreased as it was discovered in case of the polymer surface. On the contrary it is possible that the strong adhesion of the steel surface was much based on the mechanical interlocking.

Following conclusions can be made based on the Bonferroni all-pairwise comparisons of the means after which the adhesion of different tapes is analysed in-depth:

1) *The package surface affects the adhesion of the tapes significantly:* The average adhesion strength and work depends on the type of adhesion surface (steel, polished silicon or molding epoxy polymer or electronic packages) and the adhesion of steel is the highest (0.984 MPa and 1384.04 Nm/mm³) while that of the polymer is the lowest (0.0251 MPa and 576.93 Nm/mm³). The conclusion is the same regardless of the evaluation criterion. The mean adhesion strength of the steel surface is over twice as high as that of the silicon and almost four-fold as that of the polymer surface.

2) *The adhesion of all four tapes studied is significantly different from one another:* Tape D has the highest adhesion strength over all treatments and studied surfaces. The adhesion strength of Tape B is the second-highest, that of Tape E is the third-highest, and that of Tape C is the lowest. However, it is interesting to observe that the work required to remove Tape E is the highest and the work needed to detach the tape with the highest adhesion strength (Tape D) is only the second-highest. This observation can be attributed to Tape E's higher tendency to form adhesive fibrils when the component is lifted as compared to the other studied tapes. Removing components from the tape C requires the least amount of work.

3) *The adhesion of all tapes increases with increase in testing temperature until the temperature of 100-125 °C is reached.* With further increase in temperature the adhesion of all tapes decreases; which was expected because 150 °C is close to

upper temperature limit of cross-linked acrylic adhesives [32]. This result indicates that the elevated temperature range of 100 – 125 °C is quite suitable for characterization of the MEMS inertial sensors from the point of view of adhesion performance of the tapes. In the range of increased temperature, all changes in the adhesion strength as well as work are statistically significant.

4) Investigation of the exposure time for elevated temperatures showed that the adhesion strength starts to decrease significantly after one hour at the elevated temperatures. There is no statistically significant change in the adhesion within 60 minutes from the beginning of the elevated temperature exposure. Thus, one hour can be considered a safe timeframe in which to carry out the functional characterization of MEMS devices, from the point of view of adhesion performance of the tapes.

Tape B

The reference adhesion strength of Tape B at room temperature for silicon is around 0.03 MPa. At 80 °C adhesion increases at first to 0.093 MPa but is decreased in 30 minutes. Adhesion at temperatures above 80 °C stays more stable.

Adhesion of Tape B on polymer is much weaker compared to that on silicon or steel. Adhesion strength stays near the reference, 0.011-0.018 MPa, at lower temperatures but there is an increase of adhesion at 125 °C at which polymer reaches its maximum adhesion strength. Extremely small work of adhesion indicates that the polymer package detaches smoothly from the tape and there is no glue fibrillation.

Brushed stainless steel surface has the highest adhesion strength of the three studied materials. The reference adhesion at room temperature is 0.075 MPa. Contrary to other surfaces adhesion stays clearly above the reference at every temperature, even at 150 °C. Adhesion on steel reaches the maximum adhesion strength at 100 °C. The adhesion work is also higher but at 90 minutes it is halved at higher temperatures although the adhesion strength stays unchangeable.

Tape C

Adhesion of Tape C stays very stable near the reference value at every temperature for silicon and polymer surfaces. The standard deviation of the adhesion strength for silicon is 0.002 MPa and for polymer 0.003 MPa. The detachment work of Tape C is unsubstancial with every surface material, below 70 Nm / mm³, although the adhesion strength is only a bit weaker when compared to Tape B. However, there is a risk that the adhesion strength is too weak for the polymer package. The adhesion strength of 0.01 MPa can be considered to be the limit for adequate adhesion. The actual 9 mm² polymer MEMS package does not stay on the tape C though the force to attach the component is increased. The main reason for the weak adhesion is the porous topography which causes that the contact area is much smaller than the size of the package bottom.

Steel surface attaches only slightly stronger to the tape than silicon. Adhesion stays around the room temperature value until it rises to 0.097 MPa at 100 °C in 90 minutes. Adhesion is higher also at 125 °C but lowers back to the reference at 150 °C. The detachment work does not change during that.

Tape D

Tape D is a UV-tape. Its adhesion is almost equal for silicon and polymer surfaces. With other tested tapes the adhesion with the polymer surface is always the weakest.

Steel has again the best adhesion; the reference strength is around 0.1MPa. 30 minutes heating at 125 °C increases the adhesion strength up to 0.22 MPa, which is the highest measured strength in the study. The detachment work is exceptionally high for steel, and slightly increased also for polymer. Adhesion decreases at 150 °C with every surface, but the clearest drop can be seen with polymer and steel.

Tape E

Tape E is also a UV-tape. It has a very soft and elastic polyolefin base film and its tack is strong. During de-taping the tape sticks on the substrate, tears easily and leaves a lot of glue residues. Adhesion strength is quite similar to Tape D. There can be seen the same trend like with other tapes that the adhesion strength raises up to 100-125 °C and drops again at higher temperature. For silicon and polymer the adhesion does not change much from the reference adhesion, but the adhesion strength between steel and the tape is almost doubled at 100 °C. The detachment work is high for every surface and the deviation is quite large, implying that there are a lot of glue fibrillation occurring until the work decreases at 150 °C.

7.3 Residue Analysis

Glue stains were concentrated in many cases around the component markings or covered them completely, which can cause problems when components are recognised by machine vision. Using a low-residue tape reduces additional rinse processes during the wafer or component testing. According to Phaoharuhan [46] this contributes to substantial testing cycle time reduction, eliminates wafer breakage and improves test yield.

Both tapes B and C left only few or no residues on the MEMS packages. The highest residue rate was 2 for these tapes. Component's surface impurities and the method how the component is removed from the tape affect the residue level more or less. Lifting the tape under the component with a pair of tweezers, while removing the component, detaches the component from the tape more gently.

UV-tapes D and E turned out to stain the components extensively at every temperature. Both covered the components with glue and large chunks of tape base film. Residue rate was in the most cases above 3 but rates 4 and 5 were also common. Tape D is more clearly depended on the heating time and temperature whereas Tape E residue levels remained same through the 90 minutes heating.

The amount of residues would have probably decreased dramatically if the tapes were exposed to UV-light.

Not only the tape and adhesive properties are causing residues. Package material topography is also important factor: Silicon surface is prone to high residue level due to its larger contact area. The WLP package has very smooth surface while polymer and brushed steel has much rougher surface and thus less contact area. Especially the polymer package has substantially smaller contact area when compared to its total size due to the package's porous structure, which is also a reason why there is not formed as strong adhesion between polymer and the tape as with other surface materials like the adhesion strength study indicated.

Beside adhesion strength, detachment work is another way to predict how much tape leaves residues on the component. High detachment work tells that adhesive starts to detach slowly from the base film and is drawn into long glue fibrils that form residues on the removed component. As Williams and Kauzlarich [42] suggested, high temperature and slow pull-off/ peel rate cause more cohesive failures.

8 Conclusions

Applicability of five commercial heat resistant wafer back-grinding and dicing tapes was evaluated for their temperature tolerance over the time frame required for functional characterization of MEMS gyroscopes. Short and long-time thermal stability, adhesion strength and work and the amount of post-detachment residue on the surfaces were used as evaluation criteria. The aim of the thermal stability testing was to find out possible physical deformations in the adhesive layer or the base film. The evaluation temperatures were 25, 80, 100, 125 and 150 °C, and the annealing times were 30, 60 and 90 minutes.

Ethylene vinyl acetate (EVA) is not appropriate base film material due to its low melting point (98 °C) Based on its poor thermal stability Tape A was eliminated from further analyses. The EVA base film started to warp after even a short temperature exposure to 100 °C. The Polyolefin (PO) base film of Tape E turned more elastic during heating which was found difficult in handling the tape. Other tapes did not show any detectable signs of degradation during the annealing treatments. The only detectable change in appearance during the long-time annealing was drying of the adhesive. However, because the time needed for gyroscope characterization seldom exceeds 90 minutes and detectable drying took place much later, during of the adhesive was now considered an issue in this application.

The adhesion strength of the tapes was determined with the help of the tensile test. Both adhesion strength and strain energy were evaluated. The strain energy was considered as detachment work. Adhesion on three different types of surfaces was studied: silicon, polymer and brushed steel. The surface types were chosen to represent typical surface materials of MEMS gyroscope packages. Adhesion evaluation results were analysed by employing the analysis of variance and Bonferroni all-pairwise comparison test for the pairs of means of adhesion strength and work.

Analysis of variance showed statistically significant (5 % risk level) differences between the main factors as well as the interaction factors. Following conclusions were made based on the Bonferroni all-pairwise comparison tests:

- 1) The package surface affects the adhesion of the tapes significantly. The adhesion strength as well as the adhesion work of tapes on steel was the highest while those of the tapes on polymer were the lowest.
- 2) The adhesion of all four tapes studied is significantly different from one another: In terms of adhesion strength, the order of superiority of the tapes was D, B, E, and C. However, the order of superiority in terms of detachment work was E, D, B, and C. The higher adhesion work of Tapes E and D can be attributed to the much higher tendency to adhesive fibril formation.
- 3) The comparison of temperature exposure means indicates that the adhesion increases up to about 100-125 °C but further increase in temperature decreases the adhesion.
- 4) Adhesion value at each temperature does not change significantly within one hour but decreases abruptly with longer exposure times. Thus, one hour can be considered a safe component testing time from the adhesion point of view.

Tape B has an adequate adhesion on every studied surface. The adhesion between the silicon surface and the tape seems unstable at low temperature but is much more stable at higher temperatures. Steel reaches its maximum adhesion already at 100 °C while polymer requires 125 °C. The detachment work of steel surface is halved at the highest temperatures while the adhesion strength remains almost the same.

Tape C has a very stable adhesion at every temperature for silicon and polymer surfaces, though the adhesion on polymer is relatively weak. The adhesion of steel increased significantly at 100 °C but is decreased back to reference at 150 °C.

Tape D was the only tape that has almost equal adhesion on silicon and polymer. Generally its adhesion strength and detachment work are quite high compared to Tapes B and C.

Tape E has also a strong tack. Its adhesion is increased to some point but at higher temperature it drops again. The detachment work is high for every surfaces implying that there are severe glue fibrillation occurring.

High detachment work can be regarded as a sign of high residue level because glue fibrillation is a significant reason for residues forming. The residue analysis revealed that the both UV-dicing tapes D and E stain components badly at every temperature. However, if the tapes would have been UV-exposed before the removing the packages, the residue level would have probably been much lower. Tapes B and C are low-residue tapes and they left hardly any residues on the components and their detachment work was also much lower.

Based on the carried out evaluation the tape B appears to be the best choice for the Test-on-Tape testing of MEMS gyroscopes. There is no physical deformation occurring in the adhesive layer or the PET base film, all package materials adhere well to it and it leaves very few or no residues on the components so there is no need for the additional rinse processes after the testing.

Tape C is a good option for testing silicon and ceramic/steel packages, or larger polymer packaged components. For small polymer packages ($< 10 \text{ mm}^2$) with highly porous surface its adhesion strength is not sufficient. Tape C's advantage over tape B is the very low detachment work; there are hardly no glue fibrillation.

9 References

Figures

Fig. 2.1 www.etc.usf.edu/clipart/71000/71004/71004_fou_gyroscop.html.

Fig. 2.2 www.electroiq.com/index/display/nanotecharticledisplay/4659348781/articles/small-times/nanotechmems/mems/sensors/2010/11/introduction-to-mems-gyroscopes.html.

Fig. 2.3 www.electroiq.com/index/display/nanotecharticle/display/4659348781/articles/small-times/nanotechmems/mems/sensors/2010/11/introduction-to-mems-gyroscopes.html.

Fig. 2.4 www.vti.fi/cmr3000/.

Figure 2.5 J. Mah, E. Wilson: “Chapter 14: *Fundamentals of Microelectromechanical Systems*”, University of Colorado at Boulder

Fig. 2.6 K. Gilleo: “*MEMS Packaging Updates*”, Advance Packaging 2005

Fig. 2.7 Afore: available at www.afore.fi.

Fig. 3.1 G.T. Barnes, I.R. Gentle: *Interfacial Science: An Introduction*, Oxford University Press, 2005, 264 p.

Fig. 3.2 D. Kopeliovich: “*Fundamentals of adhesive bonding*” Substech, 2009, www.substech.com/dokuwiki/doku.php?id=fundamentals_of_adhesive_bonding.

Fig. 3.3 P.M. McGuiggan, A. Chiche, J.J. Filliben, D.J. Yarusso: “*Peel of an Adhesive Tape from a Temperature-gradient Surface*”, *International Journal of Adhesion & Adhesives* **28**, (2008), pp. 185–191.

Fig. 3.4 www.eastman.com/Markets/Tackifier_Center/Pages/Block_Copolymer.aspx.

Fig. 3.5 Müller, H. W. J.: “*Vinyl ether polymers*” in *Handbook of Pressure Sensitive Adhesive Technology*, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp. 494-507.

Fig 3.6 FINAT, “*FINAT Technical Handbook*” , 6th edition, 2001.

Fig 3.7 M. Turunen, P. Marjamäki, M. Paaanen, J. Lahtinen, J. K. Kivilahti: “*Pull-off Test in the Assessment of Adhesion at Printed Wiring Board Metallisation/epoxy Interface*”, *Microelectronics Reliability* **44**, (2004), pp. 993–1007

Fig 3.8 Y. Y. Lin, C. Y. Hui , Y. C. Wang: “*Modeling the Failure of an Adhesive Layer in a Peel Test*”, Journal of Polymer Science Part B: Polymer Physics, **40**, Issue 19, (2002), pp. 2277–2291.

[1] L. Foucault (1852) "*Sur les phénomènes d'orientation des corps tournants entraînés par un axe fixe à la surface de la terre — Nouveaux signes sensibles du mouvement diurne*" (*On the phenomena of the orientation of rotating bodies carried along by an axis fixed to the surface of the earth — New perceptible signs of the daily movement*), Comptes rendus hebdomadaires des séances de l'Académie des Sciences (Paris), **35**, pp. 424–427. Available on-line (in French): www.gallica.bnf.fr/ark:/12148/bpt6k2992n/f428.image.

[2] J. G. F. Bohnenberger: “*Beschreibung einer Maschine zur Erläuterung der Gesetze der Umdrehung der Erde um ihre Axe, und der Veränderung der Lage der letzteren*” Tübinger Blätter für Naturwissenschaften und Arzneikunde, **3**, 1817, pp. 72–83.

[3] W. R. Johnson: “*Description of an apparatus called the rotascope for exhibiting several phenomena and illustrating certain laws of rotary motion*”, The American Journal of Science and Art, 1st series, **21**, 2, 1832, pp. 265–280.

[4] Gyroscope.org, www.gyroscopes.org/history.asp.

[5] Gyroscope.org, www.gyroscopes.org/uses.asp.

[6] M. Saukoski: “*System and circuit design for a capacitive MEMS gyroscope*”, Doctoral Dissertation, Helsinki University of Technology, 2008.

[7] N. Yazdi, F. Ayazi, and K. Najafi: “*Micromachined inertial sensors*” The Proceedings of the *IEEE*, **86**, 8, (1998) pp. 1640–1659.

[8] Electroiq.com, www.electroiq.com/index/display/nanotech-article-display/4659348781/articles/smalltimes/nanotechmems/mems/sensors/2010/11/introduction-to-mems-gyroscopes.html.

[9] VTI news: “*CMR3000 challenges the current consumer gyro market offering*”, 3/2010, www.vti.fi/en/newsevents/enews/3/2010_enews/cmr3000_challenges_the_current_consumer_gyro_market_offering/.

[10] L. Liwei: “*Packaging Schemes for MEMS*”, University of California at Berkeley, 2004.

-
- [11] J. Toivanen: “ *Mikromekaanisten kulmanopeusanturin kalibrointialgoritmien kehittäminen*”, diplomityö, Teknillinen korkeakoulu, Sähkö- ja tietoliikennetekniikan osasto, 2008.
- [12] K. Gilleo: “*MEMS Packaging Updates*”, Advance Packaging, (2005).
- [13] J. Mah, E. Wilson:” *Chapter 14: Fundamentals of Microelectromechanical Systems*”, University of Colorado at Boulder.
- [14] P. Martin, *Electronic Failure Analysis Handbook*, 1st ed., 1999, McGraw-Hill Professional, 520 p.
- [15] AEC-Q100:“*AEC-Q100-REV-G Stress Test Qualification for Integrated Circuits*”, Automotive Electronics Council, 2007.
- [16] Afore: “*Product-wafer level testing*”, www.afore.fi/products-wafer_level_testing.
- [17]G.T. Barnes, I.R. Gentle: *Interfacial Science: An Introduction*, Oxford University Press, 2005, 264 p.
- [18] G. L Nelson and J. V. Koleske: ”Adhesion”, in *Paint and coating testing manual*, ASTM, Chap. 44, 1995, pp. 513-523.
- [19] B. V. Deryagin and N. A. Krotova: “*Adhesion*”, Izd. Akad. Nauk SSSR, Moscow, 1949.
- [20] K.L Mittal,: ”*Adhesion Science and Technology Seminar, Part 5: Theories or Mechanism of Adhesion*”, Neste, June 16-17, 1997, pp. 1-36.
- [21] D. Kopeliovich: “ *Fundamentals of adhesive bonding*” Substech, www.substech.com/dokuwiki/doku.php?id=fundamentals_of_adhesive_bonding, 2009
- [22] J. Johnston: *Pressure Sensitive Adhesive Tapes*, 2nd ed., Northbrook, IL: Pressure Sensitive Tape Council, 2003.
- [23] P.M. McGuiggan, A. Chiche, J.J. Filliben, D.J. Yarusso: “*Peel of an Adhesive Tape from a Temperature-gradient Surface*”, *International Journal of Adhesion & Adhesives*, **28**, (2008), pp. 185–191.
- [24] G. L. Buttler: ” *Natural Rubber Adhesives*” in *Handbook of Pressure Sensitive Adhesive Technology*”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 260-293

-
- [25] E. E. Ewins et al.: ” *Thermoplastic Rubbers: A-B-A Block Copolymers*” in Handbook of Pressure Sensitive Adhesive Technology, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 317-373.
- [26] J. J. Higgins, F. C. Jagisch, N.E. Stucker: ” *Butyl Rubber and Polyisobutylene*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 374-395.
- [27]H. W. J. Müller: ” *Vinyl ether polymers*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 494-507.
- [28] L. A. Sobieski, T. J. Tangney: ”*Silicone Pressure Sensitive Adhesives*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 508-517.
- [29] D. Satas: ” *Pressure Sensitive Adhesives and Adhesive Products in the United States*” in Handbook of Pressure Sensitive Adhesive Technology, 2nd Edition, Van Nostrand Reinhold, 1989, pp 3-5.
- [30] J. A. Schlademan: ”*Tackifier Resins*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 527-544.
- [31] A. Zettl,: ” *Modification of Acrylic Dispersions*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 457-493.
- [32] V. Rajekas,: ”*Bond Strength and Its Prognosis*” in Handbook of Pressure Sensitive Adhesive Technology”, Satas, D. 2nd Edition, Van Nostrand Reinhold, 1989, pp 115-157.
- [33] D. Satas: ”*Handbook of Pressure Sensitive Adhesive Technology*”, Van Nostrand Reinhold, pg 656-657, 1989
- [34] D. R. Moore: ” *An Introduction to the Special Issue on Peel Testing*”, International Journal of Adhesion and Adhesives, **28**, 4-5, (2008).
- [35] FINAT, www.finat.com.
- [36] D. J. Yarusso: ”*The Mechanics of Adhesion*”, Elsevier, New York (2002), p. 499–533.
- [37] FINAT: ” *FINAT Technical Handbook*”, 6th edition, 2001.

-
- [38]ASTM D4541 - 09e1: “*Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers*”, www.astm.org/Standards/D4541.html, ASTM.
- [39] M. Turunen, P. Marjamäki, M. Paajanen, J. Lahtinen, J. K. Kivilahti: “*Pull-off Test in the Assessment of Adhesion at Printed Wiring Board Metallisation/epoxy Interface*”, *Microelectronics Reliability*, **44**, (2004), pp. 993–1007.
- [40]Y. Y. Lin, C. Y. Hui , Y. C. Wang: “*Modeling the Failure of an Adhesive Layer in a Peel Test*”, *Journal of Polymer Science Part B: Polymer Physics*, **40**, 19, (2002), pp. 2277–2291.
- [41] T. R Hull, J. S. Collington, A. E. Hill: “*Measurement of Thin Film Adhesion*” *Vacuum* **37**, 3-4, (1987), pp. 327-330.
- [42] J. A. Williams and J. J. Kauzlarich: “*Application of the Bulk Properties of an Acrylic Pressure-sensitive Adhesive to Peeling*”, *J. Adhesion Sci. Technol.*, **21**, 7, (2007), pp. 515–529.
- [43] J. S. Milton, J. C. Arnold: *Introduction to Probability and Statistics: Principles and Application for Engineering and the Computing Sciences*, McGraw-Hill Series in Probability and Statistics, 3rd ed., 1995, pp. 535-655.
- [44] J. S. Milton, J. C. Arnold, 1995, pp. 549-552.
- [45] 3M: “*3M High Performance Double Coated Tapes with Adhesive 375*”, Technical data sheet, 2007.
- [46] P. Phaoharuhan: “*Post Clean Elimination at Backgrind*”, *Electronics Packaging Technology Conference*, (2003), pp. 663 – 666.