CZECH TECHNICAL UNIVERSITY IN PRAGUE

Faculty of Electrical Engineering

Department of Electrotechnology

Influence of mechanical stress on properties of conductive adhesive joints

Master's Thesis

Study program: Electrical Engineering, Power Engineering and Management

Specialization: Technological Systems

Supervisor: doc. Ing. Pavel Mach, CSc.

Usman Tariq

Prague 2017

Czech Technical University in Prague
Faculty of Electrical Engineering

Department of Electrotechnology

DIPLOMA THESIS ASSIGNMENT

First name and family name of student: **Usman Tariq**

Study programme: Electrical Engineering, Power Engineering and Management
Specialisation: Technological Systems

Title of Diploma Project: **Influence of mechanical stress on properties of conductive adhesive joints**

Guidelines:

1. Study principle and basic theories of conductivity of electrically conductive adhesives and their using in electronics.
2. Study materials about measurement of low impedances and nonlinearity.
3. Prepare samples of adhesive joints using adhesive assembly of 0R0 resistors according to the instructions of a supervisor.
4. Measure resistance and nonlinearity of adhesive joints.
5. Apply mechanical stress on the joints according to the instructions of a supervisor.
6. Measure resistance and nonlinearity of adhesive joints after the mechanical load.
7. Try to explain changes caused in the joints by the mechanical stress.

Bibliography/Sources:

[1] Licari, J. J., Swanson, D. W.: Adhesives Technology for Electronic Applications - Materials, Processing, Reliability, Elsevier Inc., 2011,
[2] Y. G. Li, D. Lu, C. P. Wong: Electrical Conductive Adhesives with Nanotechnologies, Springer, Germany, 2010, pp. 176-178 (Chapter 4)

Diploma Project Supervisor: **doc. Ing. Pavel Mach, CSc.**

Valid until the end of the summer semester of academic year 2017/2018

Signature of supervisor	Signature of Head of Department	Signature of Dean

Declaration

I declare that I have produced the submitted work independently and that I have provided all the information sources used in accordance with the Methodological Guideline on Compliance with Ethical Principles in the Preparation of University Graduate theses.

Acknowledgement

My deepest appreciations to doc. Ing. Pavel Mach, CSc. for endless assistance at every step from the start and until the completion of this thesis. Without him, this thesis would not be possible. I also want to pay my regards to Mrs Ing. Ivana Beshajová Pelikánová, Ph.D. for providing the required space and equipment in the laboratory and never-ending help at each step during the practical work.

Contents

Abstract

The thesis is focused on changes of the resistance and nonlinearity of current-voltage characteristic of adhesive joints formed of electrically conductive adhesives It is divided in three parts. The first part comprises of theory covering from the most basics of conductive adhesives, their working principle, properties, advantages and disadvantages, evolution of the technology with time and importance. The second part covers a larger part of thesis which describes in detail the experiment, preparation of samples, processing samples and exposure to stress in different forms, testing samples after the application of stress upon them, collection of data, processing of data, problems encountered during the experiment, critical factors and analyzing factors of the data gathered. The third part of the thesis covers the analysis of data, possible explanations and conclusions of the outcomes and summary of all the work.

Key words

Electrically conductive adhesives, Isotropic conductive adhesives, mechanical stress, ultrasonic stress.

Abstrakt

Práce je zaměřena na sledování změny odporu a nelinearity VA charakteristiky vodivých spojů tvořených elektricky vodivými lepidly. První část uvádí základní informace o vodivých lepidlech. Zabývá se jejich vlastnostmi, principy jejich funkce, jejich výhodami a nevýhodami a dále se také věnuje důvodům používání vodivých lepidel, technologickému vývoji jejich výroby a jejich významem pro použití v elektrotechnice a elektronice. Ve druhé, stěžejní části, je detailně popsán experiment s vodivými lepidly, příprava vzorků, zpracování vzorků a vliv různých forem namáhání na jejich parametry. Testované vzorky byly namáhány mechanicky statickým napětím a také zatěžovány ultrazvukem. V práci jsou popsány i kritické momenty, které se během testování vyskytly a způsob vyhodnocení naměřených dat. Analýzu výsledných dat a diskuzi příčin změn přináší třetí část práce. V závěru jsou shrnuty výsledky celého experimentu.

Klíčová slova

Elektricky vodivá lepidla, isotropicně vodivá lepidla, mechanické namáhání, ultrazvukové namáhání

Introduction

Using of electrically conductive adhesives is one possible way for making conductive joints in electrical and electronics applications. Many of the electrical joint applications are highly challenging by either being temperature sensitive, in micro-electronics applications where the components are often extremely sensitive to heat, under high mechanical or vibrational stress as in large electrical generators where the equipment is very heavy and produces extreme vibrations and environmental degradation, due to exposure to highly moist environment. The technology of ECAs is still under scrutiny over their performance and lacks sufficient data or maturity to be completely able to replace the lead soldering technology in some special applications. ECAs have come a long way and improved greatly in their performance over the last years but still they are not near good enough to be comparable, as their electrical, mechanical and climatic properties, with solders.

Efforts to improve the ECAs technology is going up with high pace to improve their conducting attributes, mechanical properties, climatic endurance and wettability and the most importantly, to reduce the costs incurred. This is mainly due in assembly of temperature sensitive components in advanced electronics and health concerns in the working environment around them.

1.1 What are electrically conductive adhesives?

Adhesives, in general, have existed since ancient times. From Egyptians, the glue from animal hides for furniture to amber for spears by Native Indians. Thanks to our modern-day technology and synthesizing capabilities, we have progressed from mud to synthetic conductive epoxies and other types of resisns, which we can customize as per our requirements. Most of our latest applications would be impossible without these adhesives.

In the simplest of definition, adhesives are glues, used to stick objects together. Individually, adhesives are electrically non-conducting or insulating. In modern times, the advancements in adhesive industry have been enormous. Industrially, adhesives come under the name of epoxies, drawn from epoxide functional group. Polyepoxides, also known as epoxy resins, are polymers, which exist in low weight molecular form or high weight molecular form. Epoxy resins are reacted together, either with themselves, or with a broad range of reactants also referred to as curatives. This reaction forms a thermosetting polymer, which possesses strong mechanical, temperature and resistive properties. Fig. 1 shows a generic epoxide, a cyclic ether with oxygen – O and CxHy bonded together in a triangle. The structure resembles an equilateral triangle enabling it to have strained structure. A compound is called an epoxy if it contains the epoxide functional group (see Fig. 1).

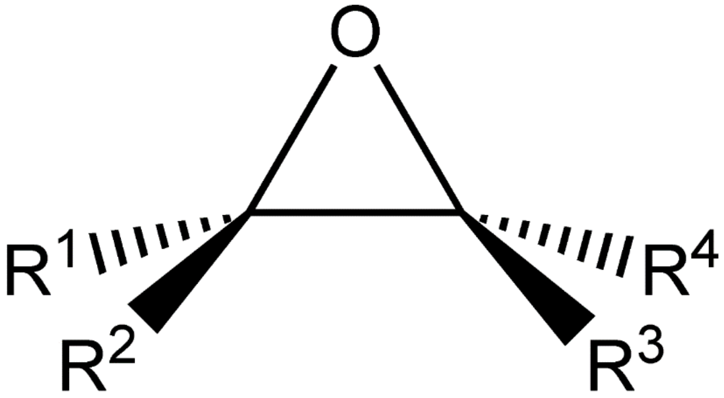


Figure 1 Generic Epoxide.

1.2 Importance of ECAs

The idea of ECAs evolved where the conventional soldering was not applicable. The high temperatures requested for soldering were damaging to heat sensitive electronics. The next question was how to make adhesives conducting? This dilemma was solved by adding conductor, filler, to the epoxy. This poses another question of which conductor to add. It is not possible to add just any one. If we add e.g. aluminum, it gets oxidized in air and covers by a thin film of an aluminum oxide (alumina). This film is insulating and therefore deteriorates the contacts between the filler particles in adhesive. Along with this, another important factor to be considered, was costs. Solders are very cheap. To minimize the cost incurred for conductive adhesives, silver was chosen as the conductor to be added in epoxies due to its high conductivity and low cost. The impossibility of using aluminum has already been mentioned and the same applies to copper. Therefore, epoxy resin filled with the silver flakes is the most common form of ECAs commonly used now.

Electrically conductive adhesives might sound very feasible and promising but they pose various drawbacks which cannot be entertained on a commercial sale just yet. One of the primary disadvantage of adhesives for commercial using is that soldering spends some seconds, whereas adhesive take time to be cured, ranging between 5 – 30 minutes or maybe even 24 hours if curing is to be done at the room temperature. However, new adhesives are being developed which offer “flash curing” feature. This means that they only need a few seconds to be cured.

In nontechnical terms, electrically conductive adhesives are known to be fundamentally environmental friendly and provide more applicable options as compared to soldering. Going into more technical description, ECAs provide both mechanical and electrical bonding for electrical joints, quick and easy bonding, can be cured at room temperature if the application is heat sensitive and fore mostly is safer than soldering in some applications. On a downside, the mechanical, electrical as well as climatic properties of adhesives are worse in comparison with the lead-free solders.

However, they have their limitations. Foremost limitations include lower electrical conductivity in comparison with the solders, rise in resistance of contacts when exposed thermally, faster corrosion compared to soldering due to the penetration of water molecules into the resin and the formation of oxide or hydroxide layers in the contacts between the filler particles and reworkability barrier. [1]

To counter the restrictions of the silver-filled epoxies, numerous designs have come to light recently. To fulfil the reworkability prerequisite, especially for high performance requirements, new thermoplastic resin is now being combined along with appropriate solvent and silver flakes. With leading researches and effort, reworkable thermoplastic conductive adhesives are developed offering higher electrical conductivity, better than Pb-Sn (Lead-Tin) solder. [2]

Along with the inability of conventional soldering to perform in several areas, one primary issues that lies with it is its environmental impact of toxic Lead(Pb). In typical Pb soldering applications, the composition of Pb is up to 40%. For most common applications, the use of lead soldering was banned in Europe starting 1st July 2006 under 'EU Directive ROHS on the restriction of the use of certain hazardous substances in electrical and electronic equipment (2002/95/EC)', with primary goal to cut down the release of Pb into the atmosphere. Only certain areas are still allowed to use lead-soldering where risk of hazard from Pb is very low and when the lead-free alloys, whose properties in long-time using are still not sufficiently known, could endanger human life. These applications are generalized by the soldering temperature below 240°C. Being said, the real issue which remains of hazard is the soldering fume which arises due to the flux (see Fig. 2). This phenomenon of flux is due mostly to colophony (rosin).

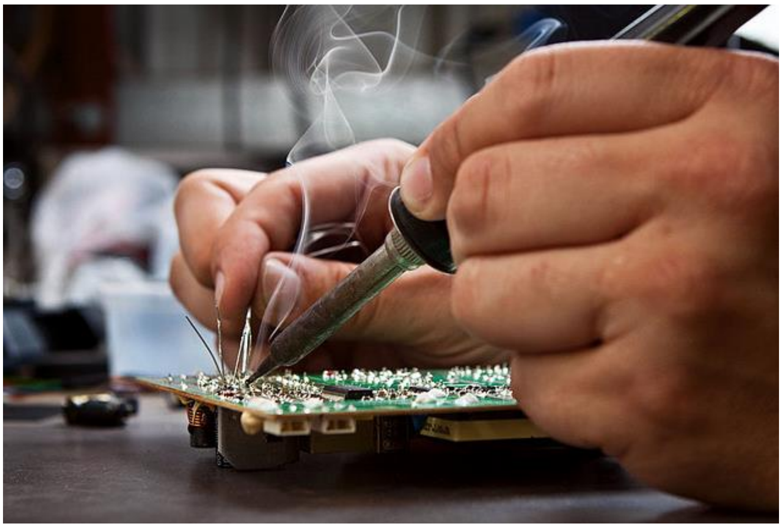


Figure 2 Solder fumes [3].

Colophony is obtained from pine and few other plants like conifers in solid form and is contained in the soldering wire. It is an important part of soldering to prevent:

1. Oxidation of components.
2. Remove contaminants.
3. Reduce surface tension of molten solder.

These fumes are well known source of respiratory sensitizer and is a major cause of work-related asthma.

The idea of replacing conventional soldering with adhesives is picking up more and more pace with new stable technologies coming on the market front which promise better performance than solders, environmental safety and, hopely in near future, price feasibility for the industry. [4]

1.3 Types of adhesives

ECAs comprise of different types, depending on the type of electrical conductivity.

1.3.1 Isotropic Conductive Adhesives:

ICAs are type of adhesives, which allow conducting of the electric current in all directions, in approximately equal magnitude. The polymer matrix, or simply the epoxy, is converted from insulator into conductor through adding a conductive filler component. Most commonly used filler is silver due to its low cost and high electrical and thermal conductivity.

This transition from insulator to conductor is highlighted by the percolation phenomenon. As the conductive filler is added, the resistivity of adhesive faces a dramatic drop with increasing concentration of the filler. This drop is until a critical concentration, the percolation threshold, at which the conductive filler forms the first conductive way between the contacts. As the concentration of conductive filler surpasses the percolation threshold, the decrease in resistivity is minimal. The polymer matrix of epoxy resin provides the mechanical interconnection to the adhesive joint. However, a higher concentration of conductive filler will weaken the interconnection. It is very important to find a balance for the conductive filler concentration, high enough to ensure good conductivity but limited to a level to keep the mechanical properties of adhesives intact. [5]

Figure 3 illustrates a basic schematic of ICA.

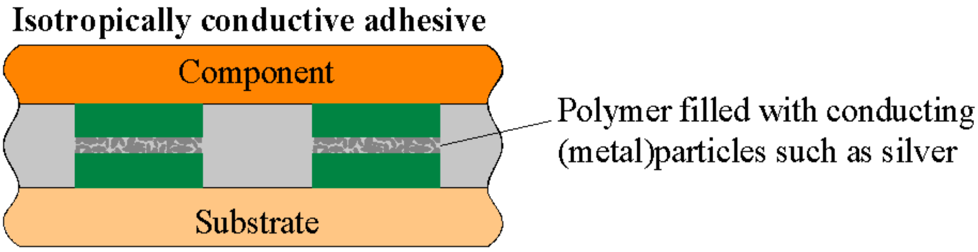


Figure 3 ICA [6].

1.3.2 Anisotropic Conductive adhesives:

ACAs are type of adhesives in which we limit the direction of conductivity. It can be only one-directional. The electrical conductivity if high in direction of the z-axix, whereas the conductivity in the x- and y-axis is determined by the electrical conductivity of the insulating matrix. Therefore, these adhesives are sometimes called "z-adhesives". To make this application work, the concentration of conductive filler is kept lower than the percolation threshold, which stops the adhesive to be in conductive mode until the interconnections are formed. The concentration of filler in adhesives with isotropic electrical conductivity is 65-80 % b.w usually, whereas the concentration of filler particles in adhesives with anisotropic electrical conductivity is 10-15 % b.w. usually. Whereas the ICAs are only cured, the ACAs must be cured under pressure.

As soon as the ACA is introduced in-between the conductors, interplanar connections are formed through the pressure and heat. The primary difference between isotropic conductive adhesives (ICA) and anisotropic conductive adhesives (ACA) is that in ACA the adhesive is in between the mating contacts, whereas ICA is on the contact. During curing using heat and pressure the conductive filler is trapped between the mating contacts. They undergo deformity to form and electrical connection. This allows the restriction of electrical conduction only in z-axis and the x-y planes remain insulated.

When the adhesive undergoes cooling, it experiences residual stresses. This is because of the polymer matrix experiences contraction. Another form of residual stress is formed when the polymer matrix shrinks while being cured. Comparatively, the stress formed during cooling is much dominant than the stress formed during curing. This stress is sufficient enough to form a low-resistance stable connection. [7]

Electrically conductive adhesives with anisotropic electrical conductivity are used for mounting of integrated circuits having low gap between leads (e.g. “fine pitch” or “ultra-fine pitch” packages), the reason being, using soldering for these packages is joined with high frequency of bridges between adjacent leads. Figure 4 gives a rough idea of the how ICA connection is formed. Fig. 5 gives a visual picture of how the percolation theory applies to ACA or ICA joints and how the percolation threshold holds in either of the adhesive joint.

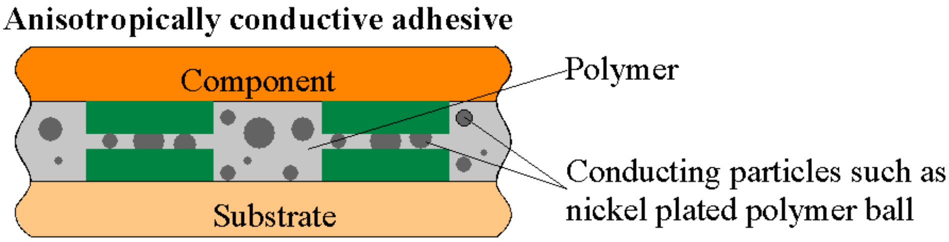


Figure 4 ACA [8].

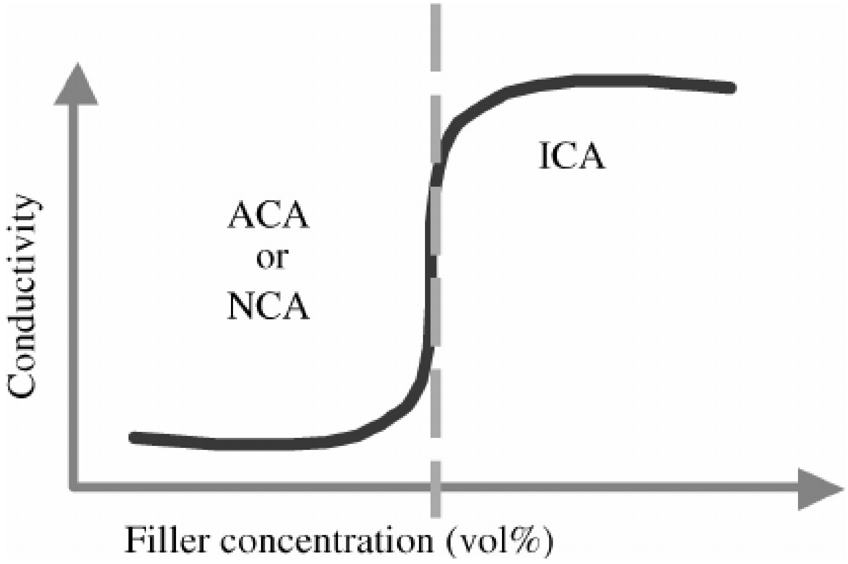


Figure 5 Percolation characteristics in ACA and ICA [9].

1.4 Critical Factors for adhesives

1.4.1 Conductive filler:

One on the most important factor for the quality of adhesives is the conductive filler used and shape and dimensions of the particles. A conductive filler is composed of powdered form of conducting elements like gold, silver, carbon or graphite, nickel and so on. What influences the quality of adhesive, measured by the resistance it displays, is the shape of these filler. The shape can either be of flakes (Fig. 6) or balls (Fig. 7), depending on the application. Flake fillers exhibit least resistance, once the curing process is finished and are used with ICA applications. This is due to the higher contact surface they provide for conduction whereas, the ball shape provides less contact surface for conduction and are applied in ACA applications where the conductivity is caused by catching of individual balls between joined contacts as illustrated in Fig. 4.

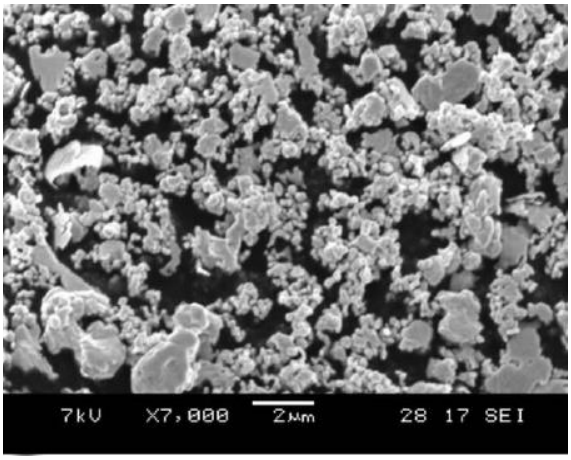


Figure 6 Silver flakes [17].

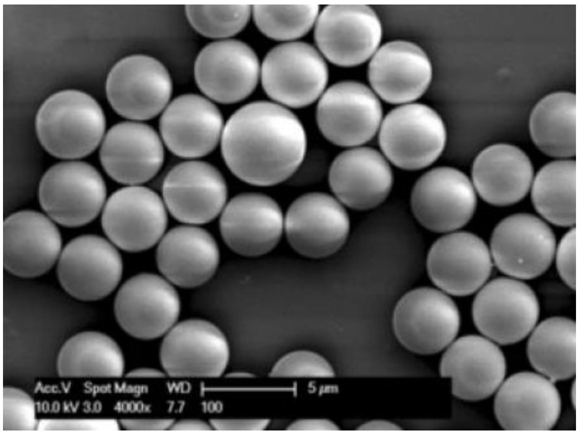


Figure 7 Ball shaped filler (PMMA particles covered with thin Ni film) [18].

However, only using flakes increases the thixotropy ratio [defines how much the fluid reduces and how quickly they return to the original state]. They are also known as non-newtonian liquids [11]. Thixotropy refers to the shear-thinning characteristic, which is time dependent. This means the adhesive will undergo reduction in its viscosity more quickly under stress as the thixotropy ratio increases. To strike an optimum level of filler and epoxy, a combination of ball and flake is sometimes used.

1.4.2 Binder (epoxy):

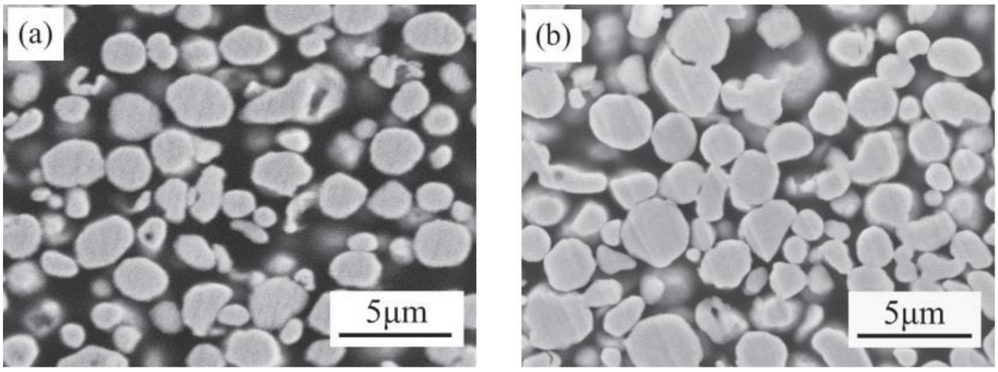
Along with filler, the binder is also very important in determining the quality of the adhesive. Binders depend on the type of application for the ECA. Binders are composed of polymer matrix, which helps to bind the filler together. As we cure the adhesive, the binder shrinks its structure. This property of shrinking structure is applied to hold the filler together and break the insulation. Reasons, why binders play a significant role are:

- Each individual binder has a different rate of shrinkage during the curing process. This implies that if the rate is higher, the filler comes closer and breaks the insulation.

- Wettability of individual types of fillers by different binders is different. This phenomenon is described by surface polarity of both filler and binder. If wettability is low, the filler is rejected by binder as it undergoes the curing procedure. The fillers don’t come in close enough vicinity of each other and thus the resistance increases.

- Each individual binder possesses different resistance. Filler being surrounded by the binder, will come closer to each other if the resistance of binder is low. [12]

- Each individual binder reacts differently to the curing process. Some binders shrink their matrix quickly, some shrink more at room temperature, some shrink more at higher temperature. As an example, the following SEM images (Fig. 8) taken from a research paper illustrate the effect [13]:



Cross-sectional SEM images of the adhesive containing Ag fillers cured at (a) 373K for 1 h.

Cross-sectional SEM images of the adhesive containing Ag fillers cured at (b) 453 K for 1 h.

Figure 8 SEM images for curing at (a) 373K and (b) 453K for 1 hour [14]

Depending on the area of application, the binder is chosen accordingly to accommodate the requirement of operation.

Experimental Part

2.1 Research

The study focuses on the influence, if any and if yes then to what extent, mechanical stress will cause on the adhesive joints. This is a very important aspect in high end performance electronics where there might be quick and dynamic applications or large electrical applications where there are vibrations within the system. By theory, there can be a certain level of improvement in the quality of joints, lower resistance, until the adhesive joint suffers a fracture. There is another possibility that the quality of joint decreases as the stress might influence the adhesive to increase in resistance. This depends on the type of binder used and their interaction with flakes when cured. In our study, we will try to determine how mechanical stress will affect the quality of joints, first with low deflection, for about 2.5mm and then higher deflection of about 5mm. The deflection will be carried out over a curved surface with the sample bolted at both ends.

Apart from mechanical deflection, the concept of ultrasonic stress will be incorporated in the study for the first time. Influence of a ultrasonic stress on the adhesive joints has not be still published in the literature. As mentioned earlier, there can be high frequency vibrations in electrical systems (e.g high powered generators). This aspect of stress in ECAs is still unexplored. Some of the possibilities include total failure of adhesive joints, due to large impulsive force applied on the joints. On the other hand, the intense vibrations might push the filler together to come closer, resulting in a decrease in resistance of the joints. This is something which might be revealed during the experimentation and analysis of the data gathered and based on initial findings, whether if the ultrasonic stress has any influence or not, we can conduct further detailed study to determine the exact nature of effect. There might be several factors that will bring about a change in the joint properties, the duration of stress, the amplitude of vibrations, the point of contact, the angle of propagation and heat generated during the vibrational stress. The research will focus on ultrasonic stress in form of vibrations over the joints using a probe directly in contact with the joints.

2.2 Strategy

The basic strategy for executing the objective of this research, in order to draw conclusions, includes:

- preparation of samples,
- measurement of initial values,
- application of stress over the samples,
- measurement of final values,
- drawing conclusion

2.2.1 Preparation of samples:

The first phase of preparation involves evaluation of available resources, quantifying the samples for each respective category and joining all the initial components to prepare the samples for measurement. We make use of the following material:

- Customized printed circuit boards (PCBs) with gold (Au) and copper (Cu) surface finish. The layout of the test PCBs has been designed to be possible to provide four-point measurement of the joints resistances.
- 0R0 resistors. The resistors for SMT adhesive assembly of the type 1206 were used.
- Adhesives. One type of a two-component and two types of a one-component adhesives were used. All adhesives were of ICA type.
- Printing Stencil.
- Pick and place SMT machine.
- Curing oven.

2.2.2 Printed Circuit Boards

The PCBs used in the experiment were ordered with customized specifications, with place to fix 4 resistors in each sample. Each resistor has two terminals along with one input and one output terminal. There are two spots from adhesives for each resistor. The samples are divided 50-50 ratio between gold and copper surface finish (see Fig. 9 and Fig. 10).

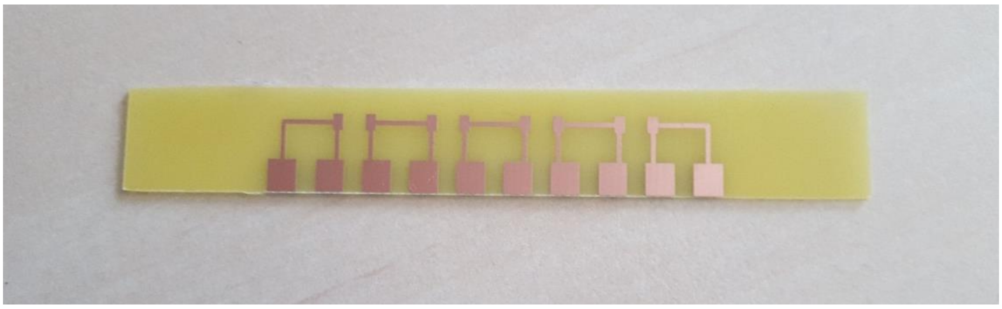


Figure 9 PCB with Cu layout.

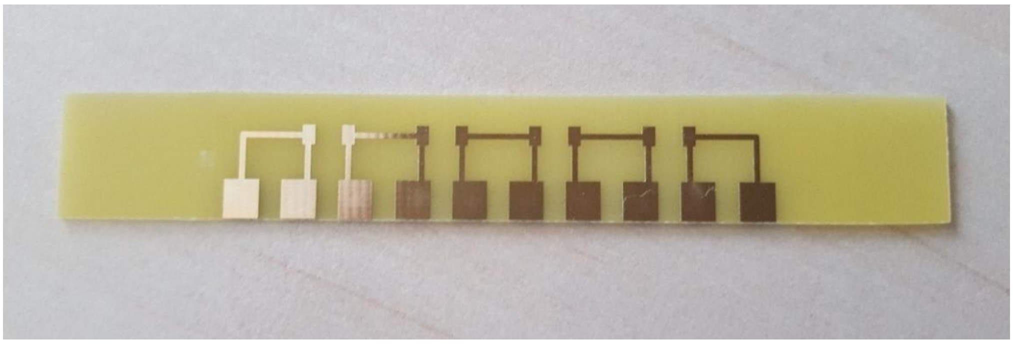


Figure 10 PCB with Au layout.

2.2.3 "Zero"-Ohm resistors

0R0 resistors (see Fig. 11), also known as jumpers, of the type 1206 to form adhesive joints have been used. The resistors have been mounted on PCBs by adhesive assembly using isotropic electrically conductive adhesives.

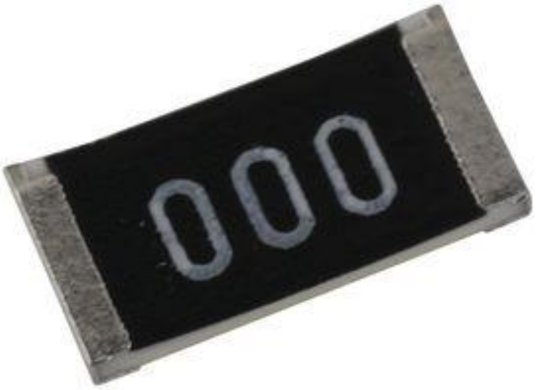


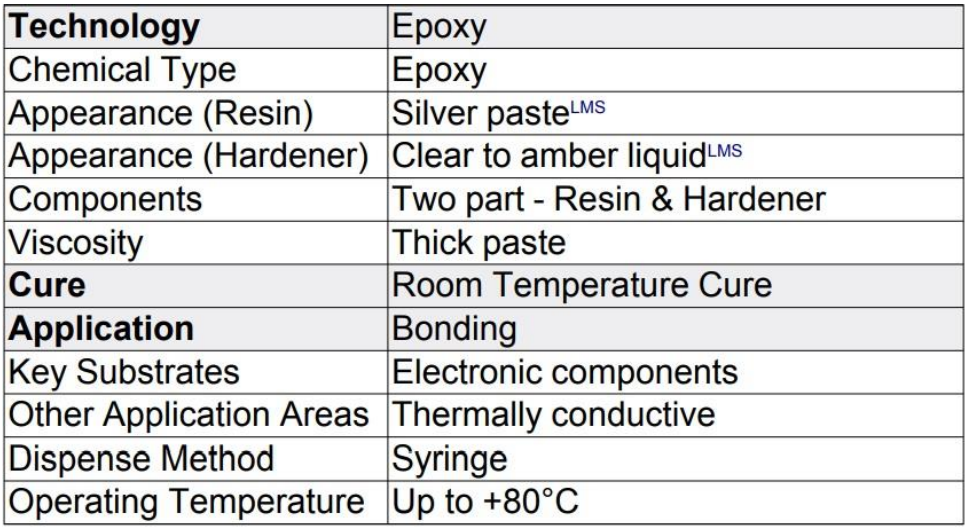
Figure 11 0Ω SMD resistor [15]

2.2.5 Adhesives

The adhesives we employed are isotropic conducting adhesives (ICA). For the experiment, as per direction from supervisor, three different brands of ICAs were acquired namely:

2.2.5.1 Henkel LOCTITE Ablestik 3888 TPK:

Loctite 3888 (see Fig. 13) is a silver filled two-component conductive adhesive with a mix ratio of 100:6 (resin:hardner). Fig. 12 shows the properties, as given in the datasheet by Henkel for this adhesive.



Technology	Epoxy
Chemical Type	Epoxy
Appearance (Resin)	Silver paste^{LMS}
Appearance (Hardener)	Clear to amber liquid^{LMS}
Components	Two part - Resin & Hardener
Viscosity	Thick paste
Cure	Room Temperature Cure
Application	Bonding
Key Substrates	Electronic components
Other Application Areas	Thermally conductive
Dispense Method	Syringe
Operating Temperature	Up to +80°C

Figure 12 Datasheet from Henkel.

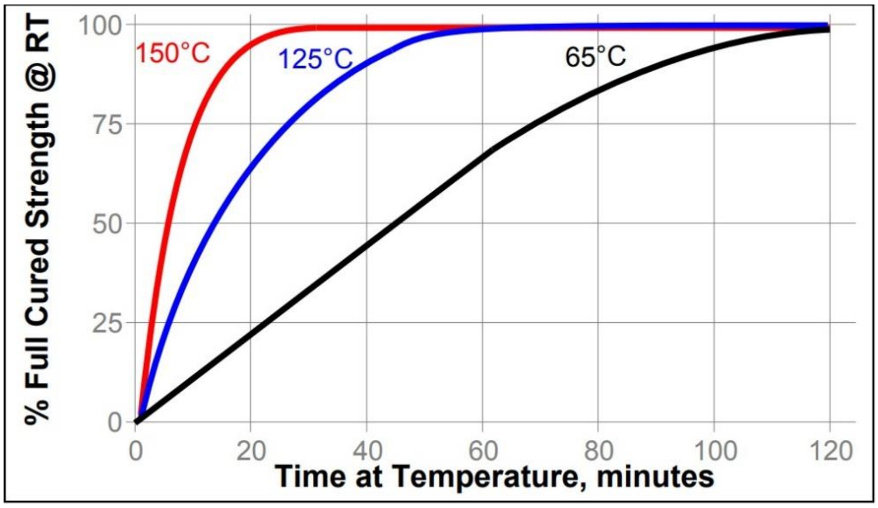


Figure 13 Curing curves obtained from the datasheet published by Henkel.

LOCTITE

ABLESTIK 3888 TPK

Made in the USA
Henkel Electronic Materials, LLC
14000 Jamboree Road, Irvine CA 92606
Telephone: 1.888.9.HENKEL (1.888.943.6535)
henkel.com/electronics

Sub-Descriptor:
ORDER #: 2000482711
IDH: 2027182
QTY:
BATCH: 046LAA2711
DOM: DEC-14-2016
EXP.DATE: JUN-15-2017
Storage Temp:Room Temp/Ambient

Henkel

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ABLESTIK 3888 Part-R

ABLESTIK 3888 PART-H

FOR INDUSTRIAL USE ONLY. Read Safety Data Sheet before use.

Figure 14 Packaging for LOCTITE 3888.

Two-component adhesives are basically manufactured in two parts, which are mixed together per the ratio at the time of consumption. With this packaging, it is not important to determine the mixing ratio, as the resin and hardner are packed in the prescribed ratios directly. The most feasible property of this ICA is its curability at the room temperature (see Fig. 14).

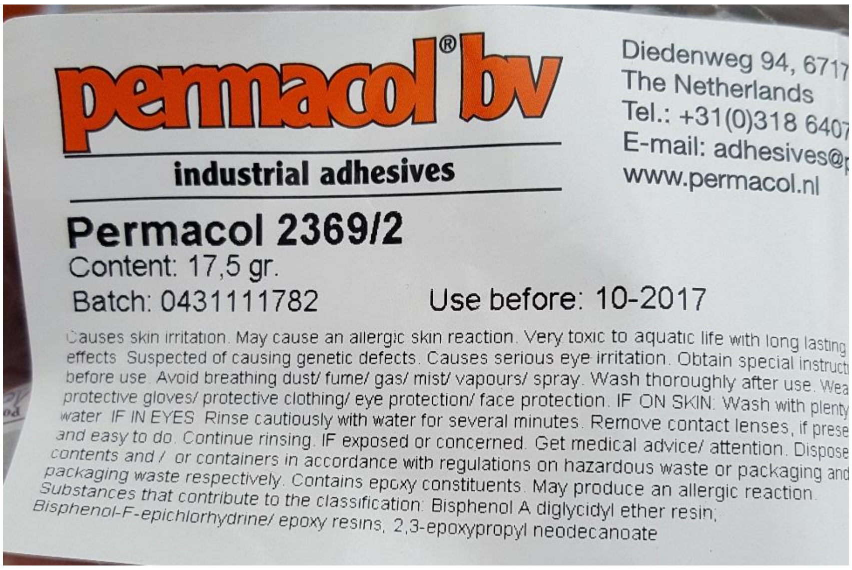
2.2.5.2 Permacol bv 2369/2:

Second choice for adhesive was 2369/2 by Permacol as shown in Fig. 15. It is a single-component ICA with fast curing properties. The data given in table 1 was acquired from the dataseet provided by Permacol for the product:

Table 1 Properties of Permacol 2369/2 as published by the manufacturer.

Base	Epoxy
Colour	Silver
Viscosity	30.000 mPa·s
Particle size	<50 µm
Application method	dispensing / stencil printing
Curing schedule 150°C	3 min.
125°C	6 min.
110°C	10 min.
100°C	15 min.
80°C	60 min
Volume resistivity	<3x10-4 Ω.cm
Hardness	70° Shore D

However, for this ICA, curing is preferred to be done at least at 125°C or more because at temperature lower than this, there would be an increase in volume resistivity. Furthermore, this adhesive should be stored in either a fridge or a freezer, with varying shelf life in either refrigerator at 4°C until 3 months or in freezer at -18°C until 6 months.


permacol® bv
industrial adhesives

Diedenweg 94, 6717
The Netherlands
Tel.: +31(0)318 6407
E-mail: adhesives@p
www.permacol.nl

Permacol 2369/2
Content: 17,5 gr.
Batch: 0431111782 Use before: 10-2017

Causes skin irritation. May cause an allergic skin reaction. Very toxic to aquatic life with long lasting effects. Suspected of causing genetic defects. Causes serious eye irritation. Obtain special instructi before use. Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray. Wash thoroughly after use. Wea protective gloves/ protective clothing/ eye protection/ face protection. IF ON SKIN: Wash with plenty water. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if prese and easy to do. Continue rinsing. IF exposed or concerned: Get medical advice/ attention. Dispose contents and / or containers in accordance with regulations on hazardous waste or packaging and packaging waste respectively. Contains epoxy constituents. May produce an allergic reaction. Substances that contribute to the classification: Bisphenol A diglycidyl ether resin; Bisphenol-F-epichlorhydrine/ epoxy resins, 2,3-epoxypropyl neodecanoate

Figure 15 Permacol 2369/2 packaging as received from the manufacturer.

2.2.5.3 Amepox Elpox AX 70MN:

Elpox AX70MN (Fig. 16) is a Polish product, which seems to be promising replacement of conventional soldering by description. Sold under the name of ‘ECO SOLDER’, AX70MN is a single component ICA with conductive silver filler concentration around 70% (see Table 2. for detailed information about the product). It is applicable with either the standard dispensing or using stencils, does not require refrigeration and has a longer shelf life (up to 6 months) as compared to other two adhesives mentioned previously. It also has a fast curing time, as fast as 3 – 4 minutes.

The following data is acquired from the datasheet for ELPOX AX70MN provided by Amepox:

Table 2 Properties of Elpox AX 70MN as given in datasheet by manufacturer.

Number of components	One
Consistency	Floable paste
Color	Bright silver
Percentage of silver (inside ready paste)	70 ± 1%
Specific gravity	2.1 – 2.4 g/cm³
Viscosity	530 000 – 560 000 cps (*)
Drying time before curing process	Not necessary
Recommended curing schedule with air-circulated oven	180°C – (6 - 10) min. 200°C – (3 - 4) min
Recommended curing schedule with heating tunnel	200°C in peak – 3 min. total time inside tunnel
Shelf life	6 months (when storage at 10°C – unopened)
Electrical resistivity	(1.0 – 2.5) x E(-6) Ωm
Range of service for continuos temperature	(-55)°C - (+200)°C
Max. operating temperature	300°C for ab 1.5 h.

Figure 16 ELPOX AX70MN packaging as received from the manufacturer.

2.3 Procedure

To cater an adequate amount of data to draw any credible conclusion, number of sample is critical to be set. We divided each case of stress specifically as No Deflection (ND), Low Deflection (LD), High Deflection (HD) and Ultrasonic Stress (US). Next step was to agree upon number of samples for each case. For each case of stress, it was decided to have 8 samples for each ECA in each case. These 8 samples are divided further into 4 Au and 4 Cu samples. Table 3 gives a clear picture of the samples:

Table 3 Organisation of samples.

ECA/Case	**ND**	**LD**	**HD**	**US**
LOCTITE	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu
Permacol	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu
Elpox	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu	4 Au, 4 Cu

In total, 96 samples were agreed upon to be prepared. Because on one sample there are mounted 4 0R0 resistors, one sample represents 8 adhesive joints. It means that for every adhesive and every situation 32 adhesive joints were fro disposal. First step in preparation was to drill holes on each end of the samples for mounting them on fixtures with bolts. The drilling position on the sample is illustrated in the Fig. 17:

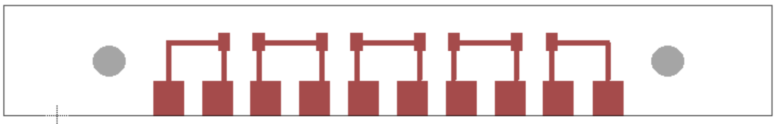


Figure 17 PCB sample Layout with hole position

The holes were made for 3.5mm each to accommodate the bolts 3mm thick. 0.5mm offset was given to accommodate the variance in deflection and set the deflection at different levels.

Next step to follow was printing with the PCBs with each ECA. For this purpose, we used a printing stencil (see Fig. 18), custom made to fit our requirements. The stencil was capable of printing 12 samples in each batch, but for accuracy purpose, only 6 sample were printed in each batch.

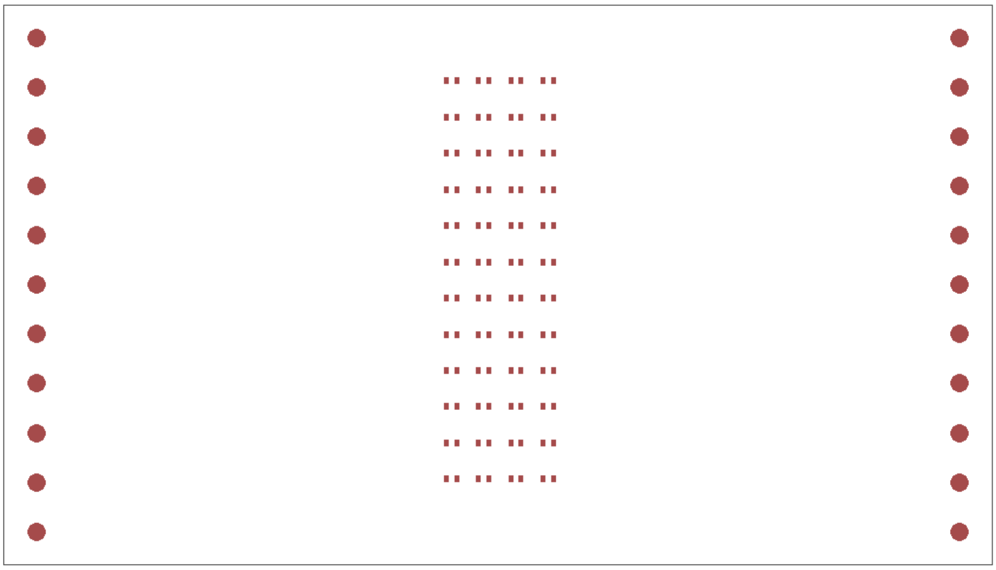


Figure 18 Layout of printing stencil

For accurate printing, setting the stencil accurately mounted on the manual application device (see Fig. 19) for printing.

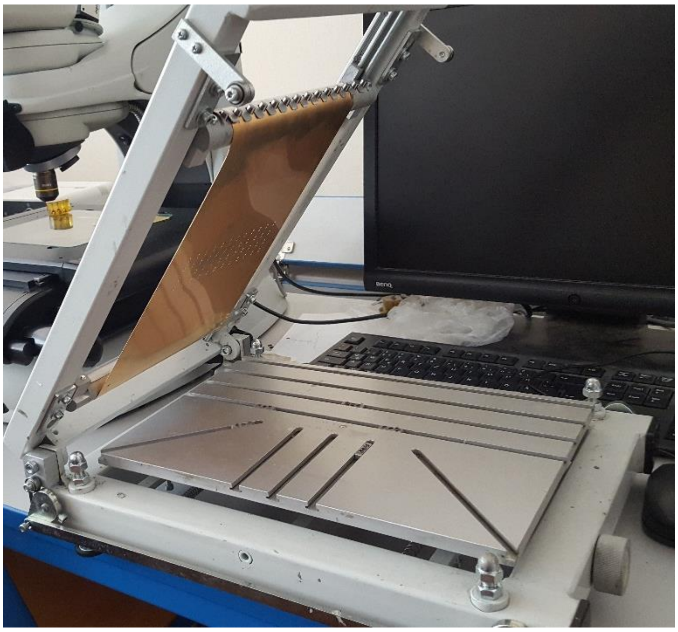


Figure 19 Manual application device for printing.

Few of the samples were wasted in the start due to inaccurate settings for the stencil but once it was set accurately, the printing was swift and easy. To spread the adhesive across the stencil, the squeeze must be swept at approximate angle of 60°. This was a critical factor for printing as a lower angle would increase the pressure on the stencil and press the adhesive causing it to spread on the PCB. An angle more over 60° was not suitable for evenly spreading the adhesive.

Instead of doing all samples at once, the task was divided for each adhesive, one at a time. After preparing first 32 samples for one adhesive, we used the SMT pick and place machine to put the 0R0 resistors on each joint.

Next is the stage of curing. After placement of resistors, the samples are left for curing, either at room temperature, or in curing oven depending on the ECA used. Table 3 summarizes the curing conditions for the three adhesives used:

Table 4 Curing time and temperatures used for curing.

LOCTITE 3888	Room temperature for 24 - 48 hrs
Permacol	125°C for 6 minutes
Elpox AX 70MN	180°C for 8 minutes

Curing is the last stage and after all the samples with each adhesive are cured (see Fig. 20), the samples are ready to undergo stress procedures.

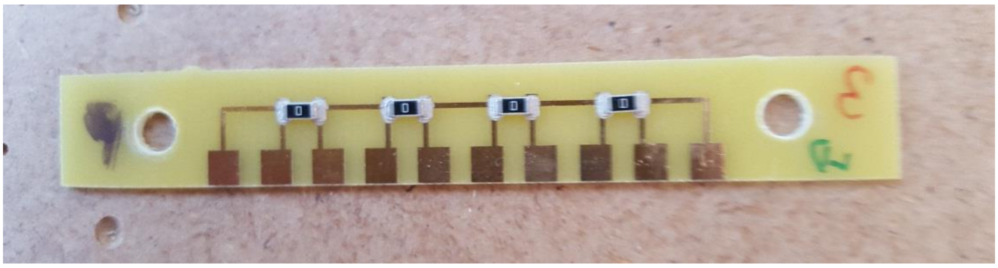


Figure 20 Resistors placed on the sample and cured.

2.4 Experimentation with samples

Experimentation part is divided in 3 parts. The first part focuses on low deflection followed by high deflection and then ultrasonic stress. These parts will highlight the procedure and conditions in which the prepared samples were exposed to undergo the changes.

2.4.1 Low deflection

To achieve low deflection, the PCB samples were mounted on a curved fixture with the help of bolts at each end. The fixture was custom built to suit our requirements. Low deflection is defined in terms of the bend a sample undergoes from the horizontal reference line along each end. Fig 21 below gives the idea of how the samples were mounted on the fixtures. For our lower deflection scenario, we keep this at 2.5mm approximately. According to the calculations made in [16] the tensile force was estimated to be 0.8 N and the shear force 10N.

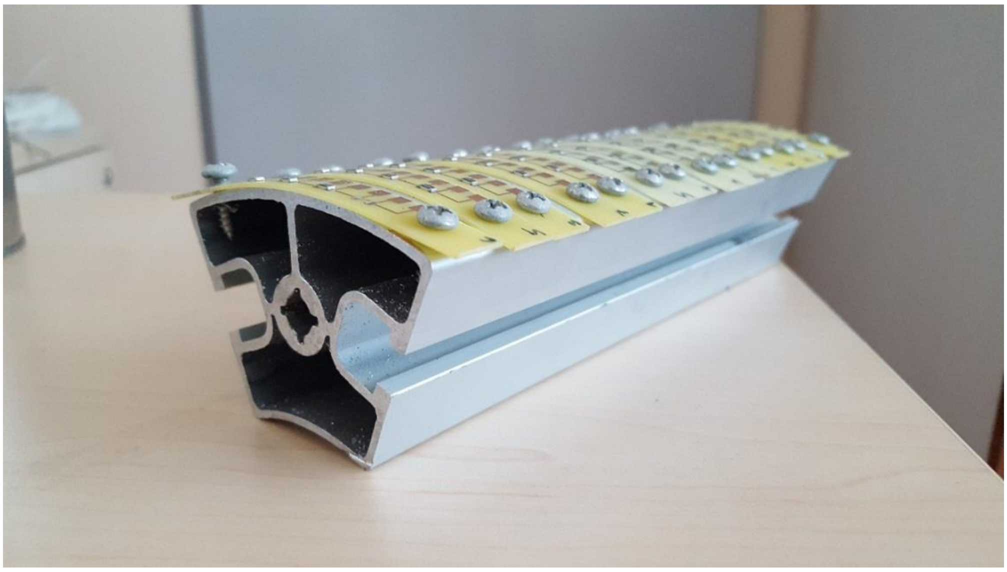


Figure 21 PCB samples mounted on curved fixture.

After mounting all the samples at approximate deflection of 2.5mm, the fixtures with samples are left at room temperature and conditions for 300 hours as per discussion with the supervisor. The mechanism of deflection can be see above in Fig. 22.

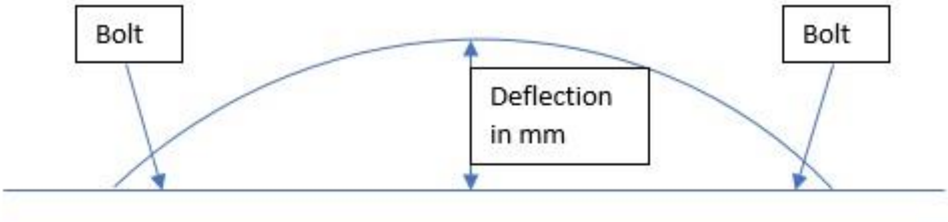


Figure 22 Mechanism of deflection in samples.

2.4.2 High deflection

The second part of experimentation consists of giving the PCB samples a high deflection of approximately 5mm. We follow the same mechanism as shown in Fig. 22, only this time bolting the PCB samples tighter. This makes the samples to bend more, thus increasing the deflection. After mounting, we follow the same protocol of keeping the samples at room temperature and conditions for 300 hours as in the case of lower deflection.

Some of the samples failed under stress of high deflection. The resistors were knocked out by the stress sample experienced. Keeping this risk in mind, we created extra samples already during the preparation part. According to the calculations made in [16] the tensile force was estimated to be 1.2 N and the shear force 20 N.

2.4.3 Ultrasound stress

This part of experimentation was difficult to put together. Firstly, we needed a source of ultrasound stress. The next step was to determine a method to transfer that stress on the samples. Out of two sources available, one being ultrasonic bath and second being ultrasonic probe, ultrasonic probe was chosen to be the source of stress for the samples. The source is Bandelin Ultrasonic homogenizer (see Fig. 23 and Fig. 24) used for homogenizing mixtures of all kinds, acceleration of catalytic chemical reactions, emulsifying up to the minimal droplet size and other various uses where ultrasonic motion is required. The equipment is programmable to specific needs.

Figure 23 Bandelin Ultrasonic homogenizer

Figure 24 Bandelin Ultrasonic homogenizing probe.

Fig. 25 gives a visual display of how the setting appear on the screen. There can be temperature control (not to let the temperature rise above a specific value), amplitude control of the vibrations, time control, pulsation control and energy display. For our requirement, we set the amplitude to be 50%, no pulsation and temperature limit to 50°C.

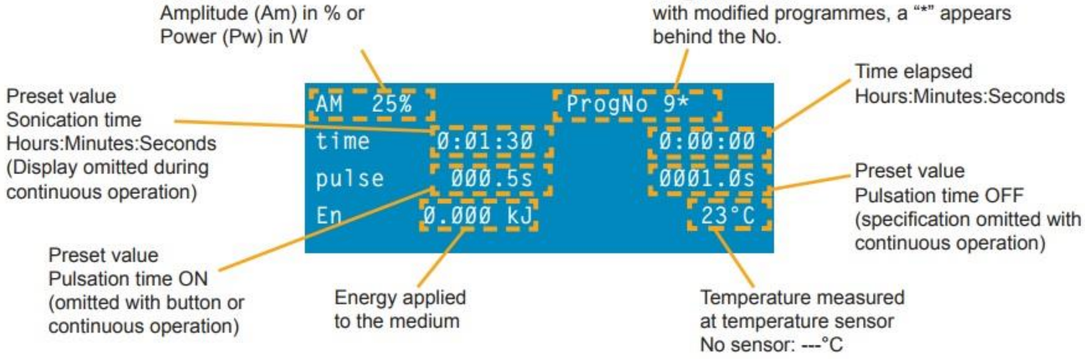


Figure 25 Bandelin Ultrasonic homogenizer HD3200 display.

The next part is to transfer this ultrasonic stress onto the samples. The method was difficult to be determined. First idea method tried was to mount the samples on horizontal platform and expose the sample to the ultrasonic stress in the middle. This method was not very adequate as the contact was not on the joints and not evenly spread across all eight joints. After various attempts, a method of manually contacting each joint to the probe was decided. One important factor in the process was to fix an exposure time. Exposure time was decided by trial and error method of exposing and then measuring the sample for any measurable changes in the values of resistance.

It is also important to keep the exposure time low as higher exposure time posed a risk of samples to fail (due to high mechanical vibrations). For the exposure time, 1 minute for each sample and 7.5 seconds approximately for each joint were agreed upon. Since the procedure was to be done manually, the exposure can be taken as approximately equal only. Each sample was handled individually, one at a time, touching each joint for approximately 7.5 seconds to the end of the probe.

The samples prepared with Elpox AX 70MN ICA failed under ultrasonic stress. At least one of the resistor or more were knocked out from each sample.

2.5 Measurement

For the measurement of results and the influence stress, if any, on the samples was the most time-consuming part. At first, it was necessary to define the factors by which the performance and changes in characteristics can be measured. As per agreement, two factors were chosen to be the measuring factors for the samples’ performance; Resistance across the joints and non-linearity of the sample.

2.5.1 Resistance

For any adhesive, its resistivity is the measure of the quality. Every adhesive is aimed to have maximal conductivity. The performance of the adhesive joints would be gauged with reference to the resistance it offers. Either the resistance should decrease, remain the same or increase after the sample went through the stress.

Since the joints and resistor offer very low value of the resistance (in milli-ohms), the accuracy of measurements was of a critical factor. For this measurement purpose, we used milli-ohmmeter Hewlett Packard (HP 4338B as shown in the Fig. 26). It is a very precise, 4-point contact, measuring device, which can measure the resistances in milliohms very accurately.

Figure 26 HP 4338B Milli-ohmmeter.

Fig. 27 below illustrates the measurement process. The values obtained for resistance of each sample can be found in detail in Appendix 1.

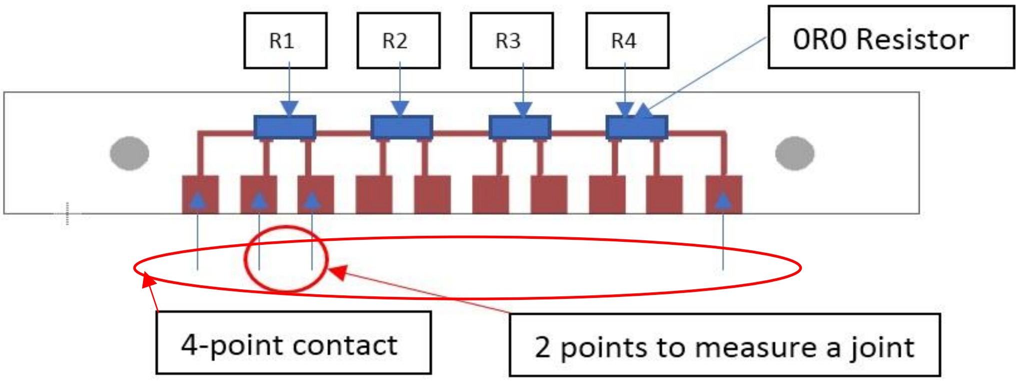


Figure 27 illustration for 4-point contact measurement across a joint.

A joint includes the resistor and two adhesive joints on each side of the resistor. The 4-point contacts are traversed through the sample covering each joint one by one. As a convention, resistor from left side is taken to be R1 and to the right end as R4.

For comparison purpose, all the samples are measured for resistance to be compared with the changes in resistance after the stress. Same procedure is repeated for LD, HD and US cases in the same way as described above.

2.5.1.1 Permacol

The chart for the resistances of the joints made of the Permacol adhesive (with Au and Cu surface finish of pads) are given below in Fig. 28.

Resistance of joints formed of Permacol adhesive

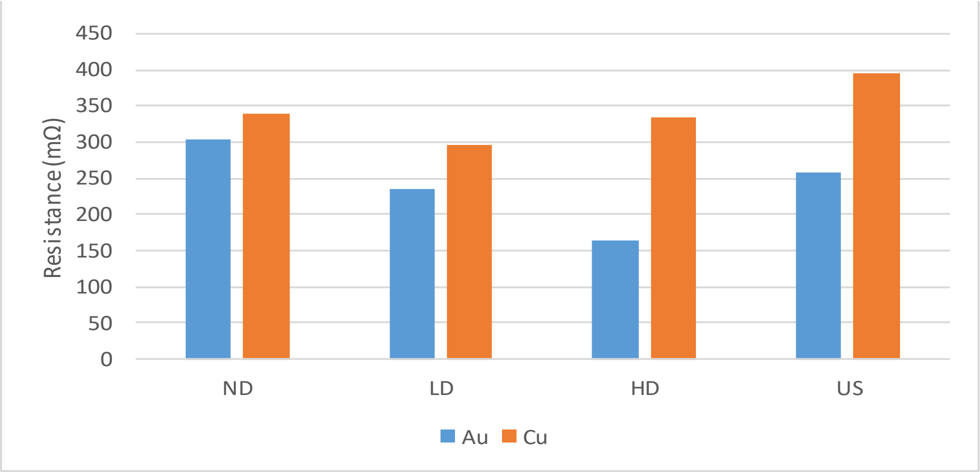


Figure 28 Permacol sample resistance calculated as median for each individual case: No deflection (ND), Low deflection (LD), High deflection (HD) and Ultrasonic Stress (US), with both Gold (Au) and Copper (Cu) contact pads.

2.5.1.2 LOCTITE

The chart for the resistances of the joints made of the LOCTITE adhesive (with Au and Cu surface finish of pads) are given in Fig. 29.

Resistance of joints formed of LOCTITE adhesive

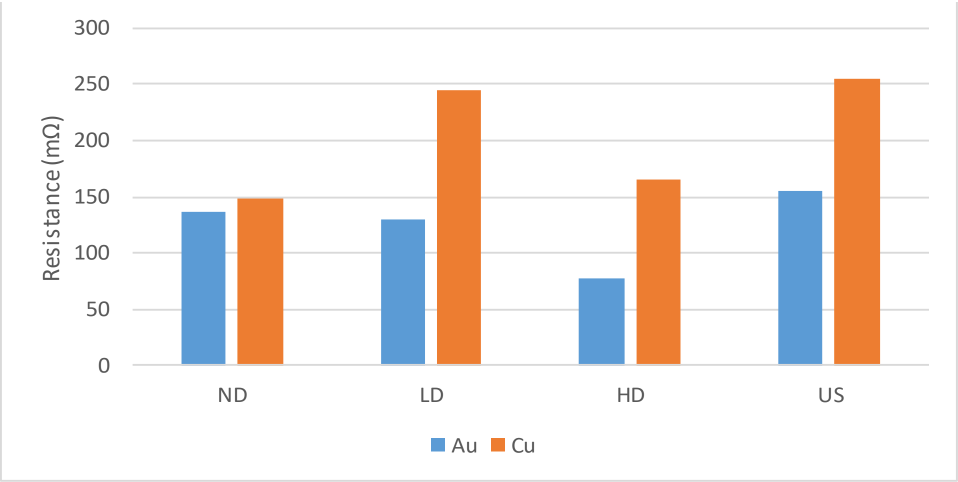


Figure 29 LOCTITE sample resistance calculated as median for each individual case: No deflection (ND), Low deflection (LD), High deflection (HD) and Ultrasonic Stress (US), with both Gold (Au) and Copper (Cu) contact pads.

2.5.1.3 AX 70MN

The chart for the resistances of the joints made of the AX70MN adhesive (with Au and Cu surface finish of pads) are given below in Fig. 30.

Resistance of joints formed of AX70MN adhesive

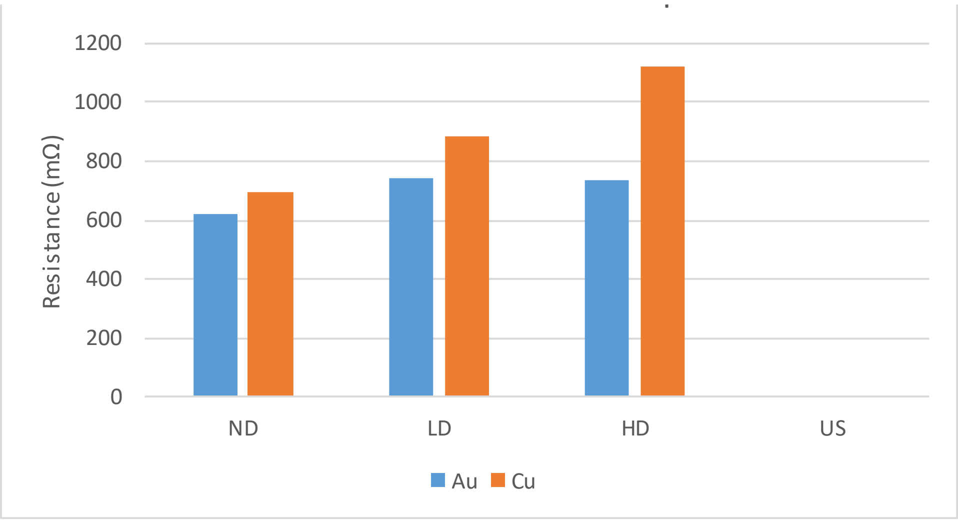


Figure 30 AX70MN sample resistance calculated as median for each individual case: No deflection (ND), Low deflection (LD), High deflection (HD) and Ultrasonic Stress (US), with both Gold (Au) and Copper (Cu) contact pads.

2.5.2 Non-Linearity

Nonlinearity of a nominally linear component (Fig. 31) is a significant parameter, which informs about the difference of and investigated component from an ideal one. The higher is quality of the nominally linear component; the lower is level of its nonlinearity. Nonlinearity is caused by inhomogeneity and unstable barriers in the component, which can, after some time, cause failure of the component. Nonlinearity is also a significant technical parameter of components, which work with more signals of different levels.

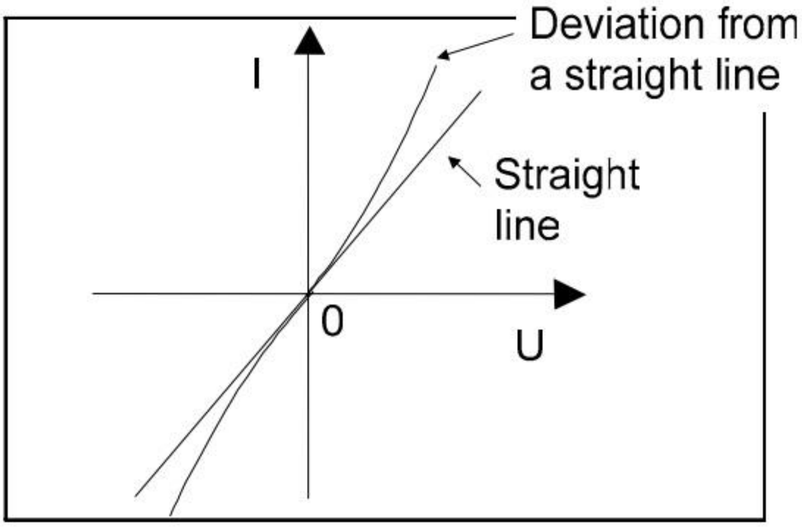


Figure 31 Current vs Voltage if an ideal and a real nominally linear component.

Basic principle used for such the measurement involves a nonlinear component, powered by a sinusoidal current and voltages of the first and third harmonics are measured. A schematic diagram of equipment for such the measurement is shown in Fig. 32.

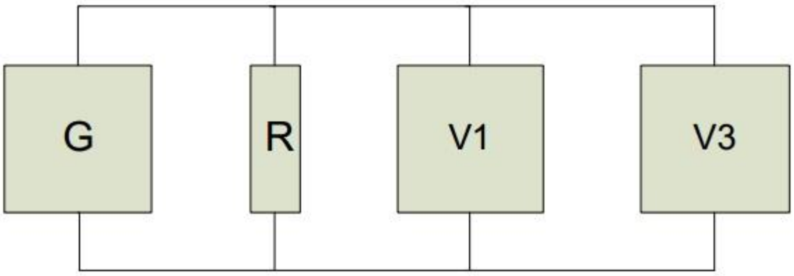


Figure 32 Schematic diagram of equipment for measurement of non-linearity.

Where G = generator of the sinus current having frequency of 10 kHz with very low distortion, R = measured joint, V1 = selective millivoltmeter with measuring frequency equal to the frequency of first harmonic component, V3 = selective millivoltmeter with measuring frequency equal to the frequency of third harmonic component.

The results for non-linearity (µV) are given as follows:

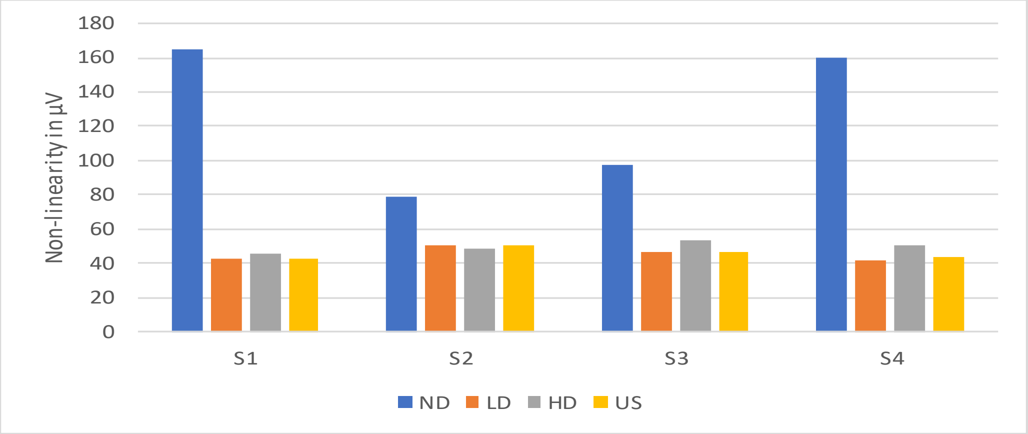


Figure 33 Permacol Au surface finish pads.

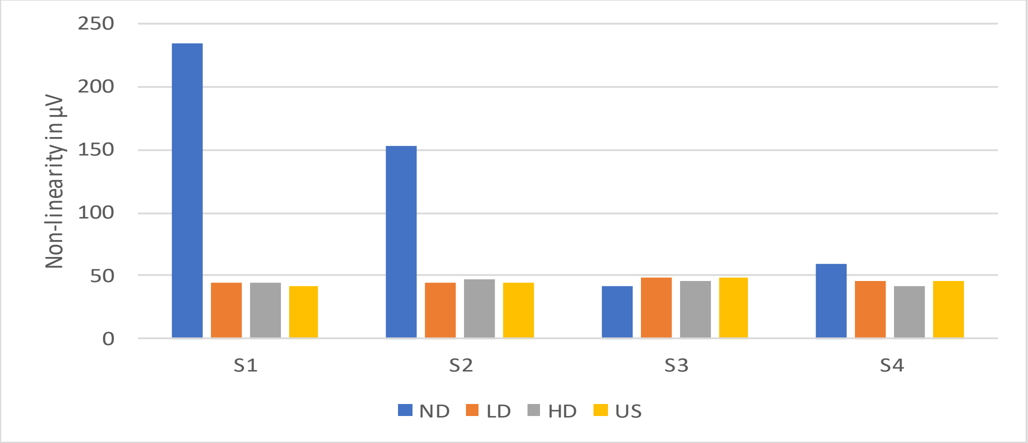


Figure 34 Permacol Cu surface finish pads.

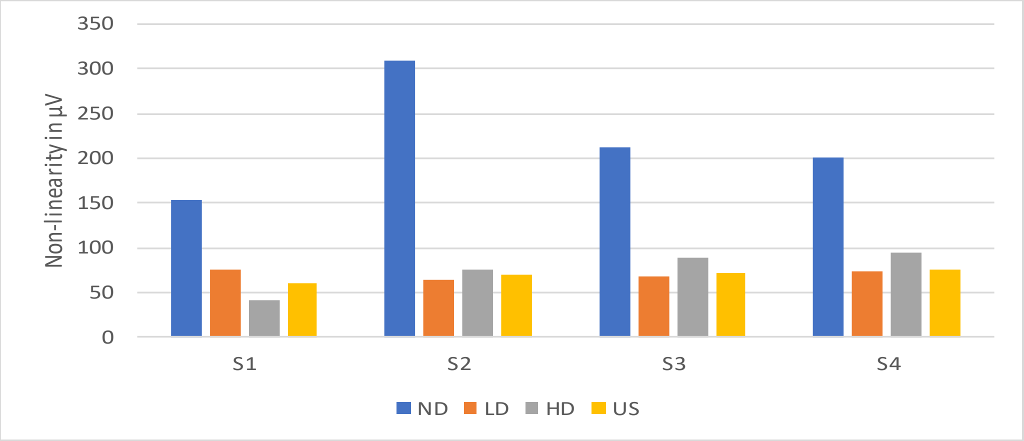


Figure 35 LOCTITE Au surface finish pads.

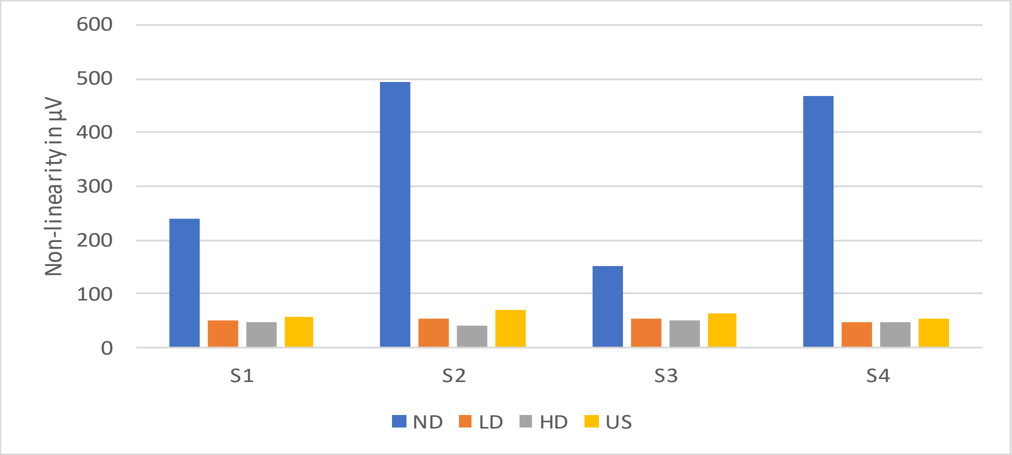


Figure 36 LOCTITE Cu surface finish pads.

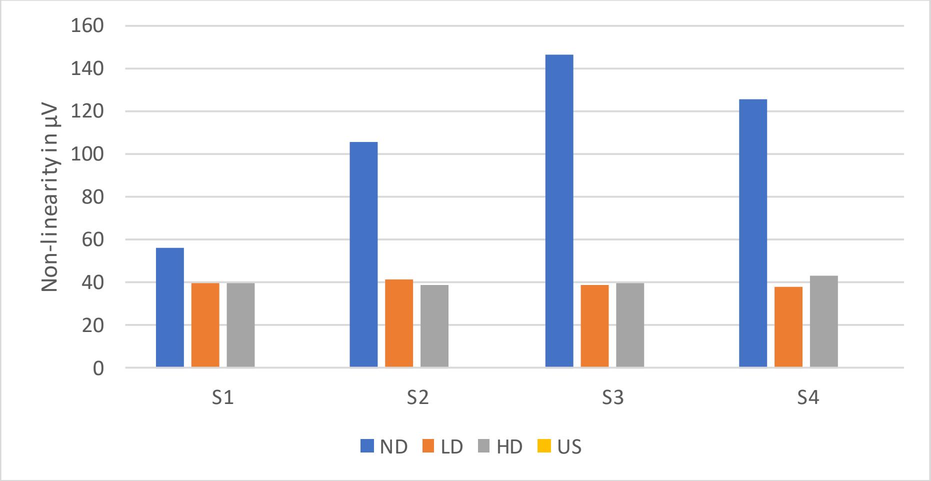


Figure 37 AX 70MN Au surface finish pads.

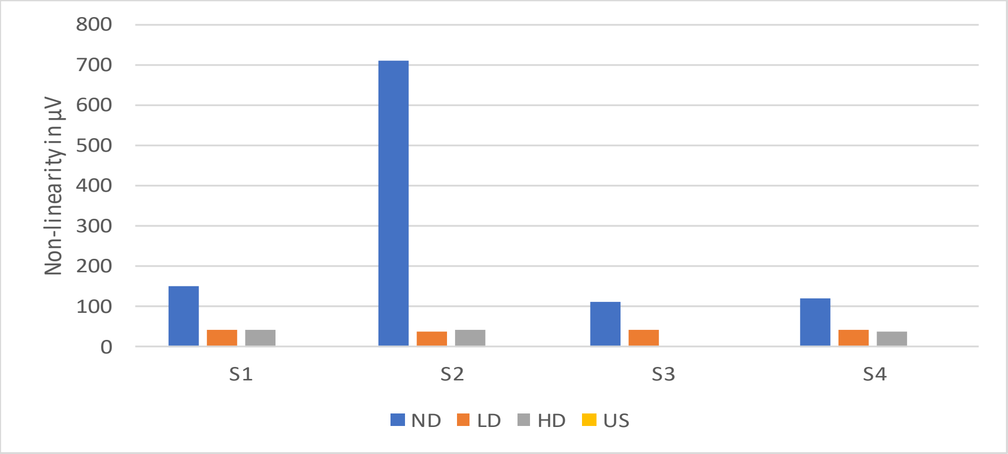


Figure 38 AX 70MN Cu surface finish pads.

As previously mentioned, the samples with adhesive AX 70MN failed the ultrasonic stress testing.

2.6 Data Analysis

In this section, we will analyze the data we gathered in the previous part. The analysis will be based upon comparison between the data before and after applied stress. Comparison will be done sequentially from each case and each adhesive under it. There were no changes observed in the no deflection (ND) scenario so we will omit that case from our evaluation.

The first scenario will be of Permacol adhesive samples, followed by LOCTITE and AX 70MN samples respectively, with Au and Cu contacts. The 'G' representing gold contact and 'C' representing copper contact. Note that to simplify our analysis, we calculated the median of all the resistances and non-linearity values obtained during experimentation and used those median values to compare.

2.6.1 Low deflection (LD) analysis

The data for Permacol before and after stress is contained in table 5.

Table 5 PERMACOL sample comparison before and after stress with median values.

	R(mΩ)Au	R(mΩ)Cu	NL(μV)Au	NL(μV)Cu
Before Stress	368.5	297	114.5	357
After Stress	235.5	287	43.95	47.55

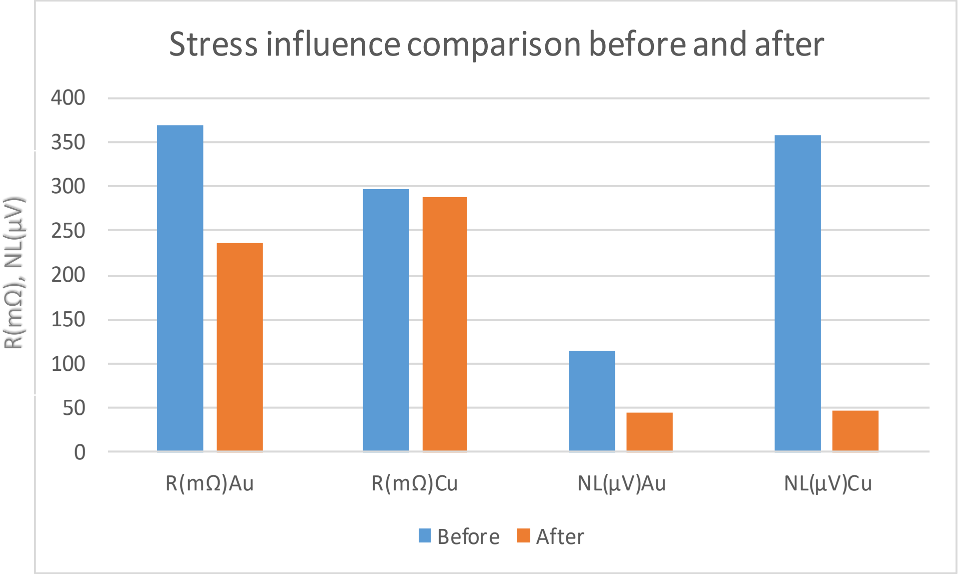


Figure 39 Stress influence, before and after state (Permacol, LD).

The data for LOCTITE before and after stress is contained in table 6.

Table 6 LOCTITE sample comparison before and after stress with median values.

	R(mΩ)Au	R(mΩ)Cu	NL(μV)Au	NL(μV)Cu
Before stress	143.5	157.5	253	238
After stress	130	245.5	70.25	52.45

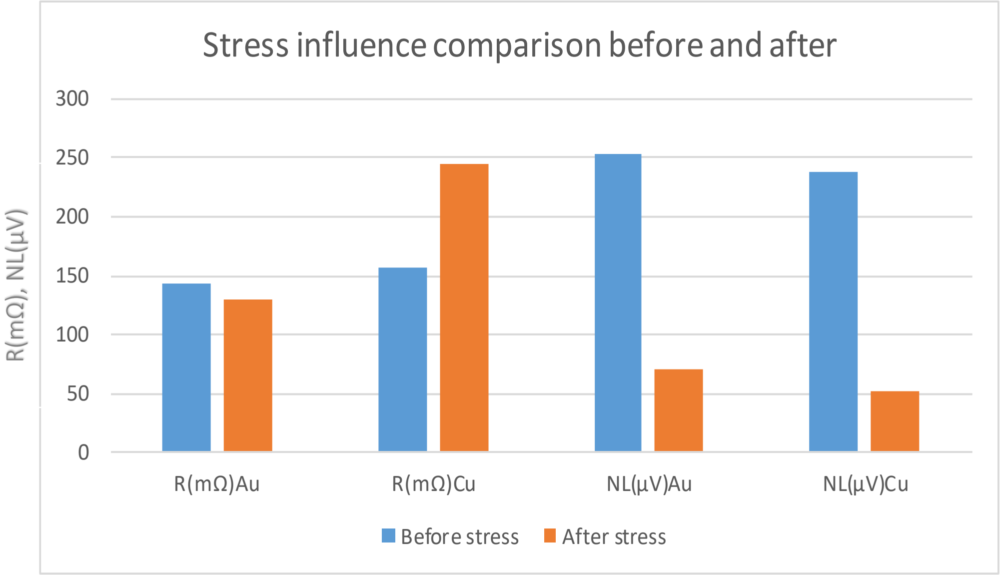


Figure 40 Stress influence, before and after state (LOCTITE, LD)

The data for AX70MN before and after stress is contained in table 7.

Table 7 AX70MN sample comparison before and after stress with median values.

	R(mΩ)Au	R(mΩ)Cu	NL(µV)Au	NL(µV)Cu
Before stress	419	705	122.5	131
After stress	742.5	886	39.65	39.7

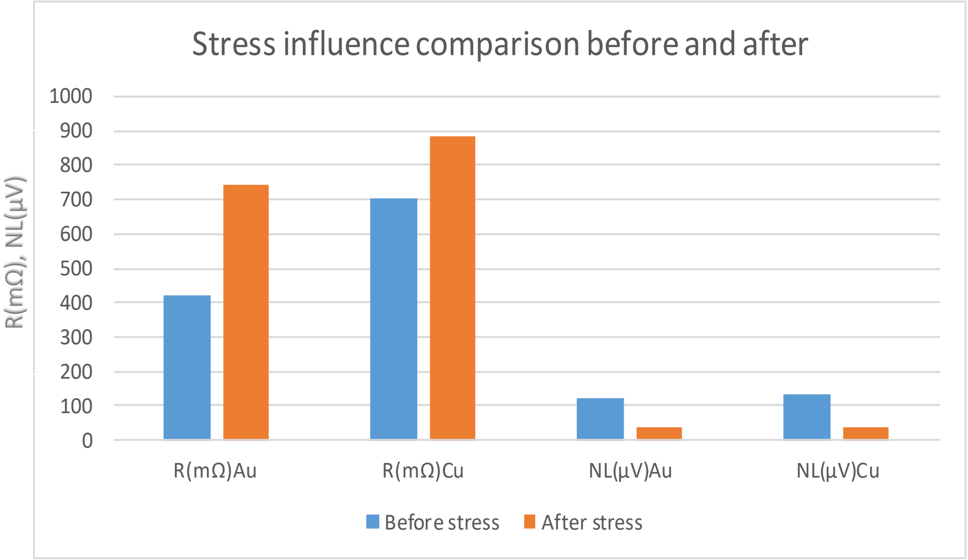


Figure 41 Stress influence, before and after state (AX70MN, LD).

2.6.2 High deflection (HD) analysis

The data for Permacol before and after stress is contained in table 7.

Table 8 Permacol sample comparison before and after stress with median values

	R(mΩ)Au	**R(mΩ)Cu**	**NL(µV)Au**	**NL(µV)Cu**
Before stress	408.5	399.5	128.5	106
After stress	165	334	49.7	44.1

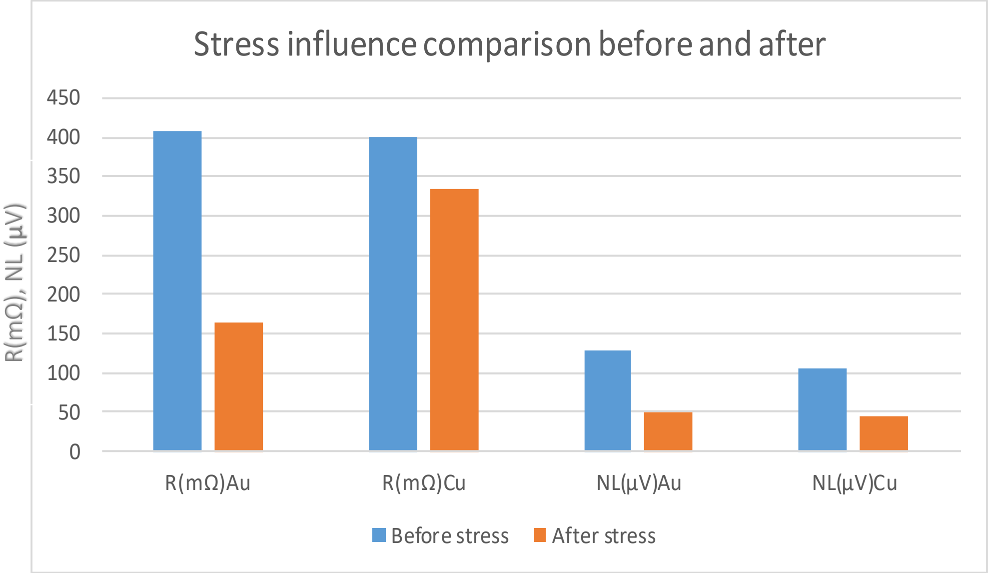


Figure 42 Stress influence, before and after state (Permacol, HD).

The data for LOCTITE before and after stress is contained in table 8.

Table 9 LOCTITE sample comparison before and after stress with median values

	R(mΩ)Au	**R(mΩ)Cu**	**NL(µV)Au**	**NL(µV)Cu**
Before stress	159	129.5	168.5	119.5
After stress	77.7	166	82.35	48.2

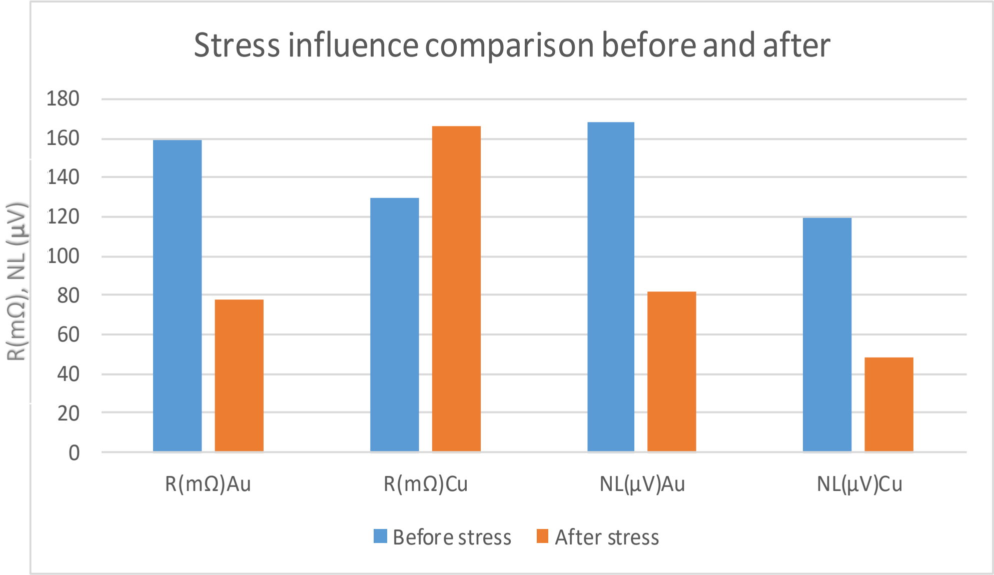


Figure 43 Stress influence, before and after state (LOCTITE, HD).

The data for AX70MN before and after stress is contained in table 10.

Table 10 AX70MN sample comparison before and after stress with median values

	R(mΩ)Au	**R(mΩ)Cu**	**NL(µV)Au**	**NL(µV)Cu**
Before stress	760	780	136.5	111.5
After stress	732.5	1110	39.85	39.6

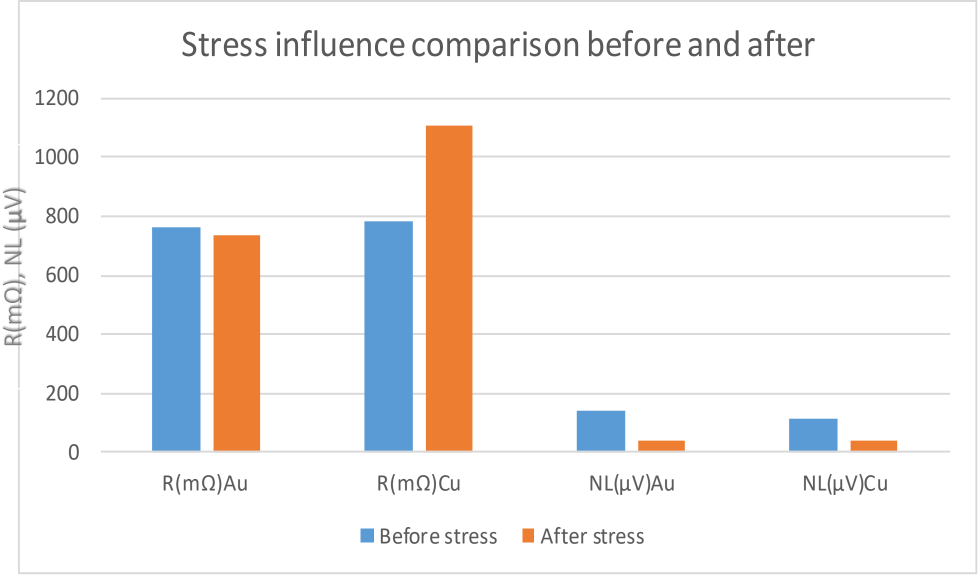


Figure 44 Stress influence, before and after state (AX70MN, HD).

2.6.3 Ultrasonic Stress (US) analysis

The data for Permacol before and after stress is contained in table 11.

Table 11 Permacol sample comparison before and after stress with median values

	R(mΩ)Au	**R(mΩ)Cu**	**NL(μV)Au**	**NL(μV)Cu**
Before stress	238.5	359	161	307
After stress	258.5	393.5	45.15	44.35

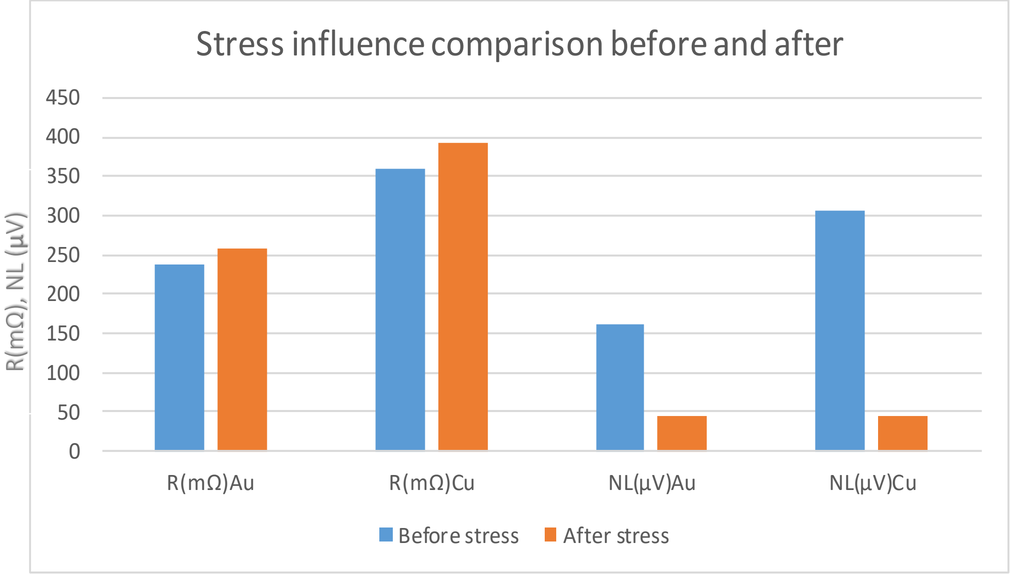


Figure 45 Stress influence, before and after state (Permacol, US)

The data for LOCTITE before and after stress is contained in table 12.

Table 12 LOCTITE sample comparison before and after stress with median values

	R(mΩ)Au	**R(mΩ)Cu**	**NL(µV)Au**	**NL(µV)Cu**
Before stress	304.5	301.5	113.5	354.5
After stress	154.5	255	70.6	60.75

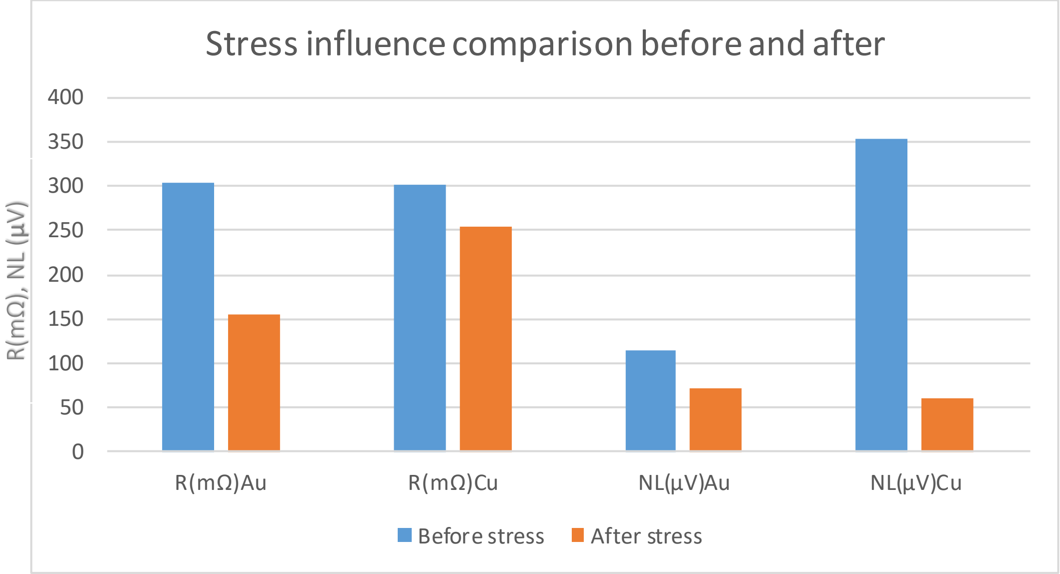


Figure 46 Stress influence, before and after state (LOCTITE, US)

3 Discussion

For discussion, our focus would be on the quality of contacts and how they are influenced with the application of stress. The stress on the joints can be categorized into three parts with the following schematic in Fig. 47:

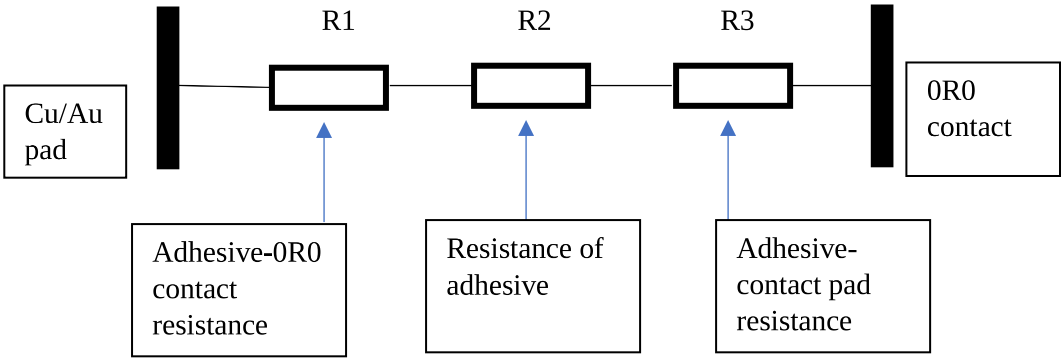


Figure 47 Schematic of resistance of an adhesive joint.

The primary governing factor for resistance in the adhesive joint is the tunneling of electrons through the oxide layer between the contacts. If we take R1 to be the adhesive-resistor contact resistance, R2 as resistance of adhesive and R3 as adhesive-contact pad resistance, the total resistance of one adhesive joint will be:

$$R_{TOTAL} = R_1 + R_2 + R_3 \qquad (1)$$

Each contact will slightly differ in its characteristics, reasons being the amount of adhesive on each contact, the area of contact with adhesive to the resistor and the area of contact with adhesive of the pad contact. These discrepancies are due to coming through the preparation of samples process. One important aspect to be considered is the sensitivity of each factor i.e. resistance and non-linearity. Resistance comprises of all mechanisms of conductivity, it means primary tunneling, but also ions migration, Schottky emission, diffusion and others, which can take place in the total conductivity of the adhesive joint, whereas non-linearity describes tunneling mechanism dominantly. Therefore, non-linearity is more sensitive to the changes in quality of the tunnel barriers between in contacts as compared to the resistance.

For our second case, where the resistance increases with stress but still the joint experiences decrease in non-linearity (e.g in Permacol C with LD, LOCTITE Cu with LD, 70MN Cu with LD, LOCTITE Cu with HD, 70MN Cu with HD and Permacol Au and Cu with US). This observation is quite evident in our samples. As mentioned earlier, there are many factors that govern the resistance but not non-linearity. This phenomenon might be explained by a situation where the flake contacts reduce, but the quality of those existing contacts increases. For example, we if we have 50 flakes in contact with each other before stress, but the connection of flakes in not of good quality. After the joint experiences stress, the flake contacts decrease, let's say now 20 flakes are in contact instead of 50. For these remaining 20 flake contacts, however these contacts are of higher quality than previous ones. This way the resistance of the joint will increase, but the non-linearity will decrease. This is also possible due to the high stress which creates cracks in the polymer structure and starts pushing the flakes away from each other.

One important factor evident from the results is the high value of non-linearity, also considered as before stress state (see Fig. 33 to 38). After application of stress it experienced a significant drop in the non-linearity. This can be possible because the measurements before stress and ND case were taken right after the curing. The curing time prescribed by the manufacturer is usually less than 80% of its full curing. Due to this, it is possible for the contacts to take time, let's say about a few hours or few days to stabilize and undergo further changes with time before it stabilizes at the room temperature and conditions. This knowledge is new and will be respected in the next stages of adhesion joints research.

The testing of influence of the ultrasound energy on quality of adhesive joints is also quite new and similar experiments were not found in the literature. It was found that the ultrasonic stress applied on joints formed of adhesive Permacol did caused small only changes of the joints resistance but caused significant decrease of nonlinearity of these joints, regardless of whether the surface finish of the pads was Cu or Au. As for the adhesive Loctite and Cu surface finish of the pads it was found approximately equal percentage decrease of the joints resistance and nonlinearity.

As for the joints of this adhesive formed on the pads with Cu surface finish, it was also found decrease of the resistance as well as nonlinearity, but the percentage decrease of nonlinearity was higher in comparison with the decrease of the resistance. It was found that the ultrasound stress influences quality of adhesive joints and can destroy the joint as well. The results found show significant influence of the surface finish of the pads on the results of the experiment.

It is necessary to add that the experimental setup for loading of the joints by the ultrasound was very simple and using more defined loading of the joints by the ultrasound the results would be more accurate. But the goal of the work was primary testing of influence of the ultrasound stress on the adhesive joints and to observe possible changes, if any. From this point of view, the experiments fully accomplished their purpose.

Conclusions

The aims of this thesis are fully studied and met. We realized the what might be the effect of the mechanical stress (low and high) on the quality of adhesive joints, making use of three different adhesives from three different manufacturers. We also explored the effect of high stress, which lead to failure of samples in high deflection and ultrasonic stress, and how it improved or degraded the quality of adhesive joints.

The part of our thesis was based on the study of ultrasonic stress on joints quality. From the results, we obtained, it was evidently clear that there is a big influence of ultrasonic stress on the quality of adhesive joints (majorly in a good way) if kept under certain conditions. I feel there is a lot of room for research in the exploration of this aspect along with the mechanical stress combined. As for future, the aspects of ultrasonic stress remain unclear as to what really causes them and how they can be defined in a pattern to use it as an advantage rather than a problem. One important aspect which can be studied in detail is the ultrasonic stress combined with deflection. To add more variable, element of hot surrounding can also be added to the study later to continue further. There are a lot of aspects which we are not able to clearly define, e.g. the level of stress which is good for the joint. A lot of progress can be made in continuing this study to further advanced levels.

The changes caused in adhesive joints by the mechanical or ultrasound stress have been evaluated by measurement of the joints resistance and nonlinearity of their current-voltage characteristic. Both these parameters seem to be good for inspections. The joint resistance is the most significant electric parameter of the joint, nonlinearity is more sensitive to changes in the structure of the adhesive cause by the load. Study of nonlinearity of the current-voltage characteristic is important not only as an indicator to changes in adhesive or contacts between adhesive and pad or contact of a component, but inspection of nonlinearity is also significant for high-frequency applications, where it can cause cross-modulation and deteriorate quality of the signal. The next parameter, which would complete the measurement of nonlinearity is joint noise. Measurement of noise is directed to next research in the field.

List of Figures

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Appendix 1

Resistance

Permacol	ND	LD	HD	US
Au	303.5	235.5	165	258.5
Cu	338.5	297	334	393.5

Median resistances for samples prepared with Permacol adhesive (Au, Cu)

LOCTITE	ND	LD	HD	US
Au	135.5	130	77.7	154.5
Cu	148.5	245.5	166	255

Median resistances for samples prepared with LOCTITE adhesive (Au, Cu)

AX70MN	ND	LD	HD	US
Au	620	742.5	732.5	-
Cu	693.5	886	1120	-

Median resistances for samples prepared with AX70MN adhesive (Au, Cu)

Appendix 2

Non-Linearity

Au	ND	LD	HD	US
S1	165	43.1	46.2	43.1
S2	79	50.1	49.4	50.1
S3	97	46.7	52.8	46.7
S4	160	42.2	50	43.6

Non-Linearity of samples prepared with Permacol adhesive (Au)

Cu	**ND**	**LD**	**HD**	**US**
S1	235	43.8	43.7	42.2
S2	153	43.6	46.6	43.8
S3	41	48.8	44.5	48.8
S4	59	44.9	41.8	44.9

Non-Linearity of samples prepared with Permacol adhesive (Cu)

Au	**ND**	**LD**	**HD**	**US**
S1	154	75.3	40.8	60.1
S2	309	63.1	76.5	69.4
S3	212	66.8	88.2	71.8
S4	201	73.7	94.3	75.2

Non-Linearity of samples prepared with LOCTITE adhesive (Au)

Cu	**ND**	**LD**	**HD**	**US**
S1	239	51.4	47.8	57.3
S2	495	53.6	41.1	69.6
S3	151	53.5	51.6	64.2
S4	470	48.9	48.6	55.3

Non-Linearity of samples prepared with LOCTITE adhesive (Cu)

Au	**ND**	**LD**	**HD**	**US**
S1	56	39.8	39.8	-
S2	106	40.8	39.7	-
S3	147	39.5	39.9	-
S4	126	39.3	42.4	-

Non-Linearity of samples prepared with AX70MN adhesive (Au)

Cu	**ND**	**LD**	**HD**	**US**
S1	150	39.6	39.6	-
S2	711	39.4	40.1	-
S3	112	39.8	-	-
S4	120	40.1	39.3	-

Non-Linearity of samples prepared with AX70MN adhesive (Cu)

As previously mentioned, the samples with adhesive AX 70MN failed the ultrasonic stress testing.