

CZECH TECHNICAL UNIVERSITY IN PRAGUE



DOCTORAL THESIS STATEMENT

Czech Technical University in Prague

Faculty of Electrical Engineering

Department of Electrotechnology

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**STUDY OF MODERN MATERIALS OF ELECTRICALLY CONDUCTIVE JOINTS IN
ELECTRONICS**

Ph.D. Programme: Electrical Engineering and Information Technology

Electro-Technology and Materials

**Doctoral thesis statement for obtaining the academic title of “Doctor”,
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The defence of the doctoral thesis will be held on at a.m./p.m. before the Board for the Defence of the Doctoral Thesis in the branch of study Electro-Technology and Materials in the meeting room No. of the Faculty of Electrical Engineering of the CTU in Prague.

Those interested may get acquainted with the doctoral thesis concerned at the Dean Office of the Faculty of Electrical Engineering of the CTU in Prague, at the Department for Science and Research, Technická 2, Praha 6.

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1. CURRENT SITUATION OF THE STUDIED PROBLEM
2. AIMS OF THE DOCTORAL THESIS
3. WORKING METHODS
4. RESULTS
5. CONCLUSION

List of literature used in the thesis statement

List of candidate's works relating to the doctoral thesis

Response / No response and reviews

SUMMARY

RÉSUMÉ

1. CURRENT SITUATION OF THE STUDIED PROBLEM

There are high requirements for an electrical joint quality. The following three main electrical parameters must be observed and they must be as low as possible: the resistance, nonlinearity of voltage-current characteristic and current noise. These parameters influence the quality of transmitted signal, especially if the signal is low or if the transmitted frequency is high.

The most common method for component connection on printed circuit boards is, already for several decades, soldering. With new materials, often based on organics, some other types of connection are necessary and the evolution showed that one way to go is the use of electrically conductive adhesives.

Soldering is the major technique for creating joints, unfortunately, soldering has some limitations and restrictions, especially after the European ROHS directive came into effect on 1st July 2006 [1]. This directive restricts the use of so called hazardous substances, like lead, cadmium, mercury etc. As lead was widely used in solders (Tin-lead solders (SnPb)), new materials had to be invented. The most used replacement for SnPb is SAC, formed from Sn (stannum), Ag (silver) and Cu (copper). The complication with this solder is its higher melting temperature, while the eutectic SnPb solder has melting temperature of 183 °C, the nowadays mostly used SAC has melting point of 217 °C. The real peak solder temperature in electronic manufacturing is therefore approximately 240 instead of 220 °C [2]. Not all components are that durable, especially organic materials like liquid crystal displays cannot withstand common soldering temperatures [3].

Beside lower curing temperature, electrically conductive adhesives allow the use of fine pitch components without the worries about short circuit bridges. The driving force for electrically conductive adhesives therefore is the requirement of increasing miniaturization.

This can be achieved by the use of anisotropic conductive adhesives (ACAs), where the filler particles are not in a mutual contact and the conductive path is realized in z-axis only.

2. AIMS OF THE DOCTORAL THESIS

Electrically conductive adhesives are an ecological alternative and studying their properties and parameters is therefore vital.

The aim of this work is a summarization of electrical connection methods with a special focus on electrically conductive adhesives, their basic diversification, advantages and disadvantages. The adhesives were also modified using different additives and their electrical and mechanical properties before and after several types of accelerated aging were measured.

Specifically, the goals are:

- to find suitability of C-V nonlinearity measurement as a possible way to judge joint quality, and also to compare it with standard resistance measurement
- to find suitable modification to lower electrical resistance of ECA
- to estimate the influence of ECA modifications, particularly modification by silver nanoparticles and multi-walled carbon nanotubes onto electrical and mechanical properties
- to estimate the effect of ECA modifications onto glass transition temperature of the adhesive

- to reveal the effect of different climatic aging on the glass transition temperature of ECA

3. WORKING METHODS

The first presented method is the incorporation of nanoparticles as additional filler. Due to their properties, a chance exists, that short time annealing will cause sintering which is accompanied by rapid resistance decrease.

Nanomaterials as Possible ECA Components

Nanomaterials exhibit some remarkable specific properties that may be significantly different from the physical properties of bulk materials.

Many materials, including pure metals, exhibit a change in properties as their particle sizes approach nanoscale dimensions. The increase in the surface-to-volume ratio, which occurs naturally as particle sizes shrink, necessarily increases the relative proportion of higher energy surface atoms. The effect may include a change in reactivity, such as in sinterability, the agglomeration of metal particles by heating. It may also appear as a change in electromagnetic properties, altering electronic or optical properties.

Nowadays, nanoparticles are employed because of their remarkably lower melting temperature compared to bulk materials. This low melting temperature of nanoparticles is due to the large ratio of surface atoms to inner atoms. For example, a gold particle of 4 nm diameter has 25 % of the atoms on the surface. This high portion of surface atoms drastically decreases the melting temperature, since smaller particles exhibit reduced interaction between the surface and the inner atoms; i.e., the surface energy of the surface atoms is reduced. To exemplify, the melting point for gold nanoparticles in the range of a few nanometers lies approximately between 300 – 400 °C versus 1064 °C for bulk gold. This behavior is depicted in the following graph. The melting temperature depression with particle size reduction is obvious. The theoretical progression has so called “tipping point”. The particle size where these changes occur depends on both the individual element or compound and its environment. The particle diameters generally needs to be somewhere below 100 nm to achieve the tipping point, where significant property changes occur. The tipping point shows as an abrupt shift in the slope of the measured curve (figure 1 on the left) of sintering temperature as a function of the size of the particle.

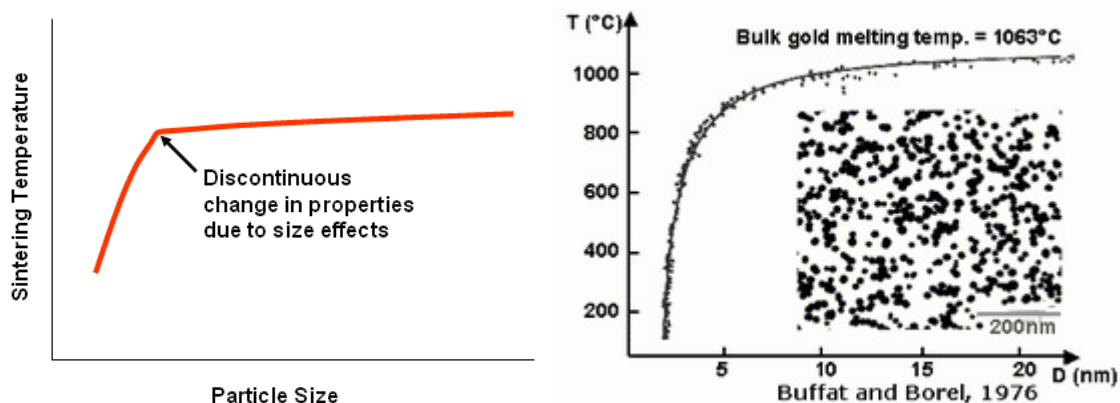


Fig. 1: Tipping point [4] and melting temperature in dependence on particle size [5]

The research by Ko et al. [5] proved that the sintering temperature for materials containing nanoparticles with an approximate diameter of 5 nm can be around 150 °C. This is a temperature that is far below the melting point of commonly used solders.

Woo-Ju Jeong et al. [6] published a work describing theoretical improvement principle after adding nanoparticles to the microparticle filled adhesive. The principle lies in change of the contact area. With microparticles only, the contact area is very small and with a decent addition of nanoparticles, this area may enlarge as depicted in figure below.

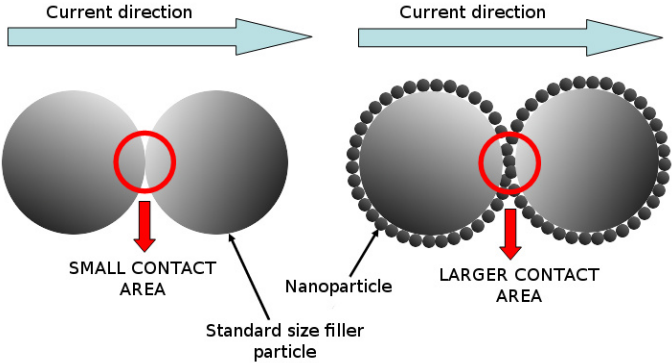


Fig. 2: Nanoparticles may enlarge the contact area

Strong Electrolytes Addition

Strong electrolytes are in solutions of any concentration fully dissociated to ions. These are nearly all salts of inorganic and organic acids, many hydroxides and strong acids. Electrolytic dissociation of ion crystal is schematically depicted in figure below.

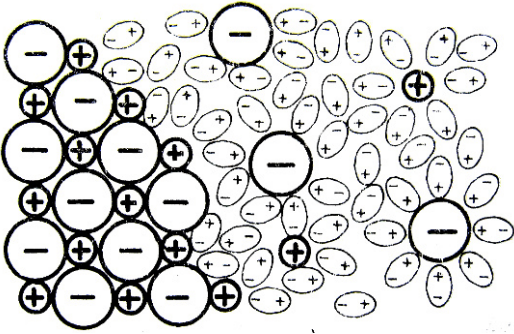


Fig. 3: Electrolytic dissociation of ion crystal in polar solvent

For this work strong electrolytes were incorporated to the adhesive. Principle of this method is based on high dissociation potential of strong electrolytes to ions due to polar solvent - in our case the Butylglycidylether - to ions. These ions subsequently increase the conductivity of the ECA. Conductive paths are created due to the presence of ions that originated by strong electrolyte dissociation

For an evaluation of joint quality, many methods are used. The most used is the evaluation of electrical resistance. It is advisable, that the modified adhesive undergo some type of stress, for example HAST (Highly Accelerated Stress Test) These tests can include the impact of temperature, humidity, etc.

Joint Resistance Measurement

Electrical resistance can be measured in different arrangements. There is the arrangement for high resistance measurement or for small resistance measurement. As our sample was a daisy chain of seven resistors with zero nominal resistance attached using electrically conductive adhesive, the arrangement for small resistances measurement with four-point probe was chosen.

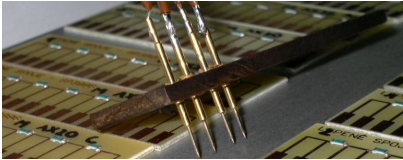


Fig. 4: measuring head for 4-point measurement

Measurement by Thermal Field Evaluation

Contactless methods of resistance evaluation based on the analysis of heat generated within a bonded joint. The resistance of the joint can be discovered by evaluating of temperature field in its proximity.



Fig. 5: Thermal field evaluation, heat propagation model

The accuracy of approximately 10 % can be achieved.

Current-Voltage Nonlinearity Measurement

The principle is to asses non-homogenities and non-stable barriers in an electronic circuit, as these could after some prolonged period of time lead to a circuit or component parameter change and circuit failure. The use of this method is also suitable for quality evaluation of electrically conductive adhesives as the non-linearity change is more distinct when compared to resistance change. [7]

Nonlinearity measurement of nominally linear electronic elements is however fairly difficult due to very low observed nonlinearity value.

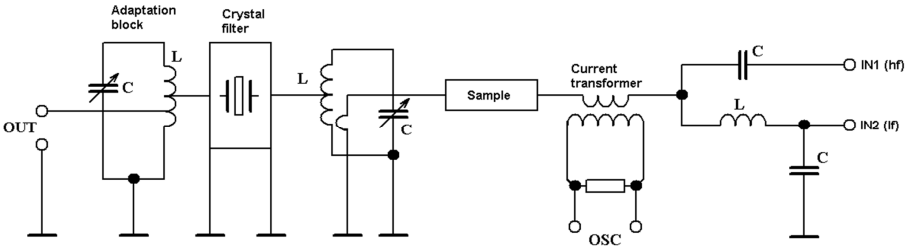


Fig. 6: Schematic circuit for current-voltage nonlinearity measurement using intermodulation distortion

Nonlinearity was evaluated on a spectrum analyzer, the value was given in dBm (decibel per milliwatt) therefore it was necessary to recalculate the value to voltage.

Mechanical Testing of ECAs

- pull-off test (perpendicular direction)
- shear strength test (parallel direction)

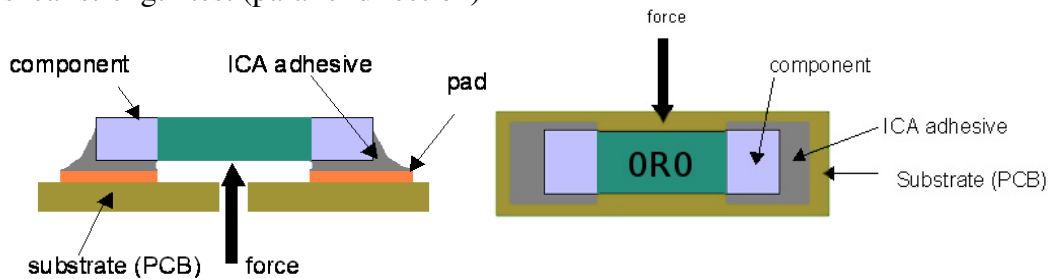


Fig. 7: Schematics of the pull off (left) and shear strength test

Structural Analysis Methods

- based on weight evaluation (T_g , G_c , GPC)
- based on energy measurement (DTA, DSC)
- Methods based on mechanical parameter measurement (TMA, DMA)

For this work, methods based on mechanical parameter measurement were chosen, specifically DMA

Dynamic Mechanical Analysis, shortly DMA, is a technique to test response of a material sample to the application of small cyclic deformation. The response to temperature, frequency and other values can be studied. The apparatus on which this test is conducted is also referred to as DMA.

Dynamic mechanical analysis (DMA) is considered one of the most accurate techniques for glass transition temperature (T_g) measurement. [8] DMA is a technique in which the deformation of a sample under oscillatory load is measured as a function of temperature. The method is capable of characterization and interpretation of the mechanical behavior of the observed sample. In short, it consists of the application of oscillating force to a sample and observation of the material response to that force. This method is particularly suitable for observing properties of polymer materials. One of the main advantages is the suitability to quickly determine polymer properties as a function of frequency, temperature or time with the use of only a very small material sample.

Glass Transition Temperature T_g Measurement

It is commonly known that substances or clusters of molecules can exist in three states of matter: solid, liquid or gas. However, if it will be a polymeric material, this division is not that simple. In other words, the material is an amorphous solid, or a glass.

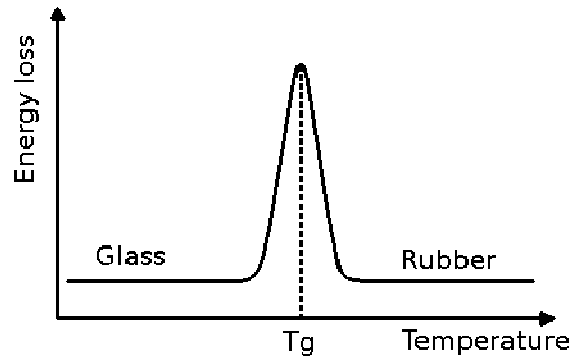


Fig. 8: Basic understanding of T_g

Dynamic Mechanical Analysis

Dynamic Mechanical Analysis (DMA), as stated earlier, can examine material response to harmonic cyclic stress as a function of time, temperature and frequency.

The DMA supplies a tiny oscillatory force (see figure 9, black curve), causing a sinusoidal strain to be applied to the sample, which generates a sinusoidal strain. By measuring both the amplitude of the deformation at the peak of the sine wave and the lag between the stress and strain sine waves, quantities like the modulus, the viscosity, and damping can be calculated.

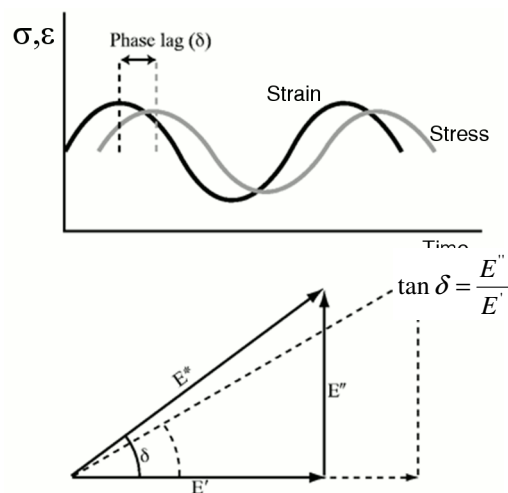


Fig. 9: Dynamic analysis of mechanical behavior, vector relationship between dynamic material properties

Type of DMA modes used in thesis:

There are many modes for DMA measurement. One of them is a three point bending that was used in this work for ECA cubicle measurement, the other is called DMA in penetration mode and was used for comprehensive comparison of ECA cylinders prepared on glass (see figure below for schematic layout and real specimen design).

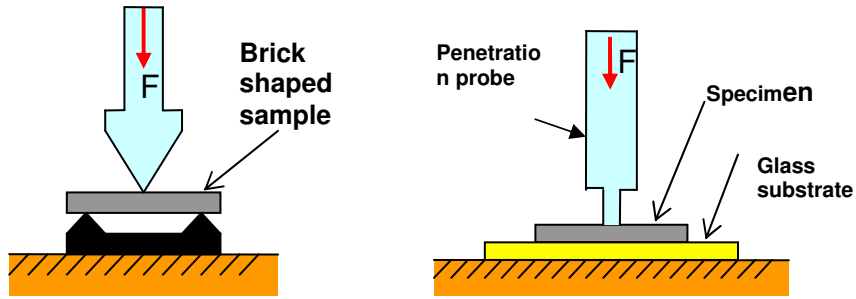


Fig. 10: Probes for DMA analysis, three point probe (left) and penetration probe (right)

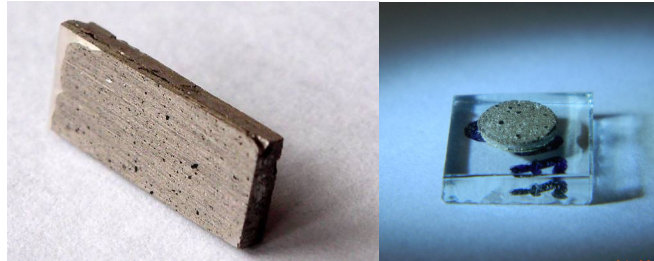


Fig. 11: Sample for three point probe measurement (left) and sample for penetration probe (right)

Interpretation of DMA diagram:

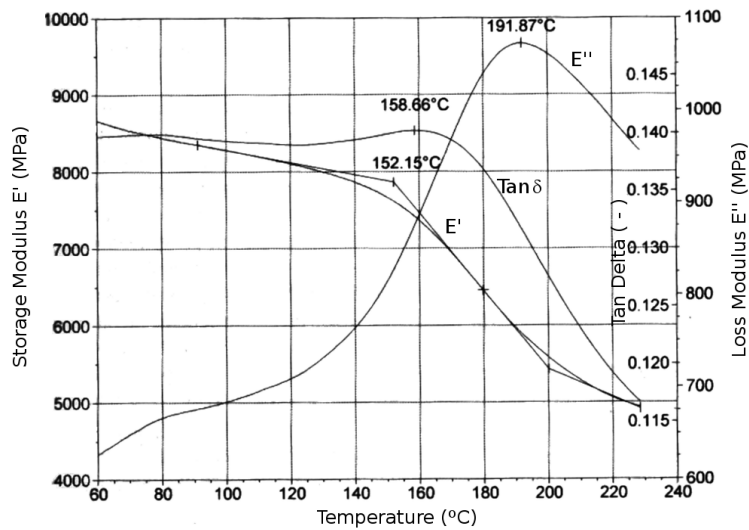


Fig. 12: DMA progression for insulating three-component composite material (mica, glass fiber, epoxy resin)

Individual modules of Storage modulus E' , Loss modulus E'' and Loss factor $\text{Tan } \delta$ are plotted as a function of temperature during the measurement.

Each measured parameter exhibits different glass transition temperature T_g and each of these three temperatures has different physical ground. The curve deflection of the storage modulus E' (also known as elastic module) from its baseline defines the temperature, at which the material begins to lose its strength, that means that the material is no longer able to withstand the load without apparent deformation being observed.

The maximum in the progression of loss modulus E'' represents the temperature at which the polymeric material undergoes the maximum change in the mobility of polymer chains.

Maximum peak in the dissipation factor ($\tan \delta$) characterizes the damping properties of materials. The most distinct peak of $\tan \delta$ occurs with amorphous polymers, which have in comparison to polycrystalline polymers lower orderliness. [9]

Peak given by $\tan \delta$ was also the first to be measured in DMA historically and therefore T_g given in literature comes mostly from $\tan \delta$ evaluation. [10]

4. RESULTS

Nonlinearity vs. Resistance Measurement

C-V nonlinearity measurement can be used as a more sensitive alternative to simple resistance measurement.

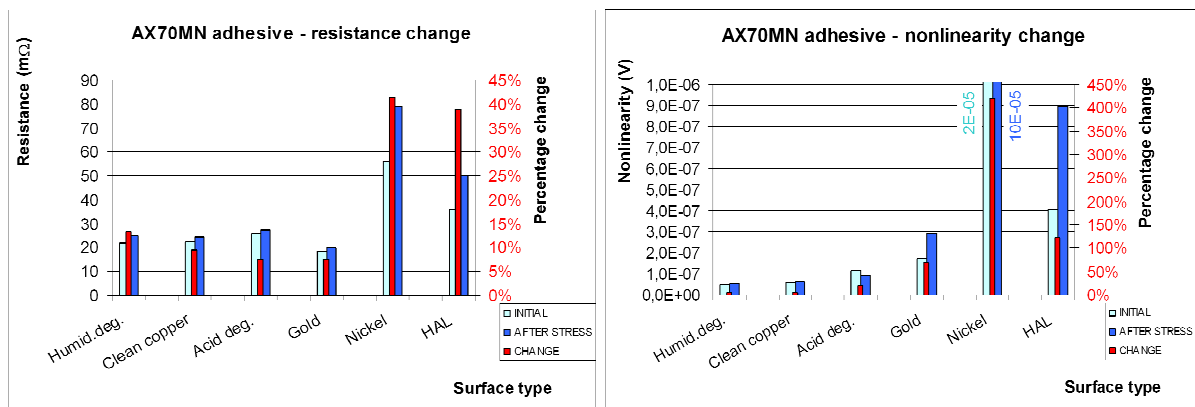


Fig. 13: Comparison of resistance (left) and C-V nonlinearity (right) measurement of ECA joint on different substrate finish.

Incorporation of silver nanoparticles to the ECA lowered resulting resistance:

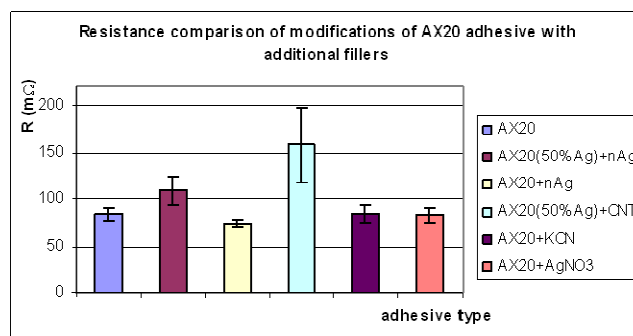


Fig. 14: ECA with nanosilver addition exhibit lowest resistance

Narbon nanotubes dispersed in ECA may increase shear strength:

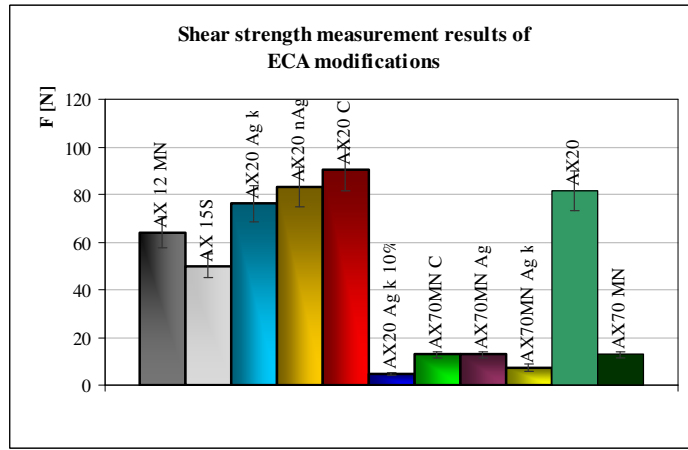


Fig. 15: ECA with carbon nanotube addition exhibits highest shear strength

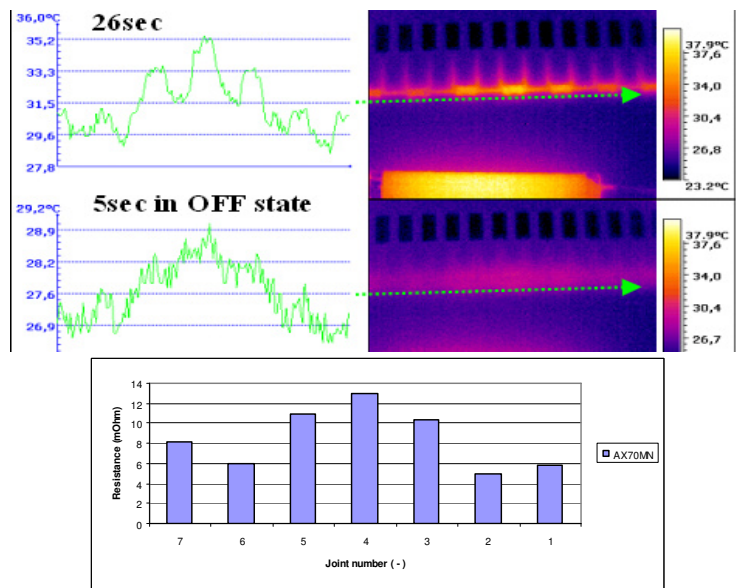


Fig. 16: Accuracy of resistance measurement was $\pm 10\%$

DMA Measurement, Tg Evaluation

DMA measurement of the cubicle proved to be unsuitable, high volume of cured adhesive caused excessive void formation in the matrix. For the final part of thermo-mechanical experiments, new types of samples were prepared (cylinder on glass substrate). Our motivation was to create conditions similar to real applications. In comparison with rectangular block samples, the amount of the adhesive was much lower and the samples were prepared to be able to fit into the DMA testing device, where the maximum proportions of the sample were limited to 10 x 10 mm.

Tg was obtained from relaxation transition peak on Tan δ . Measurement was conducted in two passes, first heating “formed” the sample and helped to finish curing processes. During the second heating the peak on tan δ corresponds to real (fully cured ECA) glass transition temperature.

Two component adhesives proved to have the peak outside of the measuring range (if at all),

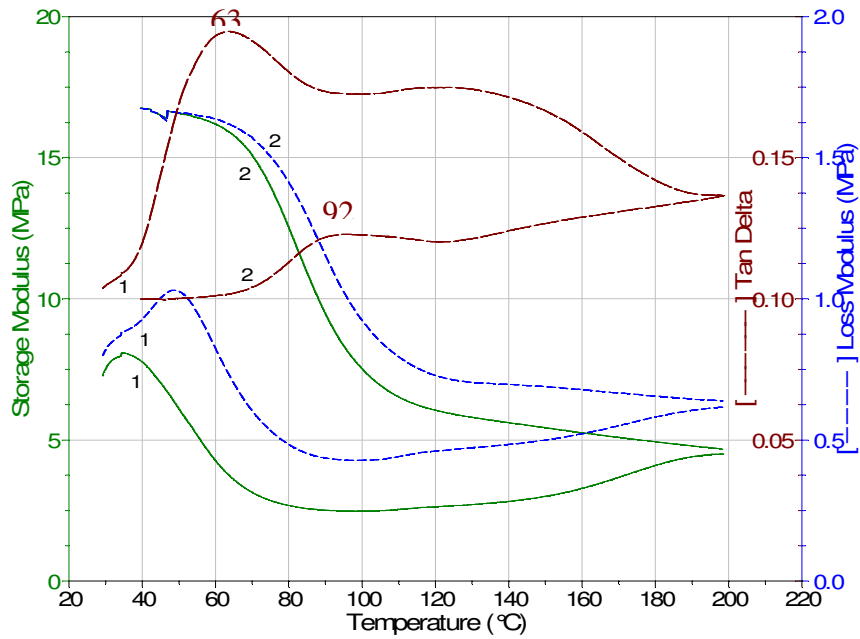


Fig. 17: Single component AX20 exhibited recognizable peaks (in contrast to two component adhesives, below)

Addition of silver nanoparticles increases maximum achievable Tg, highest glass transition temperatures were observed with samples which were subjected to heat aging.

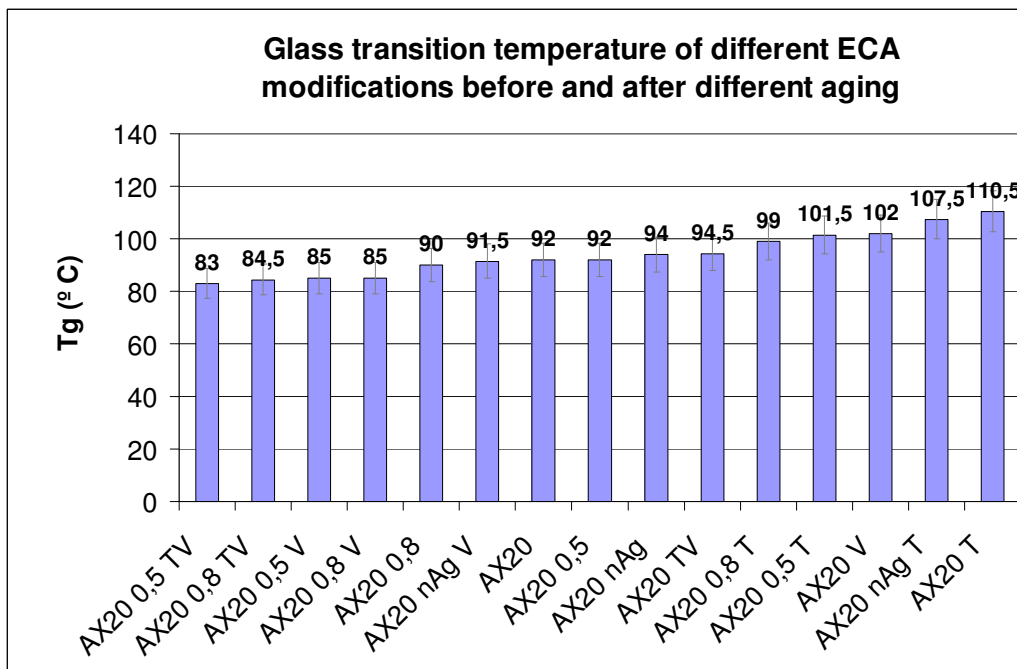


Fig. 18: Glass transition temperature in dependence on adhesive modification

5. CONCLUSION

The main goal of this work was to study modern materials for electrically conductive joint creation, with the focus on isotropically conductive adhesives and their modifications using nano-materials.

C-V nonlinearity measurement is more sensitive alternative to simple resistance measurement.

Thermal field evaluation as a joint quality determination method was possible with 10 % accuracy.

The addition of silver nanoparticles in order to increase conductivity proved to be suitable choice.

A fully unexplored research area was the nanoparticle modification influence on glass transition temperature of an ECA.

The glass transition temperature T_g may explain different behavior between a single component ECA with low T_g temperature and two-component ECA, where the T_g lies above the measured area.

Incorporation of both nanoparticles and carbon nanotubes decreased the glass transient temperature of the ECA.

The author of this work believes that the goals stated in the beginning of the thesis were fulfilled.

List of literature used in the thesis statement

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List of candidate's works relating to the doctoral thesis

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RESUMÉ:

Elektricky vodivý spoj je nejvíce frekventovaným prvkem v elektrických zařízeních, studium jeho vlastností je tedy velmi důležité.

Elektricky vodivá lepidla jsou ekologickou alternativou k pájkám – neobsahují olovo a jejich vytvrzovací teplota je nižší, než teplota pájení. Jejich výhodou je i to, že se jedná o bezoplachovou technologii.

Cílem této práce je shrnutí metod vytváření elektricky vodivých spojů s použitím elektricky vodivých lepidel (ECA). Je uvedeno jejich rozdělení, základní principy elektrické vodivosti, možnosti použití, výhody a nevýhody. Konkrétně byla posuzována lepidla s isotropní elektrickou vodivostí (ICA).

Elektricky vodivá lepidla (ICA), v jejichž matrici jsou standardně zastoupeny stříbrné částice velikosti 5-10 μm , byla modifikována přidáním nanočástic stříbra ($\sim 100 \text{ nm}$), uhlíkovými nanotrubičkami, a jinými látkami (silnými elektrolyty). Vlastnosti takto upravených lepidel byly sledovány v závislosti na aplikovaném stárnutí, dále vzhledem k čistotě a druhu použitého substrátu, atd. a byly porovnány s vlastnostmi lepidel bez úpravy.

Konkrétními cíli bylo

- Zjištění vhodnosti měření nelinearity voltampérové charakteristiky jako způsobu posuzování kvality provedeného elektrického vodivého spoje ve srovnání s měřením elektrického odporu.
- Pokusit se zjistit, způsob úpravy ECA k dosažení nižší hodnoty velikosti odporu realizovaného spoje
- Posoudit vliv úpravy ECA přidáním nanočástic stříbra a přidání mnohostěnných uhlíkových nanotrubic na elektrické a mechanické vlastnosti elektricky vodivých lepidel
- Posoudit vliv provedené modifikace a typu stárnutí na teplotu skelného přechodu daného ECA

Provedenými měřeními byla ověřena možnost posuzování spojů realizovaných elektricky vodivými lepidly pomocí měření nelinearity metodou založenou na vyhodnocení inrermodulačního zkreslení. Tato metoda se ukázala být adekvátní alternativou k měření odporu spoje. Jedná se sice o metodu složitější, zato však velmi citlivou a její výsledky tak mohou poskytnout o daném spoji komplexnější informaci.

Experimentálně byla též ověřena možnost posouzení kvality elektricky vodivého spoje vyhodnocením teplotního pole v okolí daného spoje. Srovnáním se čtyřbodovou metodou měření elektrického odporu bylo dosaženo přesnosti přibližně 10%.

Přidání nanočástic stříbra do ICA se ukázalo jako výhodné. Bylo dosaženo zlepšení elektrického odporu při současném zachování ostatních vyhodnocovaných parametrů.

Dosud neprobádanou oblastí bylo zjišťování vlivu příměsí nanočástic na teplotu skelného přechodu ECA. Posuzování teploty skelného přechodu může umožnit pochopení rozdílného chování jednotlivých druhů ECA, například rozdíly mezi jedno- a dvousložkovými ECA lepidly v závislosti na jejich stárnutí. Bylo zjištěno snížení teploty skelného přechodu jak po přidání nanočástic stříbra, tak po přidání uhlíkových nanotrubic.