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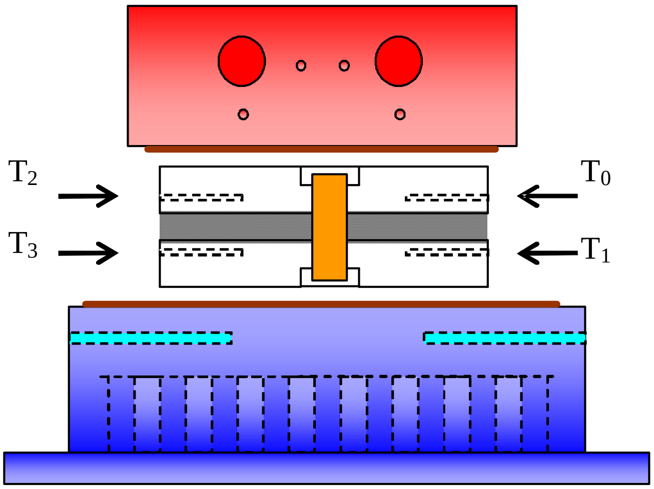

Risto Hienonen, Jari Keskinen &
Timo Koivuluoma

Reliability of materials for the thermal management of electronics

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Keywords thermal interface material, electronics, measurements, thermal impedance, thermal conductivity, phase change materials, reliability, environmental testing

Abstract

The main goal of this project was to research the properties and reliability of the thermal interface materials used in electronics. The selected materials for measurements and tests contained elastic pad sheets, phase change materials and greases. The thermal impedance at pressures of 0,1...0,8 MPa was measured from the 34 materials. From these 14 materials were selected for the reliability tests.

A new measurement method for thermal impedance and a new type of test structure for material testing was developed. The new test structure for materials was designed for use in environmental tests. The test structure makes it possible to select test surface quality, test pressure and material thickness.

The materials were tested in various environmental conditions (high/low temperature, temperature/humidity cycle, temperature cycling) to see how their properties change in these conditions and what is the life endurance in use.

The main criteria for evaluation of materials was the measured thermal impedance ($K \cdot cm^2/W$) at power densities from 10 to 25 W/cm^2. The high power density means that samples are heated during the measurements into the high operating temperature. Greases, phase change pads and graphite pads had the lowest impedance value.

The measured thermal impedance showed that the pressure and the wettability of materials have in many cases a larger effect on the impedance than the thermal conductivity of the bulk material. Therefore the impedance should be measured with the low pressures (< 0,3 MPa), which are achievable in practical solutions.

The tested materials (14 types) performed quite well in the reliability tests, although there were some samples, which had quite large changes of impedance values. The long term behaviour of the tested materials seems to be good.

Quite important observation of the tests is that the variation from sample to sample in initial and final measurements is larger than the changes during the tests. Therefore the user should take care of the surface quality and contact pressure while calculating the total variation of thermal impedance for the whole life cycle of products.

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Avainsanat thermal interface material, electronics, measurements, thermal impedance, thermal conductivity, phase change materials, reliability, environmental testing

Tiivistelmä

Projektin tavoitteena oli tutkia elektroniikassa käytettyjen lämpöä johtavien materiaalien ominaisuuksia ja luotettavuutta. Tutkitut materiaalit olivat joustavia levyjä, faasimuutokseen pohjautuvia kalvoja ja rasvoja. Hankkeessa mitattiin 34 erityyppisen materiaalin lämpöimpedanssi pintapaineella 0,1...0,8 MPa. Näiden mittausten perusteella valittiin 14 materiaalityyppiä luotettavuustesteihin.

Materiaalien olosuhdetestejä varten kehitettiin uusi lämpöimpedanssin mittausmenetelmä ja siinä käytettävä testirakenne. Testirakenne mahdollistaa pinnan laadun, pintapaineen ja materiaalin paksuuden valinnan kulloisenkin tarpeen mukaan.

Materiaaleja testattiin useissa ympäristöolosuhteissa: korkea ja matala lämpötila, vaihteleva lämpö ja kosteus sekä pitkäaikainen lämpötilan vaihtelu. Tavoitteena oli saada tietoa materiaalien ominaisuuksien muutoksista ja pitkäaikaisesta stabiilisuudesta käyttöolosuhteita vastaavissa rasituksissa.

Lämpöimpedanssia käytettiin pääkriteerinä arvioitaessa materiaalien ominaisuuksia. Lämpöimpedanssi ($K \cdot cm^2/W$) mitattiin tehotiheyksillä 10…25 W/cm^2. Pienimmät lämpöimpedanssin arvot oli rasvoilla, faasimuutosmateriaaleilla ja grafiittikalvoilla.

Mittausten perusteella pintapaine ja materiaalien kyky mukautua asennuspintaan vaikuttivat useissa tapauksissa lämpöimpedanssiin enemmän kuin materiaalin lämmönjohtavuus. Tämän vuoksi lämpöimpedanssi tulisi mitata riittävän alhaisella paineella (< 0,3 MPa), joka on saavutettavissa käytännön jäähdytysratkaisuissa.

Luotettavuustesteissä olleet materiaalit (14 tyyppiä) toimivat hyvin testeissä, vaikkakin muutamilla näytteillä oli melko suuri lämpöimpedanssin muutos. Materiaalien stabiilisuus pitkäaikaisessa lämpövaihtelussa oli hyvä.

Materiaalien lämpöimpedanssin vaihtelu näytteestä toiseen oli saman materiaalin sisällä suurempi kuin testien aikana tapahtuneet muutokset. Tämä korostaa materiaalien huolellisen asennuksen, pintojen laadun ja pintapaineen hallinnan tärkeyttä arvioitaessa lämpöimpedanssin vaihtelua tuotteen koko elinaikana.

Preface

The idea for this project “Reliability of materials for thermal management of electronics” developed in the co-operation group of thermal designers who need knowledge of the use properties of various thermally conductive materials in their daily work. The number of heat management materials is quite large and the knowledge about the properties and usability of these materials has been fuzzy. The original project plan was developed in the expert group “KOTEL TR 18 Thermal design of electronics” together with VTT Technical Research Centre of Finland.

The project was executed during the years 2004 and 2005 together with the project partners and Tekes, the Finnish Funding Agency for Technology and Innovation.

This project is a co-operation project which has got the research resources from the partners and VTT. Financing came from the partners and Tekes. The partners in this project were ABB Oy Drives, OEM Electronics Oy (Aspecs Oy), Nokia Networks Oyj and Sanmina-SCI EMS Haukipudas Oy. VTT, Jari Keskinen as coordinator, made the measurements of thermal conductivity with Hot Disk method. ABB Oy Drives, Timo Koivuluoma as project manager, managed the thermal impedance measurements vs. pressure at ABB Oy Drives. VTT, Risto Hienonen as project manager, managed the reliability tests, developed with the partners the new thermal impedance measurement system for environmental tests and wrote this publication together with Jari Keskinen and Timo Koivuluoma.

The managing group for the project consisted of

Timo Koivuluoma	ABB Oy Drives
Risto Laurila	ABB Oy Drives
Kari Suomela	Nokia Networks Oyj
Timo Ruotsalainen	OEM Electronics Oy (Aspecs Oy)
Ari Toivonen	OEM Electronics Oy (Aspecs Oy)
Marcus Lahtinen	Sanmina-SCI EMS Haukipudas Oy
Hannu Hossi	VTT
Risto Hienonen	VTT
Jari Keskinen	VTT.

Espoo 30.10.2006

Risto Hienonen

Contents

List of symbols

A	area of the material [m^2]
L	thickness of material [m]
Q	time rate of heat flow [W] or [J/s]
Q/A = q =	heat flux, or time rate of heat flow per unit area [W/m^2] or [W/cm^2]
$R_{material}$	thermal resistance of piece of material [K/W]
T	temperature
TIM	thermal interface material
ΔT_{HC}	temperature difference from hot side to cold side of material [K]
λ	thermal conductivity of material [W/(m·K)]
$\theta_{material}$	thermal impedance of material [$K \cdot m^2/W$] or [$K \cdot cm^2/W$]
θ_{tot}	total thermal impedance of material and its interfaces
$\theta_{int\ cold}$	thermal impedance of the cold interface
$\theta_{int\ hot}$	thermal impedance of the hot interface

1. Introduction

The excess heat generated in electronics is a permanent problem to the users and designers of electronics, which translates into reliability problems. The thermal heat management materials help in getting a solution to the ever-growing power density demand and rising integration level and miniaturization of electronics in all sectors (from mobile phones to energy production). In this project the researched materials are called “thermal interface materials” (TIM) because they are meant for use as quite thin sheets or layers between the hot component and cooling surface. Materials used for construction of structure parts are not researched.

The actual power density (heat flux) in integrated circuits and power components of electronics is often over 100 W/cm^2, which means very high temperature hot spots. Because of this the need for good heat conduction routes is a reality. This concerns also the lower power density levels in various applications.

The very important role of the heat management materials is to establish a good thermal interface between the heat source and cooling parts. Actually these materials may improve radically the contacts between various surfaces where usual mechanical contact gives insufficient thermal contact. The heat management materials offer versatile ways for conducting excess heat from the hot high power density places to less critical areas and outside from the electronics. The use of these materials makes possible even fully passive cooling of electronics without moving parts.

The possible materials for better heat conduction are in general terms numerous e.g. solid, paste and grease. Every material type has its own way of use and own basic properties, which limit the use of the individual material to certain applications.

The thermal conductivity of heat management materials range from below 1 W/(m·K) values to near copper level 400 W/(m·K) and even to thousands of W/(m·K). The user should know how to use every material type, because the high bulk heat conductivity of materials is not always important in practical designs. More decisive is the value of the thermal impedance (temperature difference) that can be reached between the hot area and the cooling system. These are dependant of interface pressure, surface quality, etc.

There may be also problems in the long term properties of these materials. Also the way of using and assembly of these materials in electronics have large effect on the long term properties and reliability of the products.

This work concentrates on three basic areas:
– the thermal impedance and conductivity of thermal interface materials
– the long term reliability properties
– the usability of interface materials in production of electronics.

The basic information of the materials is collected from literature and specifications of the manufacturers. Thermal impedance and conductivity is measured and the reliability is tested by the tests designed for this project, and the usability information is gathered up from the designers and the own experiences of VTT. A new method was developed in this project for the measurement of the thermal impedance of material samples near practical and controllable conditions in environmental tests.

2. Goals

The main goal of this project is to research the properties and reliability of the thermal interface materials used in electronics. The materials are tested in various environmental conditions to see how their properties change in these conditions and what is their life endurance in use.

The project goals were the following:

- information gathering and selection of materials to be researched
- refining the information of material properties for the thermal designers
- measurement of the thermal impedance and conductivity of selected materials, and development of measurement methods for the samples in reliability tests
- reliability tests of materials in various environmental conditions
- mechanical and usability properties of various thermal interface material types in production of electronics
- chemical compatibility with other materials in electronics
- recyclability and composition of thermal interface materials
- development of models for material specifications and gathering information about the standards used
- publication containing the main results of the project.

The original project plan contained more measurements of electrical, physical and chemical properties of the thermal interface materials, and as an end goal was to study the recyclability and chemical composition of those materials accepted for use. Unfortunately the project resources were decreased from the original plan, and the work was limited as reported here.

3. Basic properties of materials

3.1 Thermal impedance

The very first affair in using the thermally conductive materials for heat management of electronics is to understand that the thermal impedance, or temperature difference per unit of heat flux, θ_{tot} (Equation 3.3) of an interface material system is a function of the

– thermal and mechanical properties of the bulk interface material
– properties of the interfaces between material and the surfaces attached.

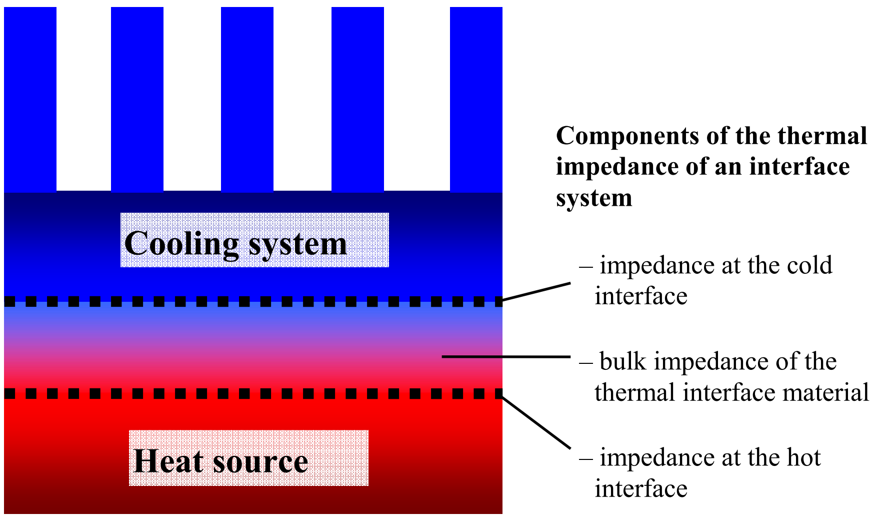


Figure 3.1. Components of the thermal impedance in a basic assembly of cooling system.

$$R_{material} = \frac{1}{\lambda} \cdot \frac{L}{A} \tag{3.1}$$

$$\theta_{material} = RA = \frac{L}{\lambda} \tag{3.2}$$

$$\theta_{tot} = \theta_{material} + \theta_{int\ cold} + \theta_{int\ hot} \tag{3.3}$$

$R_{material}$	thermal resistance of piece of material [K/W]
λ	thermal conductivity of material [W/(m·K)]

L	thickness of material [m]
A	area of the material [m^2]
$\theta_{material}$	thermal impedance of material [$K \cdot m^2/W$] or [$K \cdot cm^2/W$]
θ_{tot}	total thermal impedance of material and its interfaces
$\theta_{int\ cold}$	thermal impedance of the cold interface
$\theta_{int\ hot}$	thermal impedance of the hot interface.

The total thermal impedance θ_{tot} should be as low as possible in cooling systems. According to the Equations 3.1 to 3.3 we can reduce the thermal impedance by

– reducing the thickness of thermal interface material (TIM)
– increasing the thermal conductivity of TIM
– reducing the impedances of the hot and cold surfaces (Figures 3.3, 3.4).

The temperature difference over an interface is dependant on the heat flow, thermal impedance and surface area according to the Equation 3.4.

$$\Delta T_{HC} = \frac{Q}{A} \cdot \theta_{tot} \tag{3.4}$$

ΔT_{HC} temperature difference from hot side to cold side of material [K]

Q time rate of heat flow [W] or [J/s]

Q/A = q heat flux, or time rate of heat flow per unit area [W/m^2] or [W/cm^2].

The properties of material and its interfaces (Figure 3.1) have usually large variations depending on the way the heat interface material is used. The thermal impedance of the interfaces may be of the same order as the impedance of the bulk material itself. Therefore it is very important to have information also on the attachment properties of the thermally conductive material and of the surface quality of the heat source and cooling system.

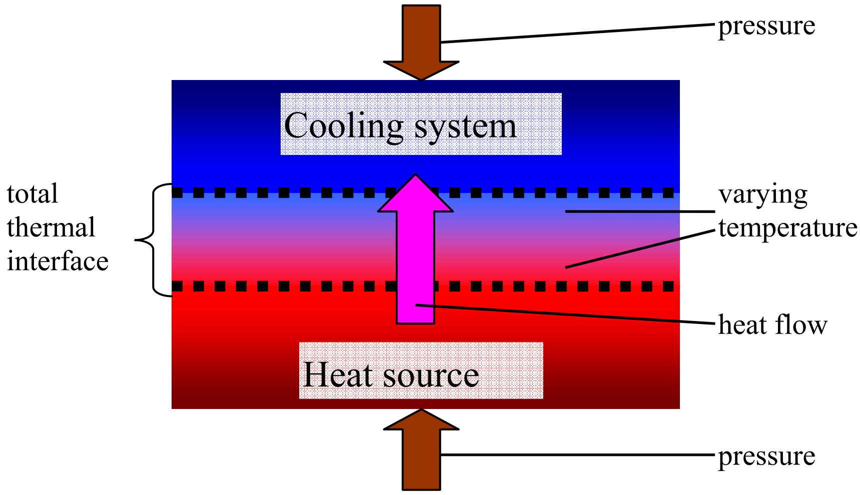


Figure 3.2. Pressure, temperature and heat flow affect the impedance of thermal interfaces.

Some materials (e.g. graphite) have different heat conductivity in various directions. The conductivity may be e.g. in a sheet ten times more in-plane direction than the conductivity through the sheet. Such properties have significant effect on the use of these materials.

The thermal conductivity of the bulk material varies when temperature, heat flow or pressure on the material is changed (Figure 3.2). These properties depend on the composition of the material, how homogenous it is, does it contain large particles, is it phase change type of material, is it solid or grease type, etc. The nominal value of thermal impedance depends on the real thickness of the used material, unfortunately the thickness is difficult to estimate because it changes with pressure and material elasticity.

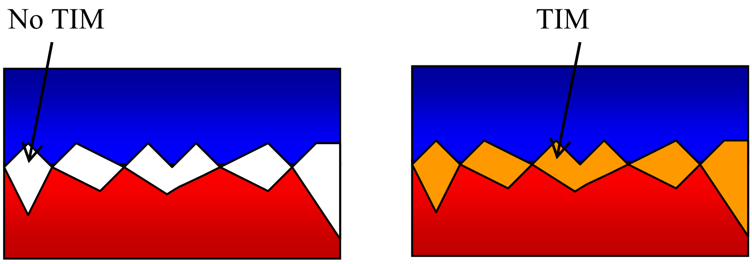


Figure 3.3. Thermal interface material (TIM) fills the gaps between the surfaces.

The interface thermal impedance depends very strongly on the pressure and surface quality. The thickness of interface depends on pressure, the interface may be uneven, have voids, air bubbles and dirt. It is often difficult to install two even surfaces together so that there is even layer of material without any irregularities or air bubbles between the surfaces (Figure 3.2). The ability of the material to intrude into the irregularities (e.g. voids) of the surfaces depends on temperature and pressure and intruding/wettability/conformability properties of the material. Actually the thermal interface material (TIM) increases the thermal contact area, which the surfaces have (Figure 3.3).

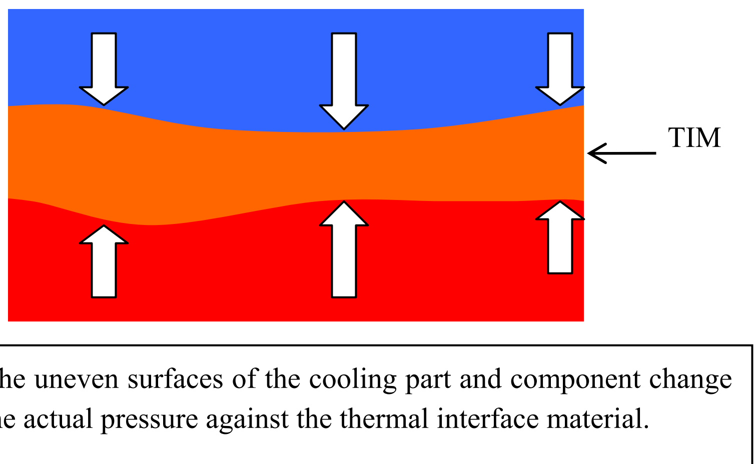


Figure 3.4. Different pressures on the thermal interface material (TIM) because of the unevenness of the surfaces.

In addition the temperature may change the interface impedance remarkably, if the bulk mechanical and thermal properties of the material change with temperature. It is also usual that the contact pressure on the material interfaces is not even (Figure 3.4), the surfaces may be uneven and the forces are not distributed evenly on the surface. It may happen that the pressure covers only partly the interface thus causing excess rise of the thermal impedance.

The usable values of thermal impedance (Equation 3.4) can be approximated from the Figure 3.5 at different power densities (heat fluxes) when the maximum temperature difference over the interface is less than 50 K.

The lowest thermal impedances of thermally conductive materials and used metal surfaces in this study are below 0,5 K·cm^2/W with greases. The best pads with < 0,3 mm thickness reach impedances 1...2 K·cm^2/W. Values over 10 K·cm^2/W are usual especially with thicker pads. These values are valid with pressure about 0,1 MPa and contain the hot and cold interfaces (Figure 3.1) of the actual material in the used test system. As a reference measurement the used test surfaces, direct metal to metal contact, without any interface material, gave an impedance about 3 K·cm^2/W. Thus the thin interface material layers offer clear advantage over the bare metal contact. In this case the quality of the metal surfaces is quite high.

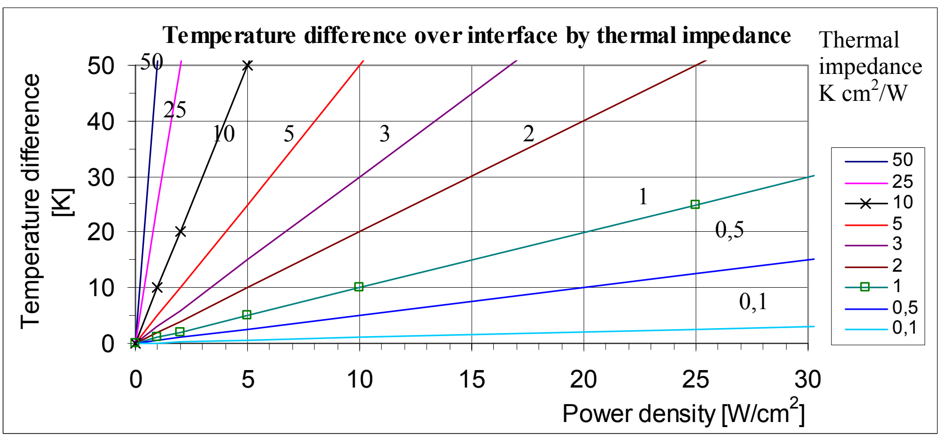


Figure 3.5. The effect of thermal impedance [K·cm^2/W] and power density (heat flux) on temperature difference over thermal interface.

Equation 3.5 gives the temperature difference over bulk material samples of various thicknesses (does not contain the interface surfaces).

$$\Delta T_{material} = \frac{Q}{A} \cdot \theta_{material} = \frac{Q}{A} \cdot \frac{L}{\lambda} \tag{3.5}$$

We can see from Figure 3.6 the usable area for various thermal conductivities of materials when power density over material is 10 W/cm^2. If thickness of material is over 1 mm then the thermal conductivity should be over 5 W/(m·K).

As mentioned above the quality of interface surfaces and the pressure over it is very decisive to the total thermal impedance over the interface.

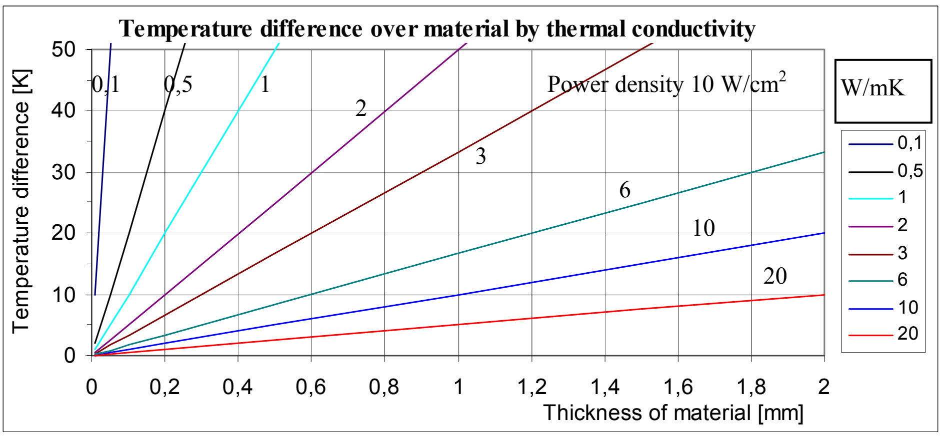


Figure 3.6. The effect of thermal conductivity [W/(m·K)] on temperature difference over interface material at power density (heat flux) of 10 W/cm^2.

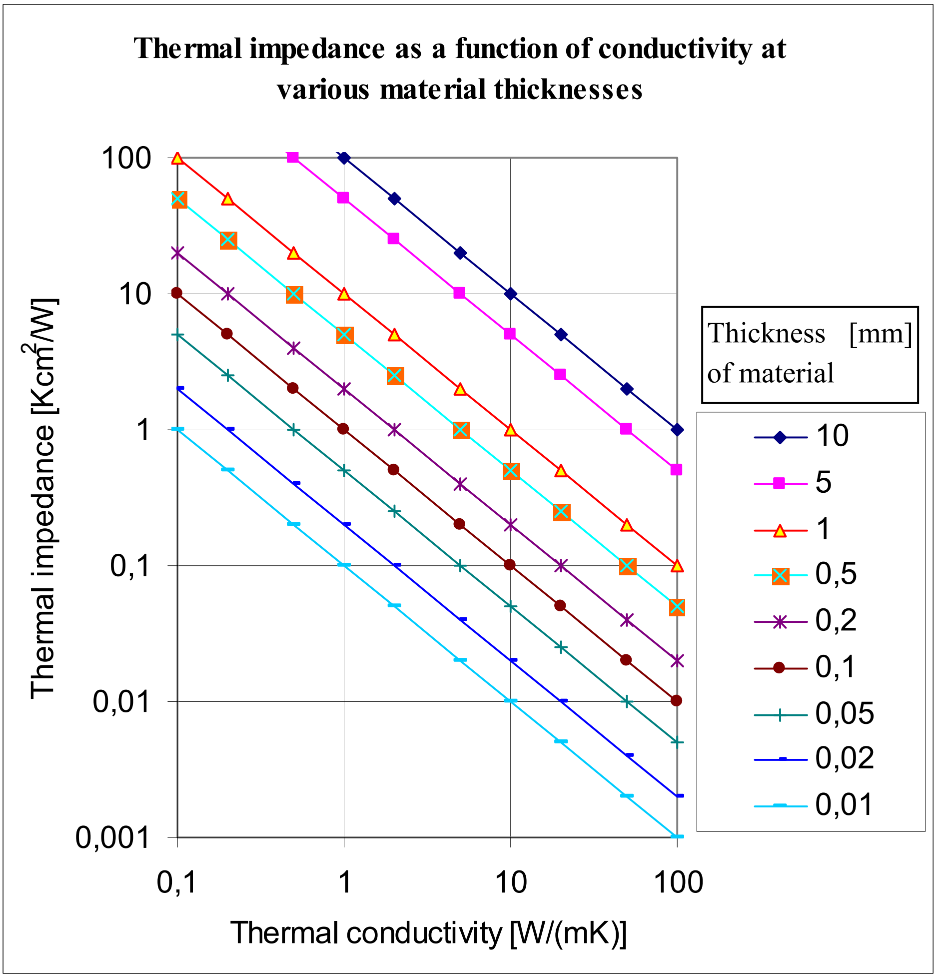


Figure 3.7. The available thermal impedance as a function of thermal conductivity at various material thicknesses.

Figure 3.7 describes the effect of conductivity and thickness of the interface material on the thermal impedance.

Impedances below 1 K·cm^2/W are only possible with thin material layers (0,05...0,5 mm) and with thermal conductivity over 1 W/(m·K). When the impedance is less than 1 K·cm^2/W the useful power density (heat flux) may rise above 25 W/cm^2.

The available lowest thickness of materials is just below 0,05 mm with greases. This means that in theory the impedance of a material could be below 0,1 $K \cdot cm^2/W$ if heat conductivity were 10 $W/(m \cdot K)$. However this level of impedance is hardly achievable because the interfaces to the heat source and cooling surface produce larger impedance than that of material in this situation.

With thick (> 5 mm) materials the impedance rises above 10 $K \cdot cm^2/W$ therefore they can be used with low power densities only.

As a point of reliability the long term stability of the heat management system depends on the stability of the above mentioned properties, the bulk material should keep steady its properties, the surface conditions should be constant, especially the contact pressure over the interfaces should be sufficient and the material should not leak out from the interface.

3.2 Thermally conductive material types

3.2.1 General aspects

Thermally conductive interface materials are available in many forms according to the way of using them in electronics. The basic forms are

- pad (sheet, foil)
- phase change pad (sheet, foil)
- tape
- filler mass (solid or liquid, also as gap filler sheet)
- grease
- paste, gel, etc.

The above mentioned material types are used mostly as thermal interface materials between the heat source and the heat sink, but the filler masses may be used as thermally conductive plate e.g. on printed wiring board to even out the temperature differences all over the wiring board. They may be adhesive and/or need extra mechanical means for having pressure over the interface.

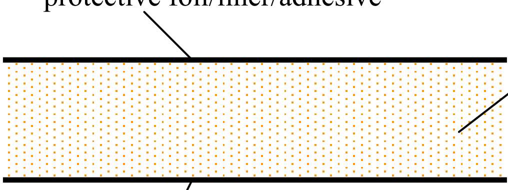


Figure 3.8. Basic structure of a pad-type material.

In addition to the above mentioned materials there are available

- thermally conductive plastics

which can be used as injection molding polymer for making parts of any form.

Thermally conductive materials consist of some binder and filler materials (Figure 3.8). The binder is often silicone elastomer, polymer, fluoropolymer, oil, etc. The filler is of thermally conductive particles, which are e.g. ceramics (Al_2O_3 aluminium oxide, TiB_2 titanium diboride, BN boron nitride, ZnO zinc oxide), graphite fiber or metal (Ag, Al).

The thermally conductive material may be supported by some carrier mesh or separate foil, which strengthens the mechanical construction of the sheet to make it more user friendly.

Materials may be electrically insulating or conductive.

The user of various material types has to consider the following general properties of various material types:

- *mechanical assembly* may be easier with solid pads than with paste/grease type
- need for *clamping hardware* is common for pad, paste and grease
- need for *electrical insulation* or *conductivity*
- thin pads need good *surface quality* to have low impedance

- pastes and greases fill better than pads the *voids and irregularities* of the interfaces
- phase change materials *compete with greases* in filling the voids and irregularities
- gap fillers allow large variation of surfaces but the thermal impedance may be high
- *electrical insulation* is achievable both with *certain* pads and pastes
- the *renewal of the heat interface* is often difficult
- the achievable *interface pressure* is mostly less than 0,1 MPa
- some materials may *leak out* from the interface.

The achievable thermal impedance depends very much on the thickness, the surface properties and pressure over the interface, therefore the user has to consider the following:

- *The lowest thermal impedance* is achievable with thin layers of greases, pads and pastes.
- Thicknesses over 1 mm means high impedance and low power density.
- The *interface pressure* should be 0,3...0,7 MPa to achieve the lowest thermal impedances specified by the manufacturers (this is not possible in most of applications).
- The *thermal conductivity* λ of a material should always be evaluated with the pressure, wettability, thickness and surface properties to get *proper thermal impedance* in the actual interface application.

3.2.2 Pad

Pads are sheet type solid materials, which are normally self supporting, flexible, elastic, and thicknesses are between 0,1...7 mm. The sheet can be cut and placed quite easily on the thermal interfaces.

The thinnest materials are used when very low thermal impedance is necessary. The thicker ones (gap filler) are used when the gap is large/varying between heat source and heat sink or the surfaces are rough or are not fully parallel. The materials may have 1...3 layers, and may have pressure sensitive adhesives and one or two additional protective films.

These materials may be delivered typically as

– sheet
– die-cut part
– roll-form.

The thermally conductive pad materials have basically the following parts:

- heat conductive binder material
- heat conductive filler material
- carrier (similar to a mesh)
- adhesive surfaces (only for fixing the material, not the contacting parts)
- protective foils (backing foil).

There is available also a polymer composite material (GELVET Honeywell) for gap filler applications. This material has thin fibers on it like artificial fur. It is used in space applications where re-workability and low outgassing is needed.

The mechanical properties include often

- good flexibility
- good compressibility
- good conformability
- good cuttability
- no leak of fluid compounds.

Depending on the type of construction the mountability properties of pads vary a lot. If materials have adhesive surfaces and are very thin then some extra accuracy in handling of materials is necessary. To attach the heat source and heat sink, the parts shall be fixed to each others with clamping device such as clips, screws or mechanical fasteners.

Also the dismounting of materials without damage on sheets may be impossible when materials are adhesive. Mostly the only way to repair a thermal interface is to remove improper material, clean the surfaces and replace it with a new pad.

3.2.3 Phase change pad

The basic construction of phase change pads is similar to the solid pads. Phase change pads have often lower thermal resistance than the solid pads. This is based on the use of polymer or wax material, which changes its phase at certain temperature (e.g. 40...80 ºC) from solid to soft paste (liquid). This means that the material fills the voids, and other irregularities of the attached surfaces better than a fully solid material (good wetting ability). Phase change materials are therefore usually thin (0,06...0,3 mm).

To achieve the full properties of phase change materials the temperature must be raised over the phase change temperature for some time, otherwise the thermal resistance may stay in the high initial value because the wetting does not happen.

The construction of phase change pads is similar to usual solid pads, but the phase change material is soft paste fluid and it is not an adhesive. So to attach the heat source and heat sink the parts shall be fixed to each others with clamping device such as clips, screws or mechanical fasteners.

The phase change sheets may consist of conformal metal carrier or graphite film, which is plated with phase change material. This construction is electrically conductive.

The phase change materials are usually highly thixotropic therefore they may not leak out.

The removal of phase change material requires mechanical means and chemical cleaning of surfaces because material adheres to surfaces harder than grease.

3.2.4 Tape

Tapes are tacky pressure sensitive adhesives whose main property is to provide good mechanical bond between the heat source and heat sink with mechanical pressure only, without a need for preheating. Material is filled acrylic polymer, Kapton film, aluminium foil, fibreglass or aluminium. Tapes are thin and their thermal conductivity is improved usually with ceramic filler materials (Al_2O_3 aluminium oxide, TiB_2 titanium diboride). The thickness is usually 0,05...1,0 mm. Thermal conductivity is mostly below 0,9 W/(m·K).

Because of the structure of tapes they are electrically isolators. The tapes are provided with extra protective foils (backing foil) for ease of handling and cutting.

The main reason to use tapes is to avoid clip or screw mounting and heat cure. Still the surface quality is critical in maintaining good adhesion.

The removal of tape material requires mechanical means and chemical cleaning of surfaces because material adheres to surfaces.

3.2.5 Gap filler mass

Filler masses are delivered as one component thixotropic paste (viscoelastic) or as two component liquid, which is spread on the thermal interface (Figure 3.9) and cured at room or higher temperature (look also Section 3.2.2 Pad). The mass is elastic and is used where e.g. several components of variable heights are connected to heat sink or enclosure. Gaps up to 3 mm may be filled with these materials.

The masses do not cause any excessive mechanical stress on component joints and printed wiring board during assembly. Filler mass may fill quite large uneven gaps between the heat source and heat sink and it is quite elastic. The compression deflection of 50% of material thickness requires only moderate pressure (e.g. < 10 kPa).

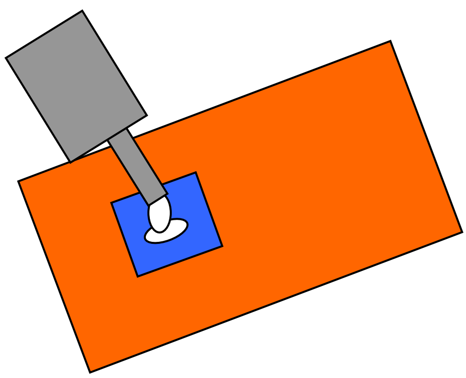


Figure 3.9. Dispensing filler mass, paste or grease on a component for a thermal interface.

The material may be used also as electrical insulator. To avoid short circuit under mechanical compression, the material may contain e.g. glass beads to prevent contact of the interface surfaces.

Some materials in contact with these may inhibit the curing of materials.

The removal of mass requires mechanical means and chemical cleaning.

3.2.6 Grease

Grease materials are used when high power density and very low thermal impedance is searched for. The binder is grease or oil (silicon, hydrocarbon), which is filled with thermally conductive particles e.g. AlN aluminium nitride, ZnO zinc oxide or Ag silver. The thickness of grease layer in the final thermal interface is low, far below 0,1 mm, depending on the particle contents, viscosity and thixotropic property of grease. Grease fills the voids and other unevenness well, but the surfaces should be at least as good as with the thinnest 0,1 mm pads.

The interface is normally electrically conductive because the attached surfaces are in contact with each others. To attach the heat source and heat sink the parts shall be fixed to each others with clamping device such as clips, screws or mechanical fasteners.

There is certain risk of leak out of grease but with proper viscosity control leak out may be negligible.

The renewal of grease interfaces requires chemical cleaning and new assembly.

3.2.7 Paste, gel

These paste-type materials are normally used as quite thin layer (< 0,1 mm) for replacing grease in thermal interfaces and/or for having fixed bond between the heat source and heat sink. The binder is silicon oil or olefin and filler is Al aluminium, Al_2O_3 aluminium oxide or Ag silver.

The properties are typically

- thin layer (< 0,1 mm)
- low thermal impedance
- the material may be electrically insulator
- good permanent bond of heat source and heat sink
- low pressure at assembly (heating during assembly improves interface)
- renewal of bond may be impossible.

The materials are one or two component, and they are cured with the help of high temperature or humidity.

Dispensing is made as in the Figure 3.9.

3.2.8 Plastics

The thermally conductive plastics for injection molding have improved thermal conductivity over the usual polymers. These materials are used as raw material for parts to replace metal as construction material.

These plastics have thermal conductivity from 1...10 W/m·K thus being on the same range as the pads. They may be electrically non-conductive or conductive.

3.2.9 Graphite fibers

Graphite fibers may have better thermal conductivity than metals. The density is lower than that of aluminium, thus if weight and high thermal conductivity is critical then these fibers offers certain possibilities. The graphite sheets have even 200…500 W/(m·K) in-plane thermal conductivity, but it is much less maybe only 1/100 of this in through-thickness [Bromanco-Björkgren 2006, GrafTech].

The fibers may be mixed with other materials, e.g. they may be mixed with thermoplastics, copper, and carbon. Values of 5 to 150 W/(m·K) conductivity is achieved with mixing graphite fibers with polymers (BP Amoco). The mixing gives mechanical and thermal properties over usual plastics and metal materials.

3.3 Thermal properties of materials

The basic bulk material property is the thermal conductivity of material. This is a vital property for all thermal interface materials, but in practical applications e.g. in thin thermal interfaces the mechanical surface properties and pressure over the interface may have larger effect on thermal impedance than the bulk material thermal conductivity. Thus it may happen that a material with better thermal conductivity is worse in relation to the thermal impedance, because its mechanical and wettability properties do not support so well the mechanical interface. Into Table 3.1 it is collected typical values of thermal conductivities and impedances of various material types.

Table 3.1. Thermal conductivity and impedance of interface material types.

Material type	**Thermal conductivity 1) W/(m·K)**	**Impedance 3) K·cm²/W**	**Recommended pressure 3) MPa**
Pad	0,3...6...16/120 2)	1...3	0,7
Phase change pad	0,7...5	0,3...0,7	0,3
Tape	0,4...0,8	1...4	..
Grease	0,6...8	0,2...1	0,3
Paste/adhesive (gap filler)	0,7...1,2	0,2...1	..
Gel	3	0,3...0,8	..

1) Values taken from the data sheets of manufacturers.
2) Last values are of one graphite material with conductivities through-thickness/in-plane.
3) [Blazej 2003.]

The available thermal conductivity values are roughly on the same range by various types. The impedances also look out quite similar with each others. But the recommended pressure by which these impedances are achieved is quite high

(Table 3.1). Therefore in practical applications where pressure is much lower, the real impedance may be much higher than in this table. See the measured values of thermal impedance in this project in Chapter 4.2.

Marotta et al. [2002] have studied the effect of interface pressure on thermal impedance of flexible graphite materials (GrafTech). According to the results (e.g. Figure 3.10) at low pressures (below 0,3 MPa) the dominant role for impedance is the gap filling of interface surfaces (wetting etc), but at higher interface pressures the bulk impedance (thermal conductivity) of interface material becomes dominant. That means simplified that at practical levels of interface pressure the absolute value of thermal conductivity of materials has not important role on the total thermal impedance of interfaces. The wettability and interface pressure are dominant mechanisms. It is important to see that at low pressures the effect of pressure is very steep on the interface impedance.

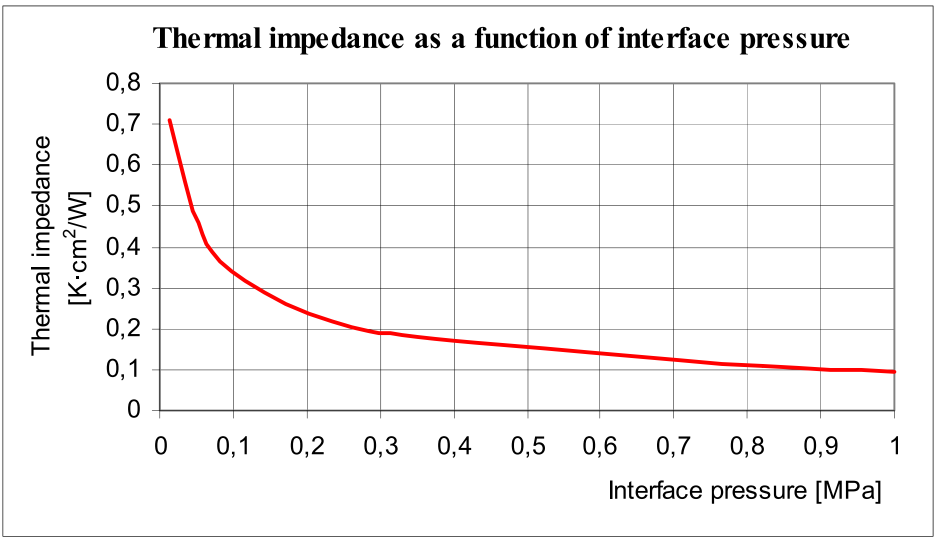


Figure 3.10. Model for the thermal impedance of an interface as a function of interface pressure, graphite material 1205 of GrafTech [Marotta et al. 2002, extract 1205 Model Predictions from the figure 3].

As a reference of published thermal impedances measured with a ASTM D-5470 based tester [Samson et al. 2005, table 3] gives thermal impedances at 0,62 MPa pressure for certain greases 0,02...0,16 K·cm^2/W and for phase change materials

0,08...0,18 K·cm^2/W. These values are criticized because they are clearly lower than in practical applications because of the high pressure used.

To help understanding various common pressure units Table 3.2 contains typical pressure values used in literature for thermally conductive materials in connection to the thermal impedance values.

Table 3.2. Conversion between pressure units MPa and PSI.

MPa	PSI
0,1	14,5
0,2	29
0,3	43,5
0,4	58
0,8	116

PSI	MPa
10	0,07
25	0,17
50	0,34
100	0,69
200	1,38

MPa = megapascal, PSI = pounds per square inch
1 MPa = 145,04 PSI

4. Measured thermal properties

The measured thermal properties of materials in this project were
- thermal conductivity with HotDisk method
- thermal impedance as a function of contact pressure with the modified method of standard ASTM D 5470-01.

Because of the nature of the materials the low thickness of material layers makes it difficult to have good results of heat conductivity of bulk materials. The impedance measurements when it includes also the interface contact impedances seem to give quite stable and repeatable results in the test conditions.

4.1 Thermal conductivity

Both basic and thin film Hot Disk methods were used in this work (see Section 7.2). The following tables give the measured values and comments for each sample. Thin samples (below 1 mm) were measured with thin film system and thicker samples as well as greases with basic system. As mentioned above, the thin film system does not allow thermal conductivities higher than 2 W/(m·K) with Hot Disk method. In case of thin film systems both insulated and non-insulated sensors were used.

For all samples several measurement parameters (heating power and time) were used. The variations in the tables for each sample are values obtained with different parameters. In principle the variation should be very small if the measurement is reliable. The reliability of the measurement can be judged from several measured values, e.g. temperature increase and in the case of thin film samples the obtained thermal conductivity for the known backing material.

As calibration samples for basic sensor materials like ceramics and stainless steel are normally used. For the thin film sensor calibrations are made with paper.

For thick samples that are not compressible the normal pressure during Hot Disk measurement has been of the order of 0,01 MPa. Since the minimum value for pressure of published values is higher, a pressure of about 0,1 MPa was used in the measurements.

Some of the thin samples mentioned on the tables were not measured because the thermal conductivity reported by the manufacturer was in the range that had been found to give unreliable results for other materials of similar type.

The measured technically correct values are **bolded** in Table 4.1. The technically correct values are near the values given by manufacturers.

The star (*) marked material types were selected for the reliability tests (see Chapter 4.2).

Table 4.1. Thermal conductivity [W/(m·K)] of pads.

Material number and type	**Measured W/(m·K)**	**Specification W/(m·K)**	**Thickness [mm] and comments**
1* Pad	No valid values	3	measured value 0,35 mm, not in the measurement range
2* Pad	**1,22–1,87**	1,6	0,23 mm
3 Pad	No valid values	3	0,254 mm, not in the measurement range
6 Pad	0,67–1,07	0,8–1	1,524 mm, sample slightly too thin for basic sensor and too thick for thin film sensor
6 Pad, as 3-fold	**0,73–0,78**	0,8–1	3x1,524 mm, basic sensor
7 Pad	0,7–5	1,0	0,25 mm, measurement not reliable, with some parameters values up to 5 W/(m·K), rough surface, probably air gaps
8 Pad	0,66–3,8	1,1	measured value 0,24 mm, measurement not reliable, with some parameters values up to 3,8 W/(m·K), after removing the glue the results were still varying
9* Pad	1,25–14,4	2,07	0,25 mm, measurement not reliable, outside range
10 Pad	**0,56–1,18**	1,5	0,27 mm
11* Pad	0,33–1,54	0,84–1,0	measured value 0,17 mm, measurement not reliable
12 Pad	..	0,84–1,0	0,5 mm

15 Pad	**0,96–1,09**	0,84–1,0	2,0 mm, 2-fold material during measurement, basic sensor pressure 1 kg/cm^2
16 Pad	..	0,41–1,1	0,25 mm
17 Pad	..	0,41–1,1	0,5 mm
22 Pad	**0,60–0,71**	0,41–1,1	3,0 mm, basic sensor, pressure 1 kg/cm^2
23 Pad	..	0,28–0,74	0,5 mm
28 Pad	0,2	0,28–0,74	3,0 mm, basic sensor, 2-fold sample (2x1,5 mm), test with low pressure (about 0,1 kg/cm^2)
28 Pad	0,45–0,52	0,28–0,74	3,0 mm, basic sensor, pressure 1 kg/cm^2
37 Pad	..	5	0,2 mm, graphite film, conductivity outside the measurement range

There are obviously problems with thin film sensor when the heat conductivity is relatively high (> 1 W/(m·K)). For paper the systems gives very repeatable results (0,1 W/(m·K)). However, it seems that for thermal conductivities from about 0,3 to 2 W/(m·K) the results are not any more reliable. In this case the obtained thermal conductivity changes as a function of applied power. The control parameters do not reveal this inaccuracy, e.g. the thermal conductivity of the backing stainless steel is acceptable.

Manufacturer of the materials No. 11 to 28 gives the thermal conductivity values for these pads as a function of pressure. Hot Disk measurements gave values similar to the ones presented on the data sheets. The thin film measurement of material No. 11 should be ignored and the value obtained for No. 12 and No. 15 used instead (they are same type).

Hot Disk measurements did not give practical information from the phase change materials (Table 4.2). In some samples it was obvious that the structure of the sample (aluminium coated with another material) was not suitable. The same comment as with thin pads applies also here: The thin film sensor is not reliable when the conductivity is of the order of 0,3–2 W/(m·K).

Table 4.2. Thermal conductivity [W/(m·K)] of phase change materials.

Material number and type	Measured W/(m·K)	Specification W/(m·K)	Thickness [mm] and comments
41 Pad, phase change	0,42–0,67	0,9	0,14 mm, the material has phase change material on aluminium, the Hot Disk method is not suitable for this kind of structure
41 Pad, phase change [1)]	0,63–1,19	0,9	0,14 mm, the material has phase change material on aluminium, the Hot Disk method is not suitable for this kind of structure
42 Pad, phase change	1,4–4,6	0,9	0,14 mm, measurement not reliable, compare with the material above, also negative values were obtained
43* Pad, phase change	..	1,6	polyimide carrier 0,07 mm, total thickness 0,16 mm, phase change, measurement not successful, gives only negative values
44 Pad, phase change	0,68–14	0,6	0,18 mm, measurement not reliable, gives also negative values with some parameters
45 Pad, phase change	..	3	0,13 mm, not measured, conductivity outside the measurement range
47* Pad, phase change	0,16–0,3	0,7	0,09 mm, measurement not reliable, gives also negative values with some parameters
48 Pad, phase change	..	5	0,25 mm, graphite film, conductivity outside the measurement range

1) This lot of material 41 was heat treated 1 h at 75 ^{o}C to melt the phase change material.

Table 4.3. Thermal conductivity [W/(m·K)] of greases and fillers.

Material number and type	**Measured W/(m·K)**	**Specification W/(m·K)**	**Thickness [mm] and comments**
56* Grease	1,49–1,60	3,5	Sensor surrounded by the liquid
57* Grease	1,72–1,77	6,6	Sensor surrounded by the liquid
61* Grease	3,93–3,99	3,8	Sensor surrounded by the material
60 Grease	0,54–0,60	0,6	Sensor surrounded by the material
62 Gap filler	1,60–1,78	0,7	Sensor surrounded by the material

These measurements of greases (Table 4.3) were performed with the basic sensor. The measurements can be considered reliable since they were repeatable and the results were similar and not dependent on the measurement power and time.

The results of greases No. 60 and 61 are similar to the figures given by manufacturer. The measured results of grease No. 62 are double the values of the manufacturer and values of grease No. 56 and 57 are far below the values of the manufacturer.

Again with thin samples of tapes in the Table 4.4 the reliability of the measurements is not good and the values obtained with varying measurement parameters are different from each other. The glue of the tapes was so strong that to prevent breaking the sensor when removing tape, the surface against thin film sensor was covered with alumina powder. The particle size of this alumina powder is about 100 nm and because of the thinness of the layer it causes only negligible change to the thermal conductivity.

Table 4.4. Thermal conductivity [W/(m·K)] of tapes.

Material number and type	**Measured W/(m·K)**	**Specification W/(m·K)**	**Thickness [mm] and comments**
65 Tape	0,32–0,56	0,6	0,125 mm, sticky surface covered with alumina nanopowder
66 Tape	0,57– 1,0	0,6	0,25 mm, sticky surface covered with alumina nanopowder
68 Tape	0,38–1,16	0,6	0,5 mm, sticky surface covered with alumina nanopowder
68 Tape	0,58–1,05	0,6	without alumina
68 Tape, 6-fold	0,50–0,57	0,6	6x0,5 mm = 3 mm, basic sensor
69* Tape	0,43–0,73	0,6	0,15 mm, sticky surface covered with alumina nanopowder
73 Tape	0,89–1,5	0,8	0,127 mm, sticky surface covered with alumina nanopowder
73 Tape	0,42–1,3	0,8	0,203 mm, sticky surface covered with alumina nanopowder
73 Tape	..	0,8	0,279 mm, not measured
76 Tape	0,50–1,82	0,37	0,13 mm, sticky surface covered with alumina nanopowder
77 Tape		0,5	0,15 mm
78 Tape		?	0,28 mm

4.2 Thermal impedance as a function of pressure

4.2.1 Test conditions in the impedance measurements

Tables 4.5–4.7 contain information about power densities and temperatures on cold and hot sides of materials during impedance measurements by ABB according to the method described in Chapter 7.3.

Table 4.5. ***Pads****, power densities and temperatures used in the impedance measurements.*

Material number and physical data	Power density in measurements	Lower side temperature	Upper side temperature
..	[W/cm^2]	[ºC]	[ºC]
1* **Pad 0,38 mm, 5 kV, λ=3, sticky1, 1,1@340 kPa**	21	58	100
2* **Pad 0,23 mm, 5,5 kV, λ=1,6, sticky1, 4@340 kPA**	13	40	102
3 Pad 0,25 mm, 4 kV, λ=3,5, 2,1@340 kPa	16	46	100
4 Pad 1, 5mm 6 kV, λ=1, sticky1, 10@30 kPa	5	22	100
6 Pad 1,5 mm, 6 kV, λ=0,8, sticky1, 12,9@0,1 MPa	4	21	103
9* **Pad 0,25mm, 5 kV, λ=2,07, 1,23@3 MPa**	20	53	97
11* **Pad 0,25 mm, 2,8 kV, λ=0,8, sticky1, 5,7@0,07 MPa**	14	42	95
12 12 Pad 0,5 mm, 3,3 kV, λ=0,8, sticky1, 9,7@0,07 MPa	10	33	99
23 Pad 0,5 mm, 1,6 kV, λ=0,28, sticky1, 16,1@70 kPa, 5,2@0,34 MPa	9	30	93
29* **Graphite** pad **0,13 mm, λ=16, 0,19@0,1 MPa**	25	65	85
30* **Graphite pad 0,13 mm, λ=16, sticky1, 0,35@0,1 MPa**	25	66	83
34 Pad 0,2 mm, 1,5kV, λ=5,5, 0,4@?	20	54	100
35 Pad 0,5 mm, 4 kV, λ=1,3, 3,8@0,1 Mpa	10	33	103
36 Pad 0,5 mm, 1kV, λ=5, 1@?	17	48	95
37 Pad 0,2 mm, λ=5,5, 0,36@?	25	66	95
38 Pad 0,175 mm, λ=6, sticky1, 0,19@?	23	63	96
40 Pad 0,5 mm, 8 kV, λ=5,6, sticky1, 1,6@30 kPa	20	55	99

* marked **bolded** types were selected for the reliability tests after impedance measurements.

*Table 4.6. **Phase change pads**, power densities and temperatures used in the impedance measurements.*

Material number and physical data	**Power density in measurements**	**Lower side temperature**	**Upper side temperature**
..	[W/cm^2]	[ºC]	[ºC]
41 Pad, phase change 65 ºC 0,14 mm, λ=0,9, 2,3@0,34 MPa	25	65	90
42 Pad, phase change 65 ºC 0,14 mm, λ=0,8, sticky1	24	65	108
43* **Pad, phase change 55 ºC 0,1 mm, 5 kV, λ=1,6, 0,8@0,34 MPa**	22	59	112
44 Pad, phase change 46 ºC, 0,18 mm, sticky1, λ=0,6, 0,11@2,1 MPa	23	60	109
45 Pad, phase change 65 ºC λ=3, 0,07@0,34 MPa	13	39	104
47* **Pad, phase change 51-58 ºC, 0,09mm, λ=0,7/75, sticky1, 0,28@0,34 MPa**	25	67	86
48 Pad, phase change 58 ºC, 0,25mm, sticky1 λ=5, 0,28@?	23	60	113

* marked **bolded** types were selected for the reliability tests after impedance measurements.

The thermal impedances as a function of pressure were measured at ABB Oy. Drives with a method similar to the standard ASTM D5470-01. The pressure over sample is controllable over 0,1...0,8 MPa range. The sample diameter is 100 mm, and the heat flow is max 2200 W allowing maximum power density of 28 W/cm^2 through material samples.

Table 4.7. ***Greases, tapes and air****, power densities and temperatures used in the impedance measurements.*

Material number and physical data	**Power density in measurements**	**Lower side temperature**	**Upper side temperature**
..	[W/cm^2]	[ºC]	[ºC]
52 Grease λ=0,8, 0,14@50 ºC	24	64	83
53 Grease λ=0,9, 0,13@50 ºC	24	64	86
54 Greaselike λ=0,6	22	59	87
55* **Grease λ=0,9**	25	63	89
56* **Grease λ=3,5, 0,16@?**	23	60	70
57* **Grease λ=6,6, 0,08@?**	24	64	75
58 Grease λ=3,8, 0,2@?	25	66	76
59 Grease λ=7, 0,04@?	24	65	91
60 Grease, λ=0,8, 0,775@138 kPa	20	55	95
61* **Grease λ=3,8, 0,11@345 kPa**	25	66	83
69* **Tape , λ=0.8...0.45/-50...+150 ºC, 3.64@?**	..	..	..
76* **Tape λ=0.37, 3.7@<7 kPa**	..	..	..
84 Air λ=0,03	20	54	97

* marked **bolded** types were selected for the reliability tests after impedance measurements.

4.2.2 Results of impedance measurements as a function of pressure

The results of impedance measurements are in the Tables 4.8–4.10.

The thermal impedances are measured as a function of contact pressure. The pressures used were 0,1 / 0,2 / 0,4 / 0,8 MPa.

Table 4.8. Thermal impedances of pads measured as a function of pressure.

Material number, type, and physical data	**Thermal impedance** [K·cm²/W]			
Pressure [MPa]	0,1	0,2	0,4	0,8
1* **Pad 0,38 mm, 5 kV, λ=3, sticky1, 1,1@340 kPa**	1,9	1,8	1,7	1,7
2* **Pad 0,23 mm, 5,5 kV, λ=1,6, sticky1, 4@340 kPA**	5,0	4,6	4,3	4,0
3 Pad 0,25 mm, 4 kV, λ=3,5, 2,1@340 kPa	3,6	3,2	2,8	2,6
4 Pad 1, 5mm 6 kV, λ=1, sticky1, 10@30 kPa	17,3	16,4	15,4	14,4
6 Pad 1,5 mm, 6 kV, λ=0,8, sticky1, 12,9@0,1 MPa	20,3	19,8	19,1	18,3
9* **Pad 0,25mm, 5 kV, λ=2,07, 1,23@3 MPa**	2,5	2,3	1,8	1,4
11* **Pad 0,25 mm, 2,8 kV, λ=0,8, sticky1, 5,7@0,07 MPa**	4,3	3,8	3,3	2,9
12 12 Pad 0,5 mm, 3,3 kV, λ=0,8, sticky1, 9,7@0,07 MPa	8,5	7,0	5,8	5,0
23 Pad 0,5 mm, 1,6 kV, λ=0,28, sticky1, 16,1@70 kPa, 5,2@0,34 MPa	14,8	8,3	5,0	3,5
29* **Graphite pad 0,13 mm, λ=16, 0,19@0,1 MPa**	0,8	0,6	0,3	0,2
30* **Graphite pad 0,13 mm, λ=16, sticky1, 0,35@0,1 MPa**	0,6	0,5	0,4	0,3
34 Pad 0,2 mm, 1,5kV, λ=5,5, 0,4@?	2,8	2,3	1,8	1,6
35 Pad 0,5 mm, 4 kV, λ=1,3, 3,8@0,1 Mpa	7,5	7,1	6,9	6,6
36 Pad 0,5 mm, 1kV, λ=5, 1@?	3,4	2,7	2,1	1,7
37 Pad 0,2 mm, λ=5,5, 0,36@?	1,2	0,9	0,7	0,5
38 Pad 0,175 mm, λ=6, sticky1, 0,19@?	1,4	1,3	1,1	0,9
40 Pad 0,5 mm, 8 kV, λ=5,6, sticky1, 1,6@30 kPa	2,3	2,2	1,8	1,7

* marked **bolded** types were selected for the reliability tests after impedance measurements.

Table 4.9. Thermal impedances of phase change pads measured as a function of pressure.

Material number, type, and physical data	**Thermal impedance** [K·cm²/W]			
Pressure [MPa]	0,1	0,2	0,4	0,8
41 Pad, phase change 65 ºC 0,14 mm, λ=0,9, 2,3@0,34 MPa	1,0	0,8	0,7	0,6
42 Pad, phase change 65 ºC 0,14 mm, λ=0,8, sticky1	1,7	1,6	1,5	1,3
43* **Pad, phase change 55 ºC 0,1 mm, 5 kV, λ=1,6, 0,8@0,34 MPa**	2,2	2,2	2,1	2,0
44 Pad, phase change 46 ºC, 0,18 mm, sticky1, λ=0,6, 0,11@2,1 MPa	2,0	2,0	1,8	1,6
45 Pad, phase change 65 ºC λ=3, 0,07@0,34 MPa	5,2	5,1	4,8	4,7
47* **Pad, phase change 51–58 ºC, 0,09mm, λ=0,7/75, sticky1, 0,28@0,34 MPa**	0,7	0,6	0,5	0,4
48 Pad, phase change 58 ºC, 0,25mm, sticky1 λ=5, 0,28@?	2,2	2,1	2,0	1,9

* marked **bolded** types were selected for the reliability tests after impedance measurements.

Table 4.10. Thermal impedances of greases measured as a function of pressure.

Material number, type, and physical data	**Thermal impedance** [K·cm^2/W]			
Pressure [MPa]	0,1	0,2	0,4	0,8
52 Grease λ=0,8, 0,14@50 ºC	0,7	0,7	0,4	0,3
53 Grease λ=0,9, 0,13@50 ºC	0,7	0,6	0,6	0,5
54 Greaselike λ=0,6	2,3	1,0	0,7	0,6
55* **Grease λ=0,9**	0,9	0,9	0,8	0,7
56* **Grease λ=3,5, 0,16@?**	0,2	0,2	0,2	0,2
57* **Grease λ=6,6, 0,08@?**	0,2	0,2	0,2	0,2
58 Grease λ=3,8, 0,2@?	0,3	0,2	0,2	0,2
59 Grease λ=7, 0,04@?	0,9	0,8	0,7	0,5
60 Grease, λ=0,8, 0,775@138 kPa	3,7	1,7	1,5	1,3
61* **Grease λ=3,8, 0,11@345 kPa**	0,7	0,6	0,2	0,2
69* **Tape, λ=0,8...0,45 /-50...+150 ºC, 3,64@?**	..	..	..	..
76* **Tape λ=0,37, 3,7@<7 kPa**	..	..	..	..
84 Air λ=0,03	2,8	2,5	1,9	1,3

* marked **bolded** types were selected for the reliability tests after impedance measurements.

4.3 Thermal impedance as a function of point temperature

The following figures (Figures 4.1–4.9) contain the same results of thermal impedance measurements as in Tables 4.8–4.10. Instead of giving the results as a function of pressure the results are given in the following figures as a function of the mean deviation of the measured six point temperatures on the hot and cold side of the material sample, the diameter of which is 100 mm. This deviation describes the homogeneity of the material and is thus certain measure to the quality of material. The measured four values of impedances correspond the contact pressures 0,1 / 0,2 / 0,4 / 0,8 MPa. All these temperature deviations (x-axes in figures) are normalized in the following results to power density of 25 W/cm^2.

In the pictures the highest values of impedances are measured at lowest pressure (0,1 MPa) and lowest impedance values at highest pressure (0,8 MPa).

The star * marked material types were selected for the reliability tests after impedance measurements.

Many of the materials have larger than 0,6 K variation of temperatures in the measured six points of the samples although the impedance values are lower than 2,0 $K \cdot cm^2/W$.

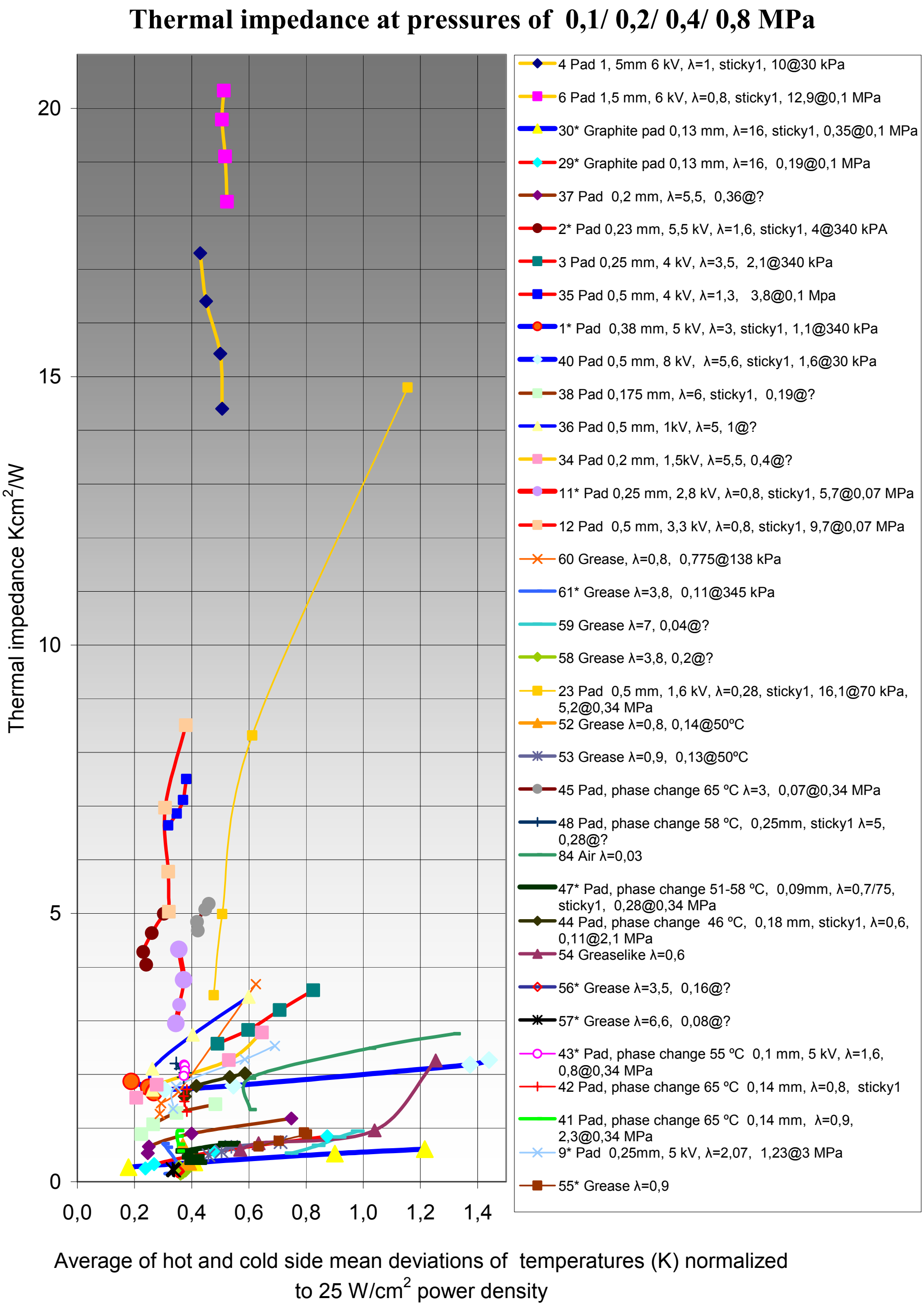


Figure 4.1. Thermal impedance of all the measured material types.

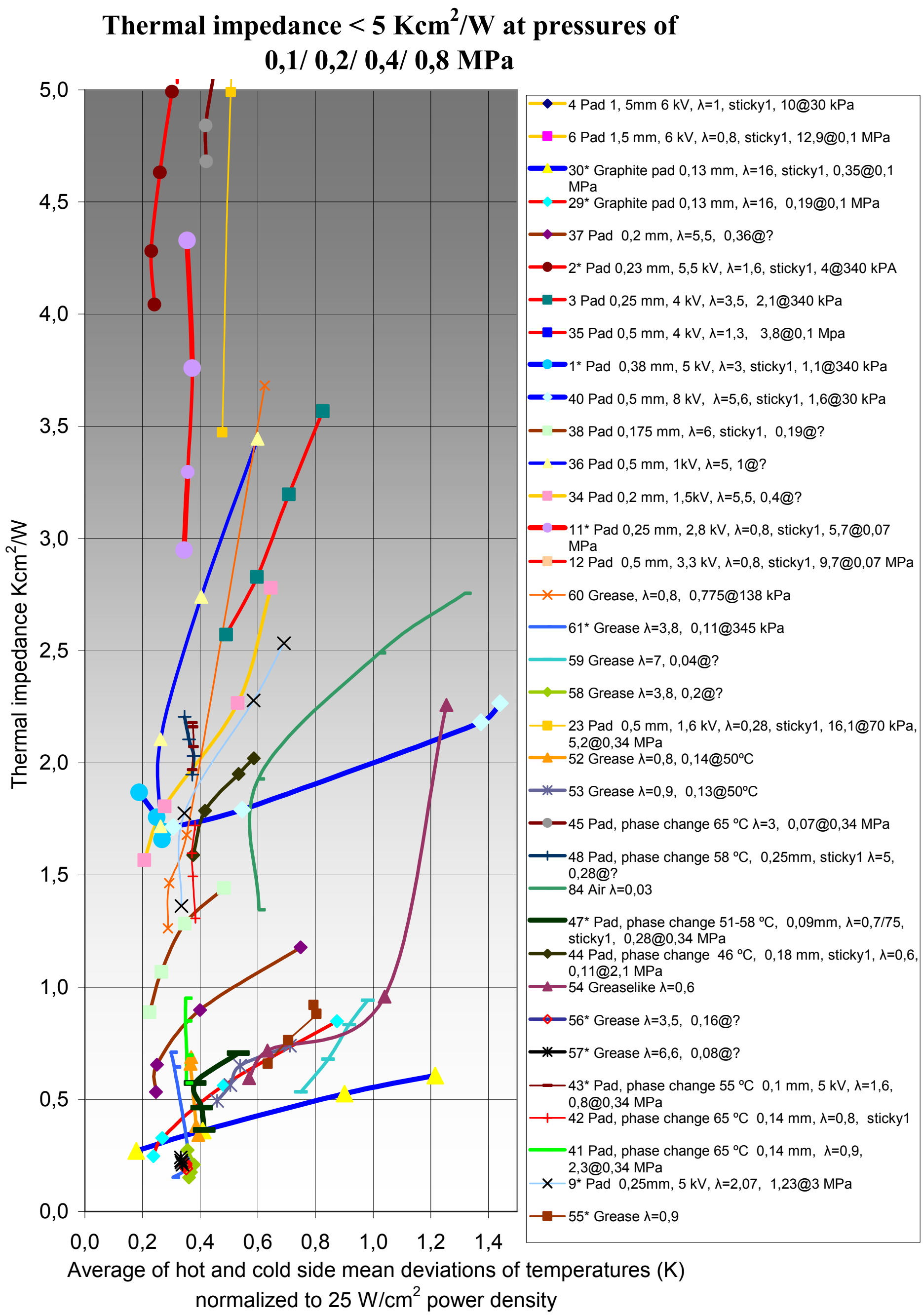


Figure 4.2. Thermal impedance less than 5,0 K·cm²/W of the measured material types.

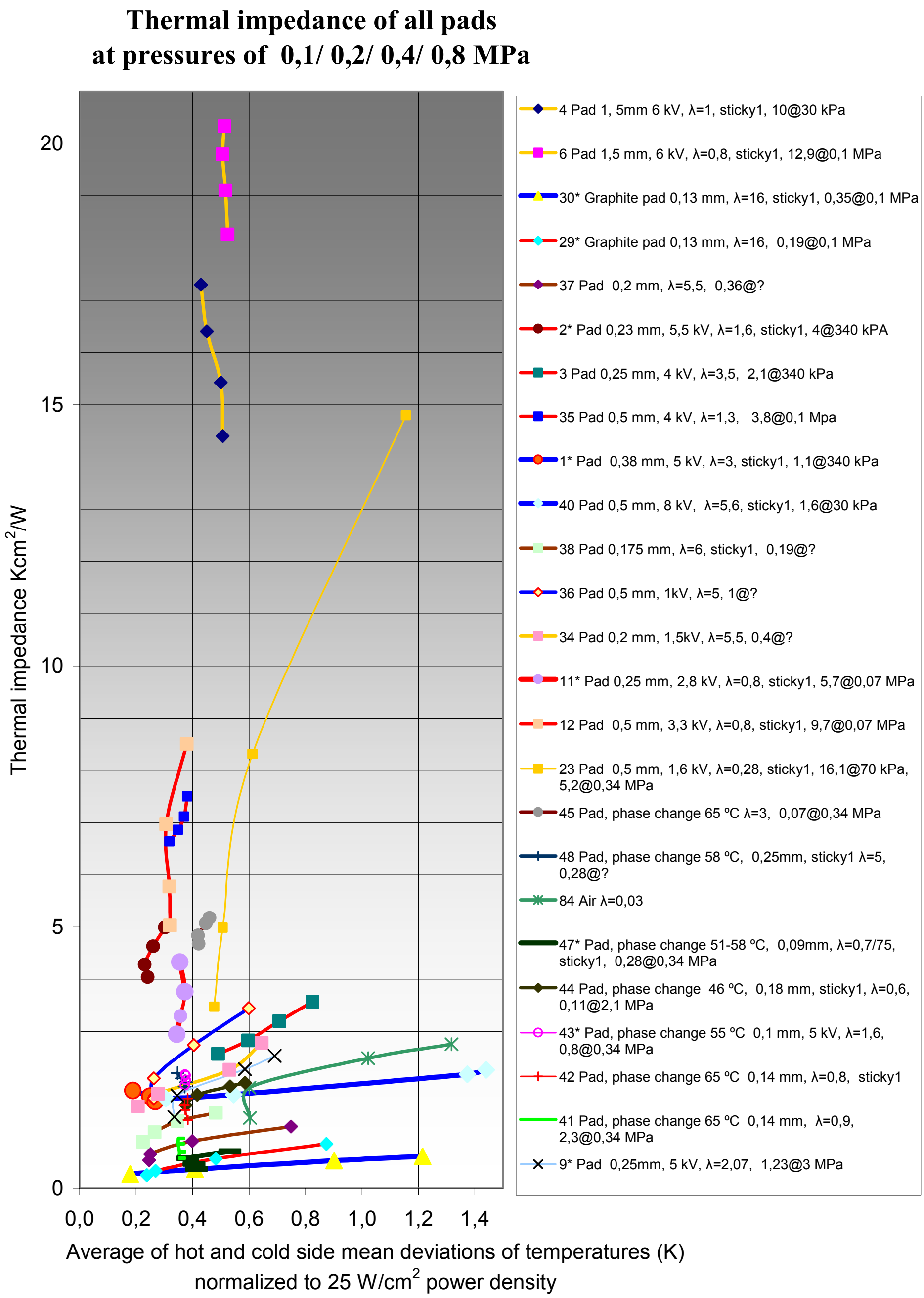


Figure 4.3. Thermal impedance of all measured pads and phase change materials.

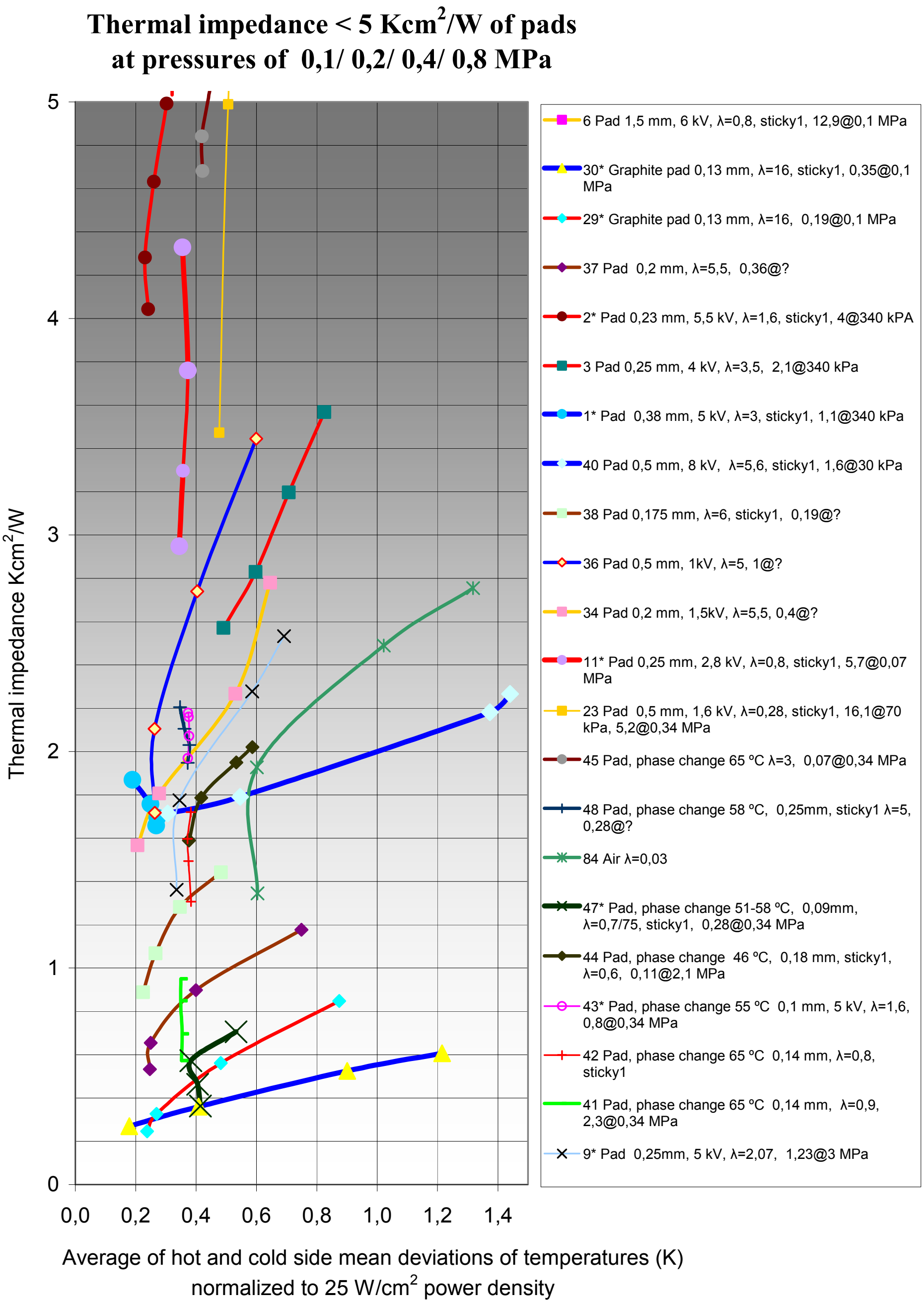


*Figure 4.4. Lowest thermal impedances of pads. The pads marked with * were selected for reliability tests.*

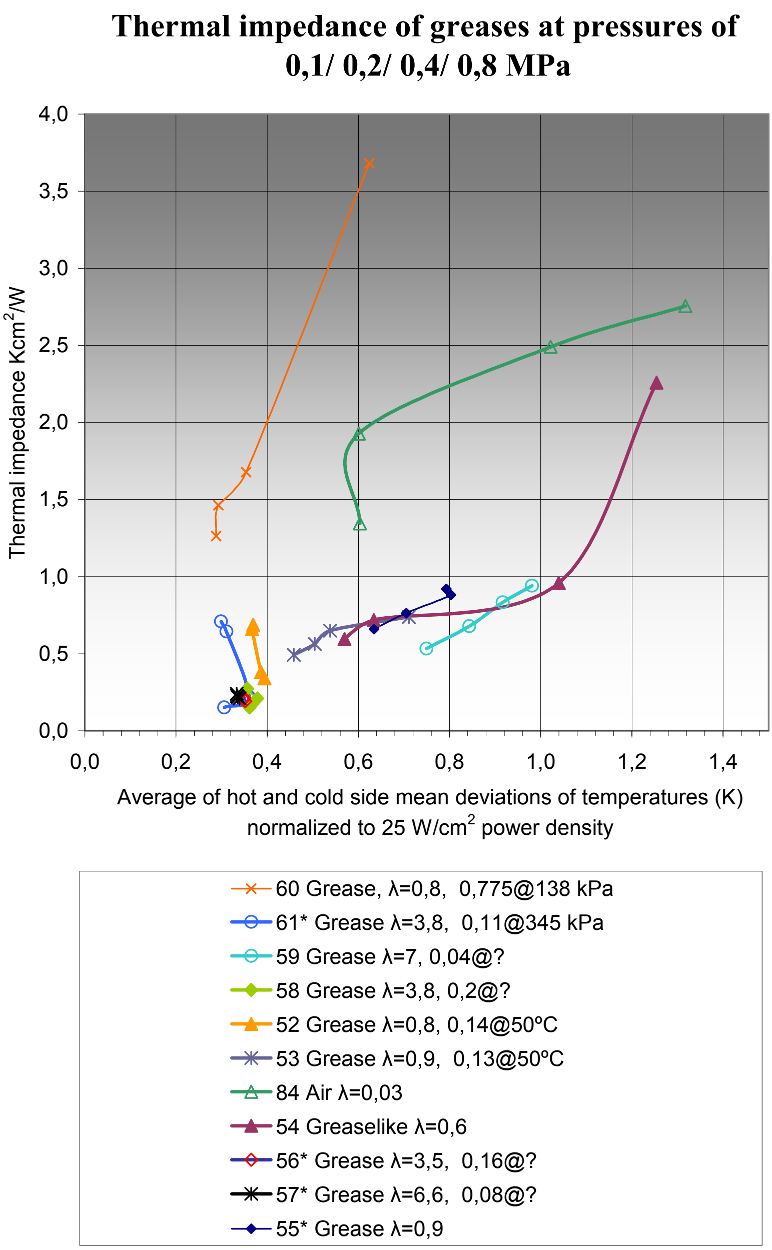


Figure 4.5. Thermal impedance of all measured greases.

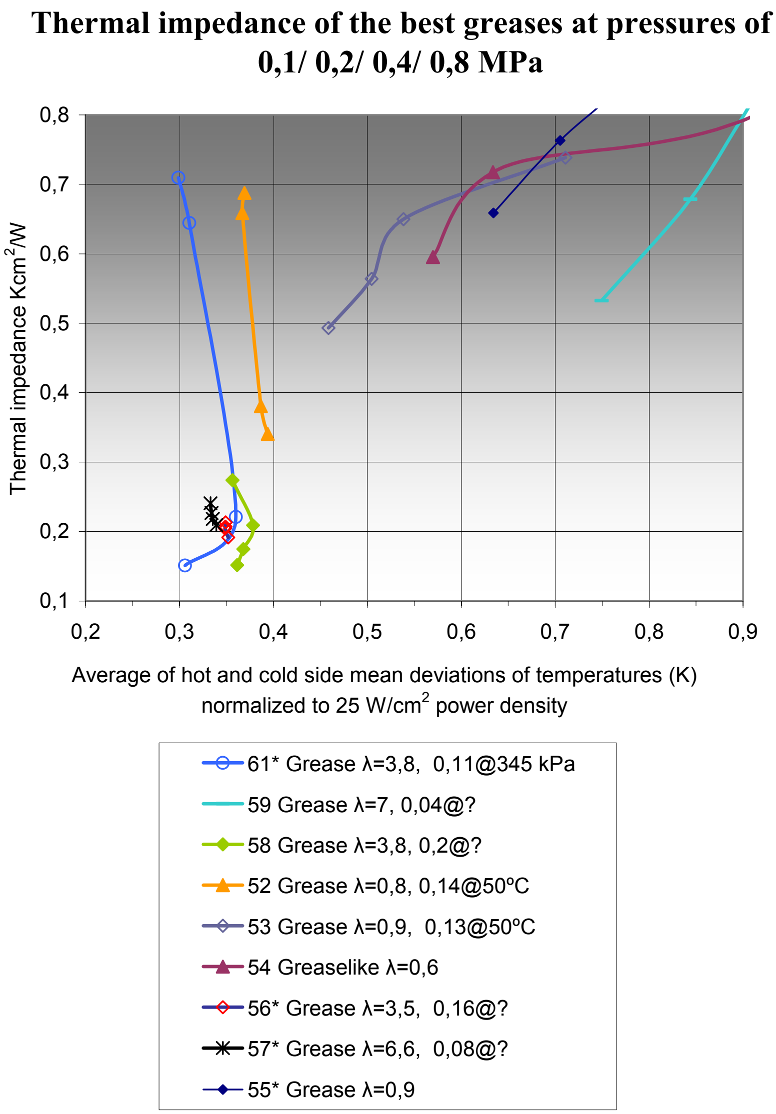


*Figure 4.6. Thermal impedance of the best measured greases. The greases marked with * were selected for reliability tests.*

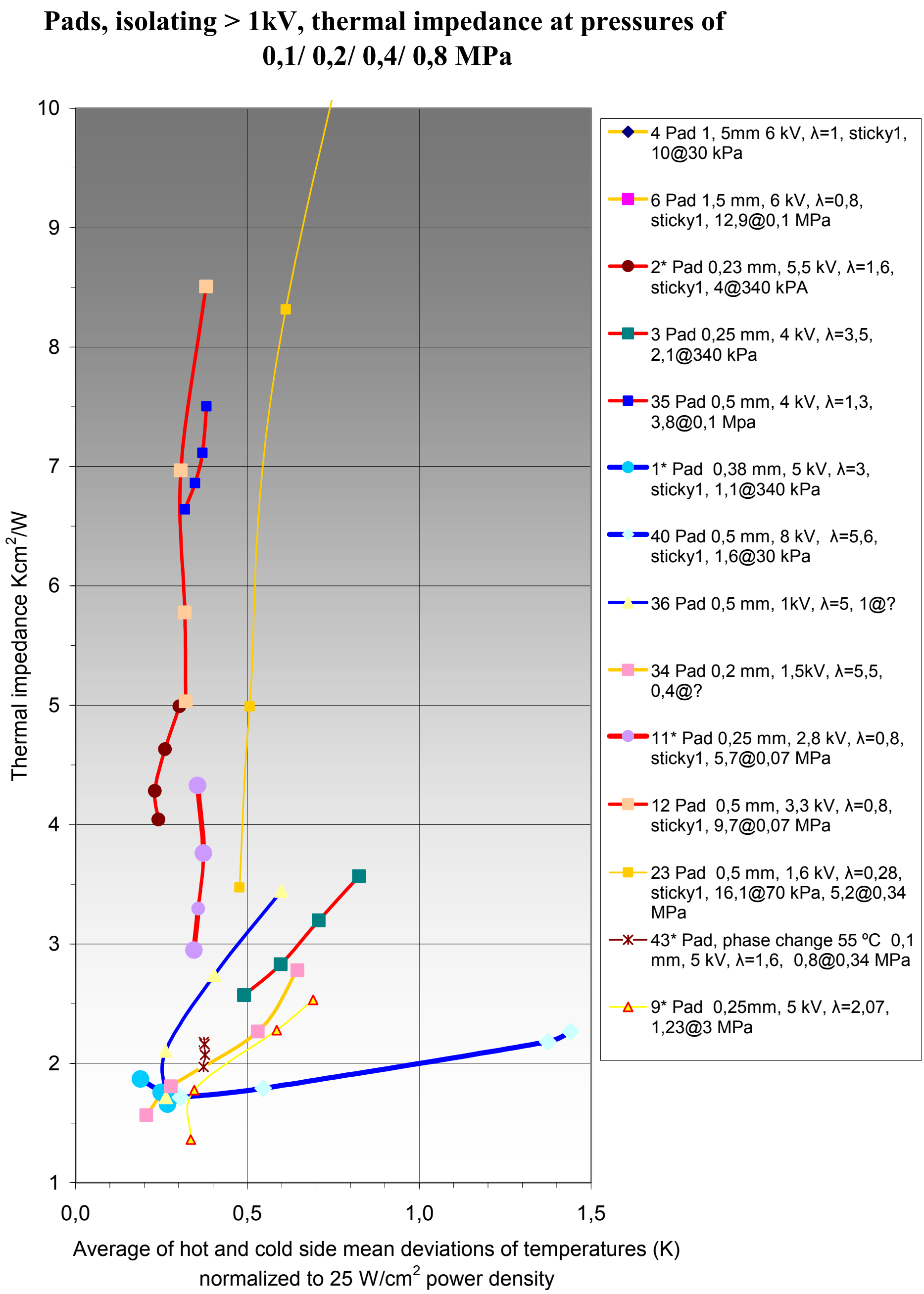


Figure 4.7. Thermal impedance of the insulating pads.

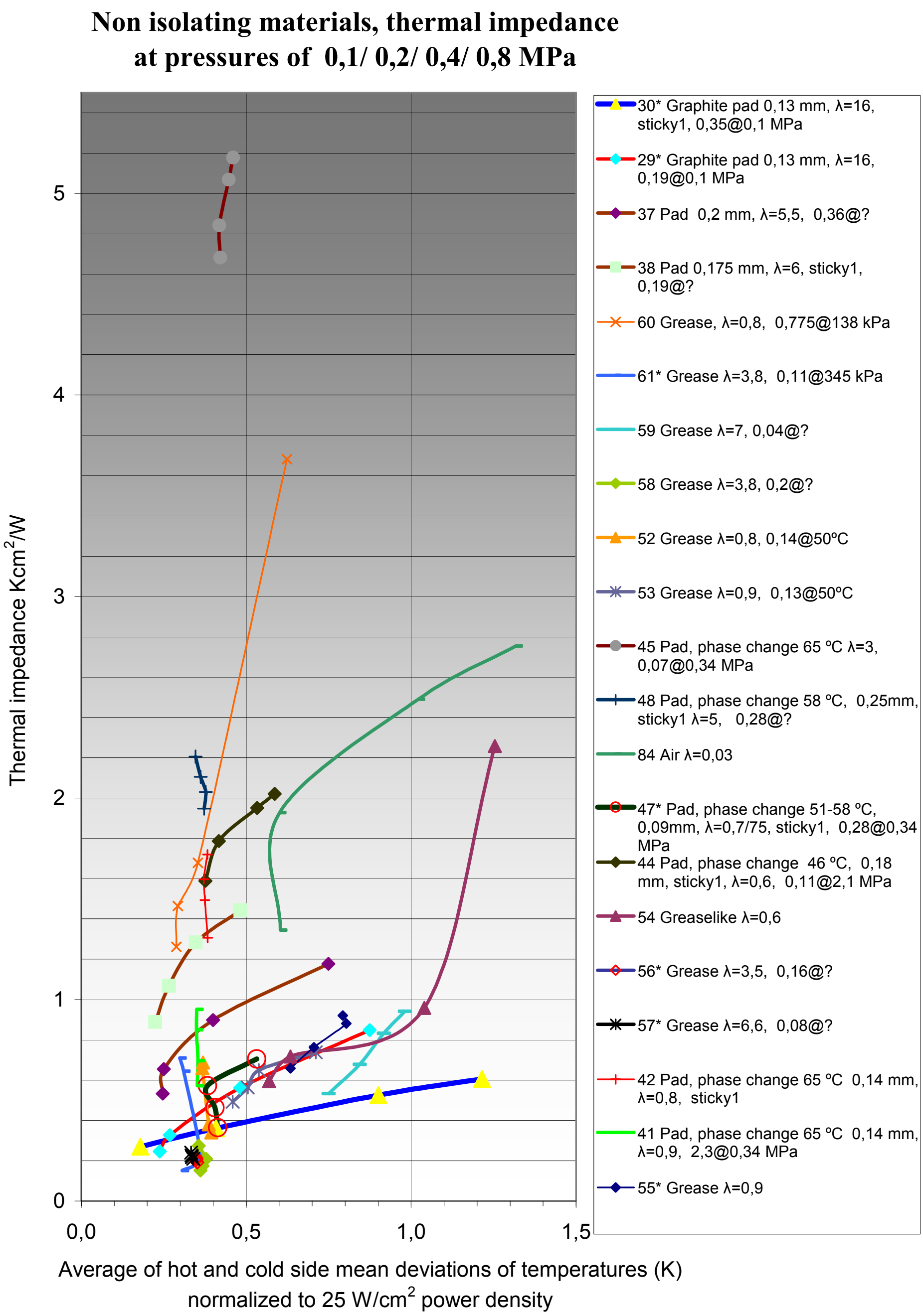


Figure 4.8. Thermal impedance of non insulating materials.

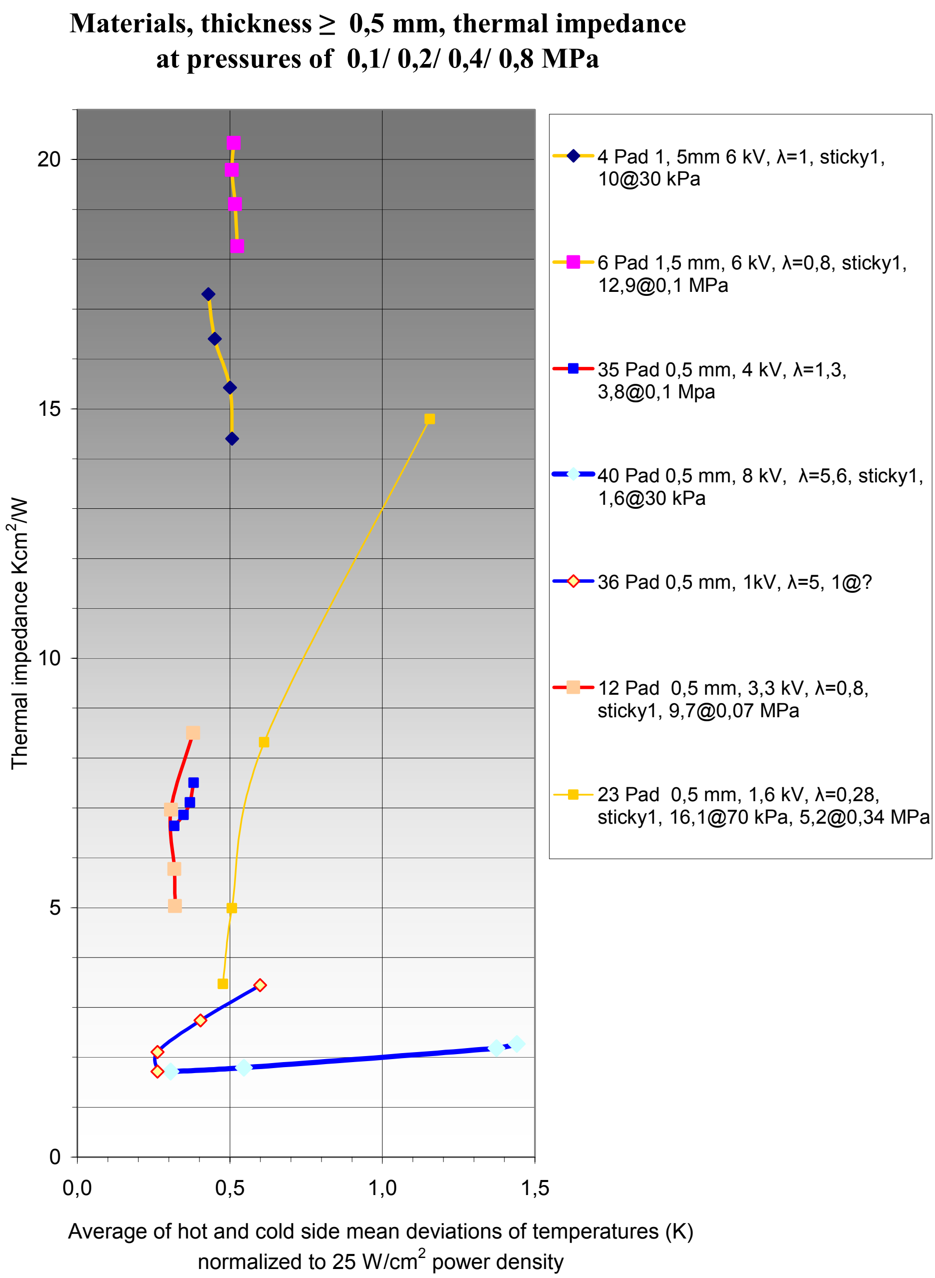


Figure 4.9. Thermal impedance of materials by thickness ≥ 0,5 mm.

Non materials by thickness ≥ 0,5 mm were selected into the reliability tests.

4.4 Correlation of thermal impedance with conductivity

The correlation of thermal impedance with the conductivity of interface materials is quite unclear in practical assemblies. Therefore it is useful to look the situation with the measured materials in this project.

Figure 4.10 contains data of the materials selected for the reliability tests. The data is given only for the measured impedances at 0,1 MPa pressure. At higher pressure the impedances are lower as seen from the results given ahead. The red curve in Figure 4.10 is a rough approximation of the correlation between conductivity and impedance.

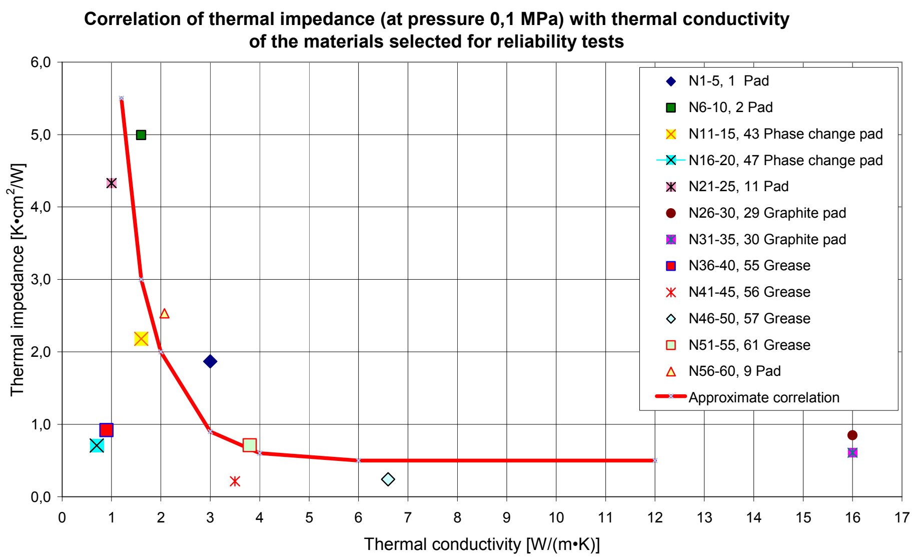


Figure 4.10. Thermal impedance as a function of thermal conductivity of materials selected for the reliability tests (no compensation of impedance because of different thicknesses).

According to the Figure 4.10 the absolute value of the thermal conductivity has quite little effect on the thermal impedance with greases and some phase change materials. These materials adapt themselves well into the interface (good wettability) and have very low thickness. The graphite materials have highest thermal conductivy, but they do not improve much the total thermal impedance. Therefore the high conductivity has less use.

The pads have generally higher impedances, which correlates with low conductivity. The graphite materials N26–30 and N31–35 have very high conductivity but the adaptability with interface is not as good as with greases. Still the graphite material reaches almost the impedance level of greases.

A similar result can be drawn if all the measured materials are presented. The highest impedances are on materials with thickness of 1,5 mm.

As a reference to the Figure 4.10 the Figure 4.11 describes the effect of interface material thermal conductivity on the thermal resistance of silicon-heat spreader interface [Viswanath et al. 2000, figure 15]. Also the thermal conductivity of heat spreader decreases the resistance. The last, green curve shows a heat spreader with a hypothetical thermal conductivity of 2000 W/(m·K).

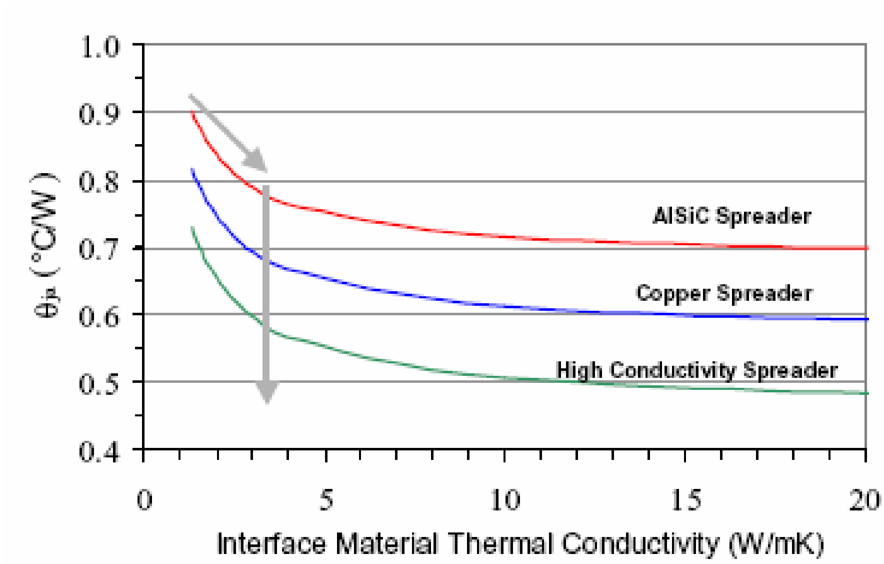


Figure 4.11. Effect of interface material conductivity and heat spreader conductivity on the thermal resistance of an interface [Viswanath et al. 2000, figure 15].

Also this Figure shows the importance of having good wettability of the interface surfaces to have lower thermal impedance.

4.5 Selection of materials for the reliability tests

Figure 4.12 and Table 4.11 contain the materials selected for the reliability tests.

The criteria for selection of materials for the reliability tests were the following:

- usable power density > 10...25 W/cm^2
- thermal impedance < 5 K·cm^2/W at 0,1 MPa pressure
- even impedance over assembly surface
- low impedance at practical (< 0,3 MPa) contact pressures
- easy handling
- possibility for electrical insulation
- thickness < 0,5 mm
- amount of materials for testing is limited by the resources for this project.

Other interests were to compare the phase change pads and graphite materials with greases.

Because the interest was on the high power density area, the thickness of materials was limited to < 0,5 mm.

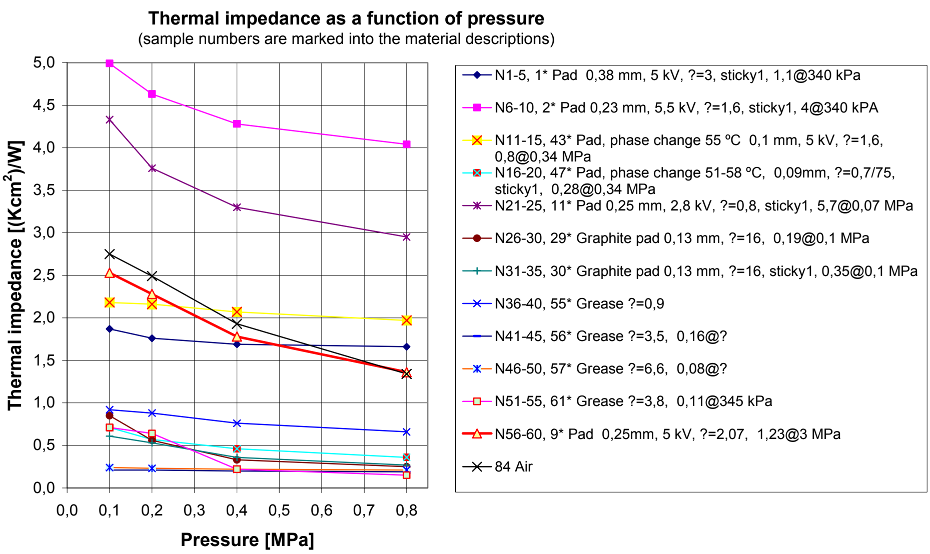


Figure 4.12. Thermal impedance of materials selected for the reliability tests.

If there is only air between the measurement surfaces then the impedance level is about 2,8 K·cm^2/W at 0,1 MPa pressure (Figure 4.12 and Table 4.11). Therefore it is natural at these power densities that materials having impedance over 5 K cm^2/W are not usable in such solutions. See also the Figure 3.5, which describes the temperature difference over material as a function of power density and thermal impedance.

Table 4.11. Thermal impedances of the materials selected for the reliability tests.

Sample numbers	**Material number and type**	**Thermal impedance** [K·cm^2/W]			
	Pressure [MPa]	0,1	0,2	0,4	0,8
N1–5	1* Pad 0,38 mm, 5 kV, λ=3, sticky1, 1,1@340 kPa	1,87	1,76	1,69	1,66
N6–10	2* Pad 0,23 mm, 5,5 kV, λ=1,6, sticky1, 4@340 kPA	4,99	4,63	4,28	4,04
N11–15	43* Pad, phase change 55 ºC 0,1 mm, 5 kV, λ=1,6, 0,8@0,34 MPa 1)	2,18	2,16	2,07	1,97
N16–20	47* Pad, phase change 51–58 ºC, 0,09mm, λ=0,7/75, sticky1, 0,28@0,34 MPa 2)	0,71	0,57	0,46	0,36
N21–25	11* Pad 0,25 mm, 2,8 kV, λ=0,8, sticky1, 5,7@0,07 MPa	4,33	3,76	3,30	2,95
N26–30	29* Graphite pad 0,13 mm, λ=16, 0,19@0,1 MPa	0,85	0,56	0,33	0,25
N31–35	30* Graphite pad 0,13 mm, λ=16, sticky1, 0,35@0,1 MPa	0,61	0,53	0,36	0,27
N36–40	55* Grease λ=0,9	0,92	0,88	0,76	0,66
N41–45	56* Grease λ=3,5, 0,16@?	0,21	0,21	0,20	0,19
N46–50	57* Grease λ=6,6, 0,08@?	0,24	0,23	0,22	0,21
N51–55	61* Grease λ=3,8, 0,11@345 kPa	0,71	0,64	0,22	0,15
N56–60	9* Pad 0,25mm, 5 kV, λ=2,07, 1,23@3 MPa	2,53	2,28	1,78	1,36
N61–62	76* Tape **λ**=0,37, 3,7@<7 kPa	..	..	..	..
N63–64	69* Tape , **λ**=0,8...0,45/-50...+150 ºC, 3,64@?	..	..	..	..
..	84 Air	2,8	2,5	1,9	1,3

1) Phase change temperature 55 ºC. 2) Phase change temperature 51...58 ºC.

5. Reliability tests

The reliability test program is planned to get practical information of the material behaviour in various environmental stresses and long term use. The properties of materials and the actual use conditions may change especially because of the changing temperature and power density in use. Therefore in this study the temperature, humidity and long term rapid change of temperature were selected as the main stresses in testing thermally conductive materials.

The material structure, composition and the way of assemblage into the user application prescribe the final thermal resistance of the heat management structure (Figure 3.1 and 3.2). To reach a repeatable way for measurements and mechanical conditions as near as possible to the user assemblages a new test structure was designed for this test program.

5.1 Test structure for materials

The main technical goal for the used test structure was to get as far as possible similar test conditions as in a practical user assembly. In addition the test structure should make possible measurement of the thermal impedance as close as possible according to the standard ASTM D 5470-01 Standard Test Method for Thermal Transmission Properties of Thin Thermally Conductive Solid Electrical Insulation materials.

The structure should maintain the stability and repeatability of

- power density through the sample of material
- even pressure against the sample
- flatness of the interface surfaces
- surface roughness at interfaces
- mechanical test structure.

These conditions should be the same through the whole test program and between various samples and independent of the material types. Very important matter was the selection of the absolute value of test pressure and the method of keeping the pressure constant during the environmental tests and measurements. The mechanical solution to fill these requirements is as described in Figure 5.1.

The test structure consists of two equal nickel-plated copper test structure halves. The material sample is placed between these halves. The pressure over the material sample is controlled by the two proper designed springs, which situate on the sides of the test structure.

This test structure with the material sample is placed into the measurement system, which produces proper power density and heat flow over the test sample (see Chapter 7.4) during thermal impedance measurements.

The copper test structure is plated overall with nickel to improve its corrosion resistance. The surface roughness of the test structure surfaces is better than 10 µm and flatness is better than 30 µm. Also the parallelism of surfaces of the test structure is within this inaccuracy. These properties of the test structure surfaces are important because the thickness of the thin solid materials may be only of the order of 100 µm and the real thickness of greases is lower than this measure.

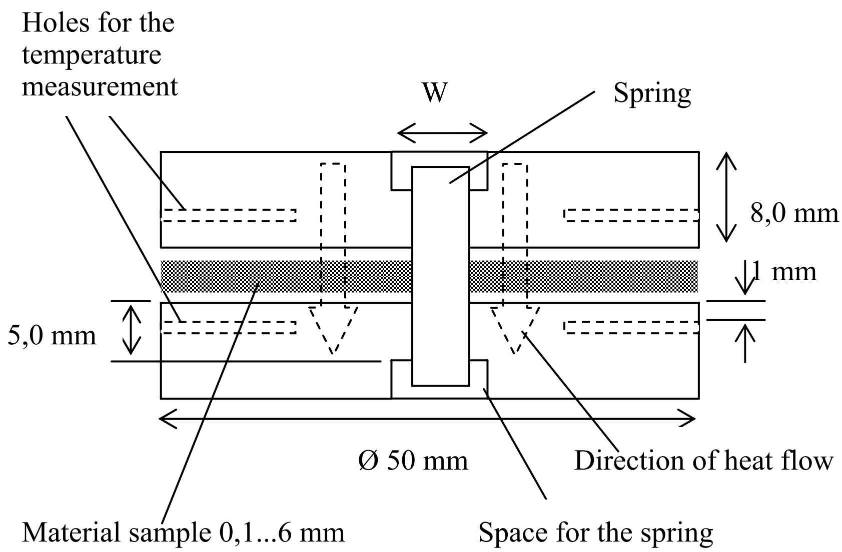


Figure 5.1. The test structure for measurement of the thermal impedance of the thermally conductive materials. The diameter of the structure is 50 mm.

5.2 Preparation of test samples

The solid test samples were cut from the sheets delivered by the material suppliers. The diameter of the material samples was 51 mm to make sure that the sample covers the whole area of test structure; the diameter of the test pill is 50 mm. This sample diameter gives some overlap of the test structure so it is possible to see how the material behaves during tests.

The surfaces of test structures were cleaned up with alcohol to remove all dirt and contamination from the surfaces.

The solid pad materials were installed first on the lower test structure half and were pressed to remove any air bubbles from the interface of material and test structure. Thereafter the guide pins, which keep the structure lower and upper halves on fixed position in relation to each others, were seated. Then the upper structure half was installed and pressed on the lower test structure half.

If the material has protective sheet on the other side then the sheet is removed and the material sample will be assembled on the lower structure half the removed side downwards.

After this the springs are assembled and the test sample is ready for measurements.

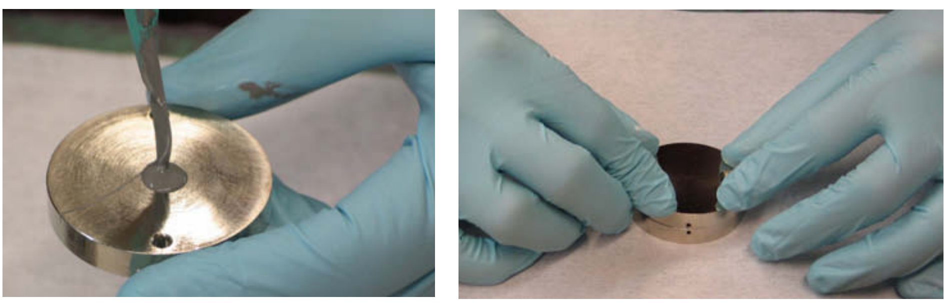


Figure 5.2. Installation of grease-type materials into the test structure.

The installation of grease-type materials into the test structure is done by dropping certain amount of grease on the middle of the lower structure half (Figure 5.2) and thereafter the upper half is put gradually on the lower half.

Thereafter the upper half is moved by circular movements on the grease until the grease comes out on every side of the test structure.

If the grease is very stiff then the grease is installed with the help of some tool on the lower test structure as even as possible. In this case some extra pressure may be needed to remove air bubbles from the interface of grease and test structure halves.

The thickness of grease layer is not known, basically the grease comes out from the interface under pressure until the opposite surfaces touch each others. So the grease fills only voids and uneven areas between the test structure surfaces.

The thicknesses of the material samples may be measured if first the thickness of the bare test structure is measured and thereafter material is installed and then the test structure with installed material is measured. However this requires quite accurate measurement system and is time consuming. Therefore such measurements were not performed in this study.

5.3 Properties of the test samples

For the reliability tests 14 material types were selected from the materials measured in Chapter 4. The amount of samples of every material type is five in reliability tests. The thermal impedance of each sample is measured in reliability tests. Visual inspection is also done during the tests. The numbering and basic properties of each selected material are given in the Table 5.1.

Table 5.1. Numbering and basic properties of the material samples in reliability tests.

Sample numbers	**Material number and type**	**Thermal conductivity**	**Nominal thickness**	**Temperature range**
..	..	λ [W/(m·K)]	[mm]	[ºC]
N1–5	1 Pad	3,0	0,381	-60...+200
N6–10	2 Pad	1,6	0,229	-60...+180
N11–15	43 Pc pad	1,6 [1)]	0,102	...+150
N16–20	47 Pc pad	0,7/75 [2)]	0,089	-60...+125
N21–25	11 Pad	0,8...1,2	0,25	...+110
N26–30	29 Graphite pad	16/120 [3)]	0,13	-35...+125
N31–35	30 Graphite pad	16/120 [3)]	0,13	-35...+125
N36–40	55 Grease	0,9	..	-50...+200
N41–45	56 Grease	3,5	..	-60...+200
N46–50	57 Grease	6,6	..	-60...+200
N51–55	61 Grease	3,8	..	...+150
N56–60	9 Pad	2,07	0,25	-60...+200
N61–62	76 Tape	0,37	0,127	-40...+150
N63–64	69 Tape	0,63	0,23	-40...+150

Pc pad = Phase change pad

5.4 Impedance measurement conditions during tests

The thermal impedance measurement in reliability tests was done by using the following values. Pressure against the test sample is 0,1 MPa nominal value. The absolute pressure varies about ±15% from sample to sample according to the properties of the used springs, thickness variations of the samples and the measurement system accuracy. The variation of pressure from measurement to another measurement of the same sample is much lower because the structures are not opened between tests and during measurements, and the measurements are made always with the same system pressure.

The used nominal power densities for various material types are given in the Table 5.2. Also the approximate values of the material lower side and upper side temperatures (see Figures 7.4 and 7.5) during the impedance measurement are given. The temperature level of materials shifts up and down depending on e.g. the power density and contact pressure used in the measurement system.

Table 5.2. Power density and approximate temperatures over the material and on the lower and upper sides of material during measurement of the thermal impedance (initial).

Sample numbers	**Material number and type**	**Power density in measurements**	**Temperature difference over material**	**Lower side temperature**	**Upper side temperature**
..	..	[W/cm^2]	[K]	[ºC]	[ºC]
N1–5	1 Pad	15	29...39	38...51	73...88
N6–10	2 Pad	10	64...74	30...36	96...108
N11–15	43 Pc pad	15	29...34	35...50	65...80
N16–20	47 Pc pad	20	11...16	50...67	61...81
N21–25	11 Pad	10	43...49	30...39	76...82
N26–30	29 Gr pad	20	15...28	56...63	74...86
N31–35	30 Gr pad	20	17...28	54...61	74...84
N36–40	55 Grease	25	9...17	64...77	76...93
N41–45	56 Grease	25	7...10	70...86	75...92
N46–50	57 Grease	25	6...8	62...79	68...84
N51–55	61 Grease	25	4...6	68...82	74...87
N56–60	9 Pad	10	30...47	29...36	64...83
N61–62	76 Tape	10	44...59	35...38	83...95
N63–64	69 Tape	10	54...57	35...36	90...93

Pc pad = Phase change pad, Gr pad = Graphite pad

5.5 Reliability test program

The test program contains tests described in the Table 5.3. The number of samples is five samples/material type.

Before making any measurements the prepared samples were stabilized at 80 ºC temperature for 12 hours. This was done to make sure that the materials could adapt themselves into the test structure and phase change phenomena could happen through the whole thickness of the material. If phase change materials were measured without preheating then upper part of material had been heated over phase change temperature, but the lower side had not experienced phase change (see Table 5.2).

Every sample is measured in the initial and final inspection (Table 5.3). After every test the three selected samples are measured (to save work only three samples are measured between tests). The samples were kept in test structures all the time from the initial inspection to the final inspection. The structures were visually inspected after every test period. The outer contact surfaces of the copper test structures were checked and cleaned after every test before measurement of the thermal impedance.

Table 5.3. Reliability test program.

Test type	Conditions	Duration	Standard
0 Sample preparation and stabilizing heating	80 ºC	12 h	..
1 Initial inspection	23 ºC	..	..
2 Stability, low temperature	-40 °C	168 h	IEC 60068-2-1 Tests A: Cold
3 Stability, high temperature	+125 °C	500 h	IEC 60068-2-2 Tests B: Dry heat
4 Composite temperature / humidity cyclic test	25/65 °C RH 93% / -10 °C	10•24 h	IEC 60068-2-38 Test Z/AD
5 Change of temperature	-10...+125 10 °C/min	500 cycles á 1 h	IEC 60068-2-14 Modified Test Nb: Change of temperature
6 Change of temperature	-10...+125 10 °C/min	500 cycles á 1 h	IEC 60068-2-14 Modified Test Nb: Change of temperature
Final inspection	23 ºC	..	..

5.6 Initial, cold, dry heat and composite test Z/AD

Figure 5.3 describes the evenness of the measured thermal impedances of the VTT test structure (ref. Figure 7.5). Mostly the back-front temperature differences are less than 10% of the mean temperature difference over the samples (Table 5.4). The pads and tapes have lowest relative back-front differences. Absolutely the back-front temperature differences are quite low in most cases. The mean values are used in all thermal impedance calculations.

The front and back area differ in certain samples quite much, which gives also some idea how even the impedance result of assembly may be in practical cases.

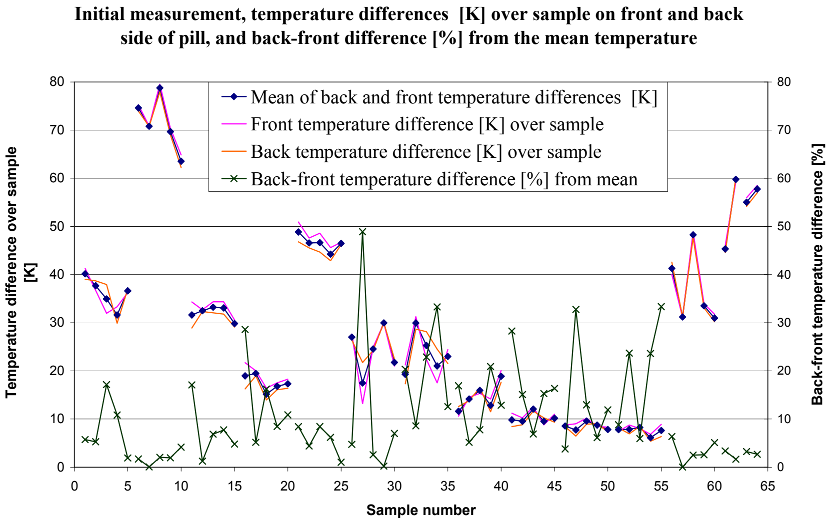


Figure 5.3. Initial measurement, evenness of measured front- and back-area temperatures over the sample in pill.

Table 5.4. Coding of Figure 5.3 sample numbers and material types.

Sample numbers	Material number and type
N1–5	1 Pad
N6–10	2 Pad
N11–15	43 Phase change pad
N16–20	47 Phase change pad
N21–25	11 Pad
N26–30	29 Graphite pad
N31–35	30 Graphite pad

Sample numbers	Material number and type
N36–40	55 Grease
N41–45	56 Grease
N46–50	57 Grease
N51–55	61 Grease
N56–60	9 Pad
N61–62	76 Tape
N63–64	69 Tape

Figure 5.4 contains summary of the thermal impedance values in the initial measurements and after the cold, dry heat and composite IEC Z/AD tests.

The **initial measurements** (Figure 5.4) at VTT show that phase change pads N16–20 and all the greases have lowest thermal impedances, less than 1 K·cm^2/W. The graphite pads N26–30 and N31–35 are the next best.

For reference the thermal impedances measured at ABB by contact pressures of 0,1 and 0,8 MPa are presented in the Figure 5.4 also.

The **initial measurements** (Figure 5.4) at VTT gave almost in every case larger thermal impedance values than those measured at ABB even with pressure 0,1 MPa. The difference is largest with pad material N6–10, which has the largest impedances.

Only the greases N36–40 and N51–55 have lower values of impedance in results of VTT than measured values of ABB at contact pressure of 0,1 MPa.

Basically the initial measurement results are on the same area in measurements of VTT and ABB.

After the **cold test** (Figure 5.4, Tables 5.7 and 5.8) the thermal impedance values have mostly minor decrease in the measured samples. Average change of various materials was between -13...+1%. There were no visible changes in the appearance of the materials in pills. Thus the storage in cold has no remarkable effect on materials.

After the **dry heat test** 500 h at 125 ºC (Figure 5.4, Tables 5.7 and 5.8) most of the measured thermal impedances are lower than after the cold test. This means that the materials are filling the voids and gaps on the surfaces thus the impedance falls, most in those materials, which have highest impedances in initial measurements. The largest changes are with the pads N6–8 and N56–58. The phase change, graphite and grease materials have only minimal changes of thermal impedance after the dry heat test.

After the **composite IEC Z/AD** test (Figure 5.4, Tables 5.7 and 5.8) the thermal impedance values have changed very little compared to the measurement before that test. Only one sample of material No. 57 Grease, has increased its impedance notable.

According to these results every material type tested performed quite well in these tests.

The differences between individual samples of the same type material are larger than the measured changes in these tests. This calls for careful assembly in using these materials. Control of surface properties, contact pressures and faultless layer of material in assembly is necessary for getting same impedance values from item to item.

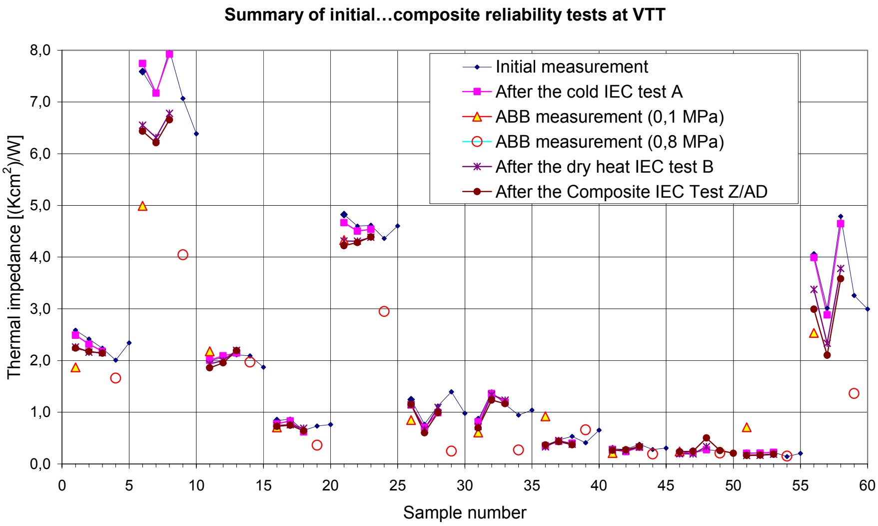


Figure 5.4. Summary of thermal impedances in initial, cold, dry heat and composite IEC Z/AD tests.

According to the results (Figure 5.4, Tables 5.5, 5.7 and 5.8) the individual samples of same material have behaved on the same way through the tests. This reflects the success of the tests and measurement system in these tests.

Table 5.5. Coding of Figures 5.4–5.6 sample numbers, material types, nominal thermal conductivity and measurement of the thermal impedances of these materials by ABB.

Sample numbers	**Material number and type**	**Thermal conductivity**	**Measured thermal impedance** [(Kcm^2)/W]		
		λ [W/(m·K)]	0,1 [MPa]	0,8 [MPa]	Power dens. [W/cm^2]
N1–5	1 Pad	3,0	1,87	1,66	20,9
N6–10	2 Pad	1,6	4,99	4,04	12,3
N11–15	43 Phase change pad	1,6 1)	2,18	1,97	21,9
N16–20	47 Phase change pad	0,7/75 2)	0,71	0,36	24,4
N21–25	11 Pad	0,8...1,2	4,33	2,95	12,6
N26–30	29 Graphite pad	16/120 3)	0,85	0,25	23,4
N31–35	30 Graphite pad	16/120 3)	0,61	0,27	23,7
N36–40	55 Grease	0,9	0,92	0,66	25,0
N41–45	56 Grease	3,5	0,21	0,19	23,4

N46–50	57 Grease	6,6	0,24	0,21	24,0
N51–55	61 Grease	3,8	0,71	0,15	24,1
N56–60	9 Pad	2,07	2,53	1,36	20,0
N61–62	76 Tape	0,37	..	..	..
N63–64	69 Tape	0,63	..	..	..

1) Phase change temperature 55 ºC. 2) Phase change temperature 51...58 ºC, polymer λ=0,7 and carrier λ=75. 3) Through-thickness λ=16 / in-plane λ=120 W/(m·K).

After the **dry heat test** the visual appearance of materials was as described in Table 5.6. See pictures in Table 5.11.

Table 5.6. Visual appearance of the test samples after the dry heat test.

Sample numbers	**Material number and type**	**Visual appearance of material samples after the dry heat test**
N1–5	1 Pad	material slightly deformed and separated from the carrier mesh
N6–10	2 Pad	no change in material
N11–15	43 Phase change pad	material deformed and flows/creeps
N16–20	47 Phase change pad	material deformed and flows/creeps
N21–25	11 Pad	no change in material
N26–30	29 Graphite pad	no change in material
N31–35	30 Graphite pad	no change in material
N36–40	55 Grease	slight creep of material
N41–45	56 Grease	slight creep of material
N46–50	57 Grease	slight creep of material
N51–55	61 Grease	creep of material
N56–60	9 Pad	some separation of carrier mesh
N61–62	76 Tape	no change in material
N63–64	69 Tape	no change in material

5.7 Cyclic change of temperature

This cyclic change of temperature test (-10...+125 ºC, 1 h cycle) was executed in two phases, after the first 500 cycles the samples were measured as well as after the second 500 cycles period. Figures 5.5 and 5.6 contain the average thermal impedances in all the reliability tests.

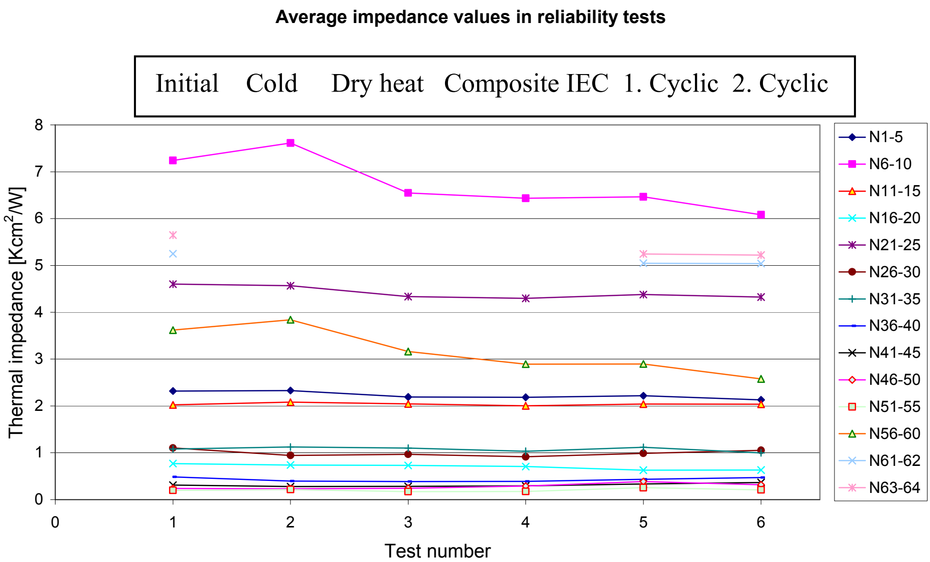


Figure 5.5. Summary of the average thermal impedances in reliability tests.

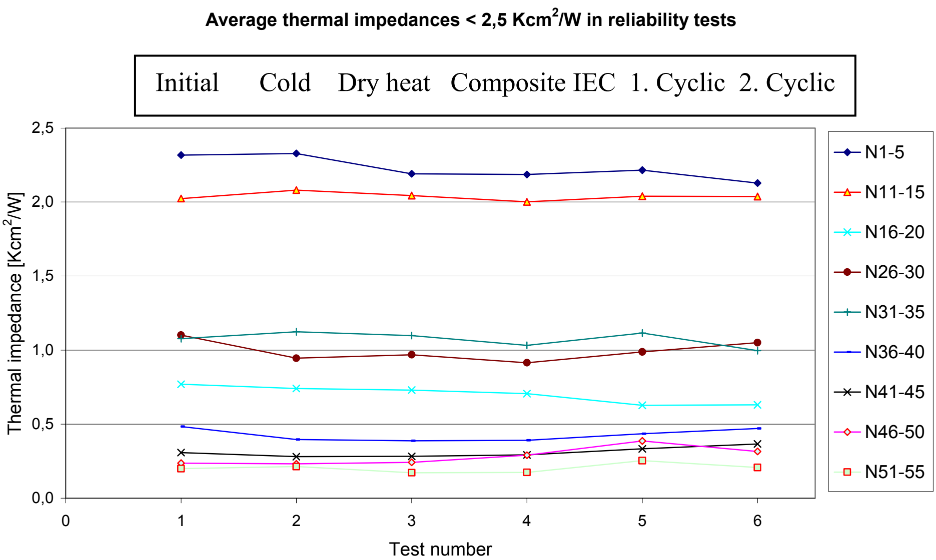


Figure 5.6. Summary of the average thermal impedances < 2,5 K·cm²/W in reliability tests.

Basically the change of temperature during the first 500 cycles of the cyclic temperature change test (test number 5) has caused minor additional changes (Figures 5.5 and 5.6, and Table 5.8) on thermal impedances.

From the Figure 5.6 and Table 5.8 it can be seen that some increase of thermal impedance has happened during the first 500 cycles (test number 5). Exception is material N16–20 “47 Phase change pad”, the impedance is slightly lower than before the test number 5.

After the second 500 cycles of the cyclic temperature change test (test number 6) the thermal impedances have changed very little and are in most cases lower than after the first 500 cycles.

Exception are samples N16–18 of material 47, whose impedances are slightly higher after the second 500 cycles (test number 6).

Final thermal impedances

The average thermal impedances were at the final measurement mostly lower than in the initial measurement (see Table 5.7–5.8, and Figures 5.5–5.6).

Exceptions are greases

– N46–50 ”57 Grease”

– N51–55 ”61 Grease”

whose average value at the end is higher than in the initial measurements.

Table 5.7. Average thermal impedances [K·cm^2/W] in various tests.

Sample s	**1 Initial**	**2 1) Cold**	**3 1) Dry heat**	**4 1) Compos.**	**5 1) 1. Cyclic**	**6 2. Cyclic**	**Final/initial [%]**
N1–5	2,317	2,328	2,192	2,185	2,216	2,129	-8,1
N6–10	7,240	7,614	6,548	6,434	6,464	6,080	-16,0
N11–15	2,024	2,081	2,044	2,001	2,040	2,037	0,7
N16–20	0,769	0,741	0,730	0,706	0,627	0,630	-18,1
N21–25	4,600	4,568	4,334	4,296	4,377	4,324	-6,0
N26–30	1,101	0,946	0,969	0,914	0,988	1,050	-4,6
N31–35	1,077	1,123	1,098	1,033	1,115	0,997	-7,5
N36–40	0,484	0,396	0,387	0,390	0,435	0,470	-2,9
N41–45	0,307	0,280	0,283	0,291	0,334	0,366	18,9 2)
N46–50	0,237	0,231	0,242	0,327	0,386	0,315	33,0
N51–55	0,199	0,214	0,171	0,174	0,254	0,207	3,8
N56–60	3,619	3,839	3,163	2,892	2,897	2,578	-28,8
N61–62	5,249				5,048	5,038	-4,0
N63–64	5,647				5,246	5,220	-7,6

1) Only three samples measured in tests 2...5; in initial and final measurements measured 5 samples/material.

2) One sample has increased the change of the five samples average, see Figures 5.5 and 5.8.

Table 5.8 contains for comparison purposes changes of the average thermal impedances of various materials from test to test. Only the average of the three samples mentioned in the leftmost column are within the figures. So it is easy to see the individual changes of all materials.

Table 5.8. Changes [%] of the average thermal impedances in various tests. Three samples/material only in comparison.

Test	**2/1 Cold**	**3/2 Dry heat**	**4/3 Compos.**	**5/4 1.change**	**6/5 2. change**	**6/1 Final/init**	**6/1 1) Final/init**
Samples	3	3	3	3	3	**3**	5
N1–3	-4	-6	0	1	0	**-8**	-8,1
N6–8	0	-14	-2	0	0	**-15**	-16,0
N11–13	1	-2	-2	2	1	**1**	0,7
N16–18	-6	-1	-3	-11	7	**-15**	-18,1
N21–23	-2	-5	-1	2	-1	**-8**	-6,0
N26–28	-9	2	-6	8	-3	**-8**	-4,6
N31–33	-1	-2	-6	8	-6	**-7**	-7,5
N36–38	-13	-2	1	12	-5	**-9**	-2,9
N41–43	-12	1	3	15	-11	**-7**	18,9 2)
N46–48	-5	5	35	18	-5	**51**	33,0
N51–53	-1	-20	2	45	-5	**12**	3,8
N56–58	-3	-18	-9	0	-5	**-30**	-28,8
N61–62				-4	0	**-4**	-4,0
N63–64				-7	0	**-8**	-7,6

1) Five samples, figures from Table 5.7.

2) One sample has increased the change of the five samples average, see Figures 5.5 and 5.8.

The rightmost column contains the average changes of all the measured five samples from initial to the final measurement.

Basically changes have been quite low. There are some anomalies, which can be seen from the Table 5.8 and Figure 5.9.

Table 5.9 contains data about the variation of measured impedances in the initial and final measurement of the reliability tests. The deviation is calculated as Dev[%] = 100*(Max-Min)/Mean.

The deviation of values is generally larger than those changes of impedance during the tests in Table 5.8. This means that the use of thermally conductive materials requires careful assemblage in production and the design of contact interfaces to reach a reasonable repeatability of production. Also in design of thermal interfaces this large deviation from sample to sample has to be taken care.

Table 5.9. Thermal impedance [K·cm²/W] and its variation in the initial and final measurement.

Test	**Initial**	**Initial**	**Initial**	**Initial**	**Final**	**Final**	**Final**	**Final**
Samples	Mean	Min	Max	**Dev %**	Mean	Min	Max	**Dev %**
N1–5	2,32	2,01	2,59	**25**	2,13	1,81	2,30	**23** *)
N6–10	7,24	6,38	8,00	**22**	6,08	5,48	6,65	**19** *)
N11–15	2,02	1,87	2,11	**12**	2,04	1,85	2,26	**20**
N16–20	0,77	0,65	0,86	**28**	0,63	0,52	0,85	**52**
N21–25	4,60	4,36	4,82	**10**	4,32	4,28	4,41	**3** *)
N26–30	1,10	0,77	1,39	**57**	1,05	0,64	1,47	**80**
N31–35	1,08	0,86	1,39	**50**	1,00	0,70	1,29	**59**
N36–40	0,48	0,36	0,65	**60**	0,47	0,36	0,60	**52** *)
N41–45	0,31	0,28	0,38	**33**	0,37	0,27	0,56	**79**
N46–50	0,24	0,21	0,28	**30**	0,32	0,21	0,58	**117**
N51–55	0,20	0,14	0,23	**42**	0,21	0,13	0,28	**70**
N56–60	3,62	3,00	4,79	**49**	2,58	2,02	3,32	**51** *)
N61–62	5,25	4,50	5,99	**28**	5,04	4,13	5,95	**36**
N63–64	5,65	5,51	5,79	**5**	5,22	5,06	5,38	**6** *)

***)** Final deviation is same or lower than initial deviation.

In many cases after the tests the deviation of impedance values is same or even smaller than in the beginning (6 materials). This means similar behaviour of various samples of the same material in reliability tests.

The largest percentual deviation have graphite and grease materials.

Final visual inspection

The material samples in pills were inspected visually (Tables 5.10 and 5.11) to see any changes in the materials. The materials are only slightly visible (see figures in Table 5.11) outside the pills because the material sample diameter was 51 mm and pill diameter is 50 mm only. The visual changes were quite tiny in the last tests “Change of temperature 1000 cycles”. The appearance was quite similar as after the dry heat test, therefore the wordings are the same. The changes at the end are clearer than after the dry heat test. Certain pads and phase change materials have some material deformation, greases creep a little and tapes have no visual deformation.

Table 5.10. Visual appearance of the test samples after all the tests.

Sample numbers	**Material number and type**	**Visual appearance of material samples after the change of temperature tests (1000 cycles)**
N1–5	1 Pad	material slightly deformed and separated from the carrier mesh
N6–10	2 Pad	no change in material
N11–15	43 Phase change pad	material deformed and flows/creeps
N16–20	47 Phase change pad	material deformed and flows/creeps
N21–25	11 Pad	no change in material
N26–30	29 Graphite pad	no change in material
N31–35	30 Graphite pad	no change in material
N36–40	55 Grease	slight creep of material
N41–45	56 Grease	slight creep of material
N46–50	57 Grease	slight creep of material
N51–55	61 Grease	creep of material
N56–60	9 Pad	some separation of carrier mesh
N61–62	76 Tape	no change in material
N63–64	69 Tape	no change in material

Table 5.11. Photographs of the test samples after the tests.

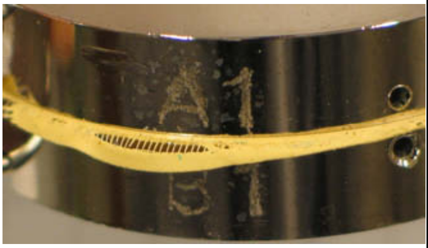
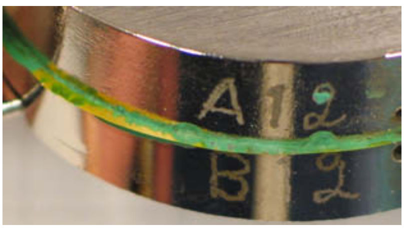
After the dry heat test	**After all tests**
	
	
	
	
	

Table 5.11 continues. Photographs of the test samples after the tests.

<table>
<tr><th>After the dry heat test</th><th>After all tests</th></tr>
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Table 5.11 continues. Photographs of the test samples after the tests.

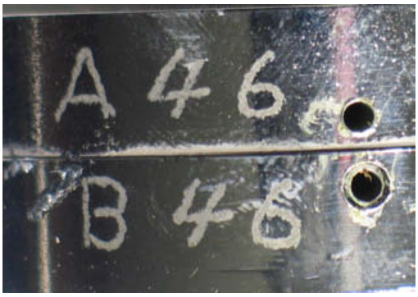
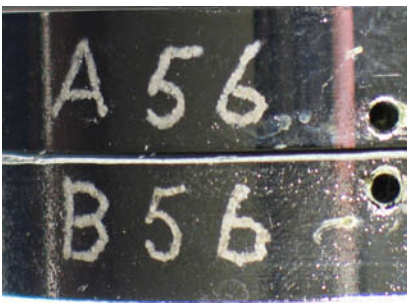
After the dry heat test	**After all tests**
	
	
	
..	

Table 5.11 continues. Photographs of the test samples after the tests.

After the dry heat test	After all tests
..	

5.8 Summary of the reliability tests

The main target of the environmental reliability tests was to see how the thermally conductive materials behave in various environmental conditions and are there some problems in the long term behaviour of the materials.

The measured property of the materials in these reliability tests was thermal impedance in a test structure designed for this purpose. Also visual inspection was used to see possible changes in materials.

Basically all tested materials had quite stable thermal impedance, the average impedance decreased in most cases during the tests. At the end of tests the most values were lower than in the beginning. Only certain greases (see Tables 5.8 and 5.9) had larger final impedances than in the initial measurement. Some samples had exceptionally large changes of the impedance, this emphasises the fact that the variation from sample to sample has been even larger than the changes because of the tests.

All the measured thermal impedances are presented in the Figures 5.7 and 5.8 whose sample coding is given in the Table 5.12.

Table 5.12. Coding of sample numbers and material types in Figures 5.7–5.9.

Sample numbers	Material number and type	Sample numbers	Material number and type
N1–5	1 Pad	N36–40	55 Grease
N6–10	2 Pad	N41–45	56 Grease
N11–15	43 Phase change pad	N46–50	57 Grease
N16–20	47 Phase change pad	N51–55	61 Grease
N21–25	11 Pad	N56–60	9 Pad
N26–30	29 Graphite pad	N61–62	76 Tape
N31–35	30 Graphite pad	N63–64	69 Tape

According to the visual inspection (see figures in the Table 5.11) certain pads and phase change materials have some material deformation, greases creep a little and tapes have no visual deformation.

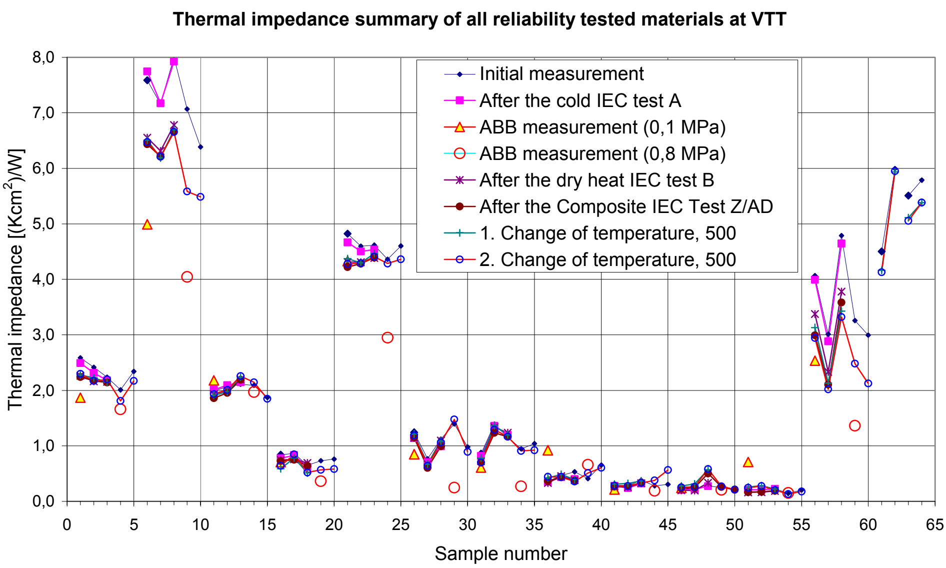


Figure 5.7. Summary of all the thermal impedance measurements after tests.

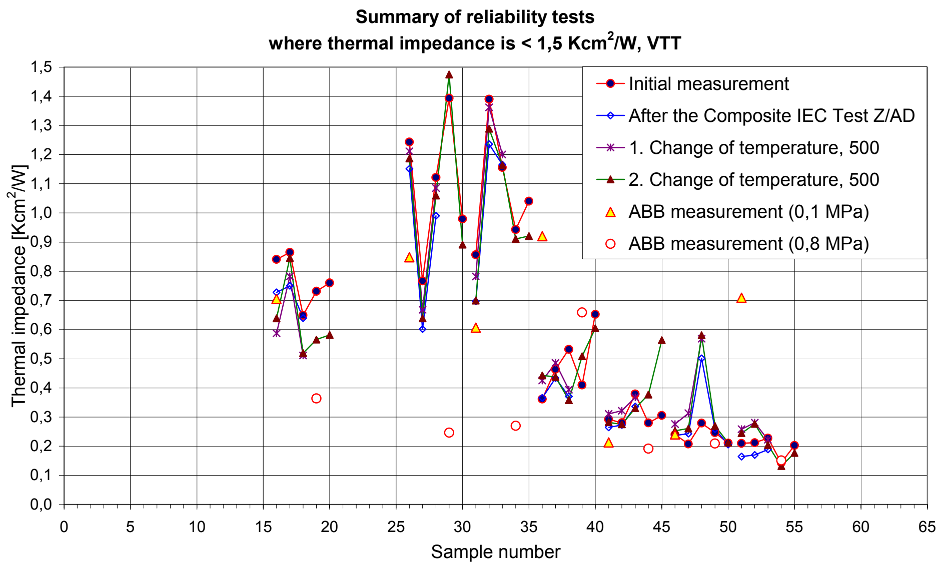


Figure 5.8. Summary of the measurements where thermal impedance is < 1,5 K·cm²/W.

Figure 5.9 contains the calculated percentual changes of thermal impedances.

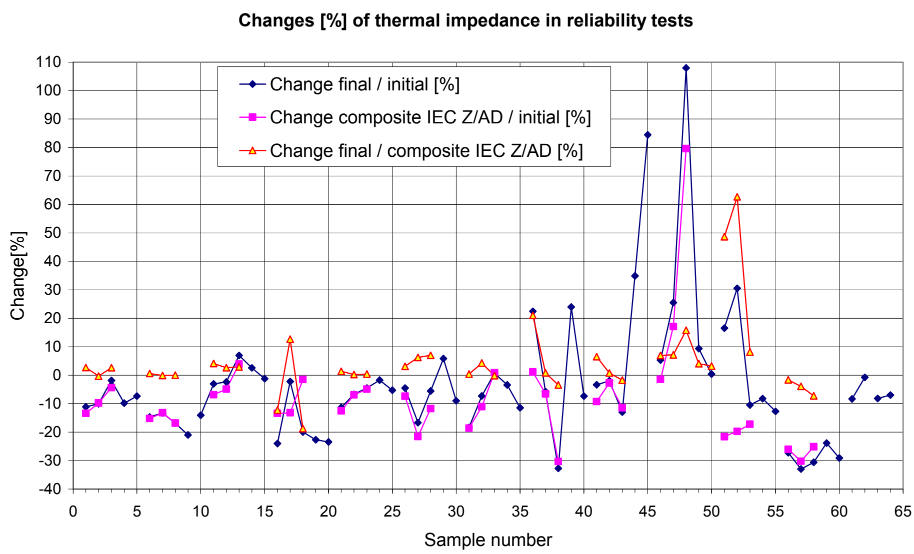


Figure 5.9. Summary of changes of the measured thermal impedances in reliability tests.

Figure 5.9 contains three series of changes of the measured thermal impedances:

1. change from the initial to the end of composite IEC Z/AD test
2. change during the temperature cycling tests (1000 cycles) final/composite test
3. change from the initial to final measurement.

All the values are percentual therefore the materials, e.g. greases, with the lowest initial impedance values (< 0,5 K·cm^2/W, see Table 5.7) may have large relative changes. Also the inaccuracy of the measurement of the lowest impedances may have some effect on the results of the best heat conductors (greases).

The first series shows that the largest change has happened with most materials before the cyclic tests e.g. in the cold -40 ºC and dry heat 500 h test at 125 ºC, and in the composite IEC Z/AD test. The common trend is lower impedance after these tests. An exception is material N46–48 No. 57 Grease, whose impedance increased.

The second series describes changes during the cyclic temperature change tests (1000 cycles). In this test almost every material has got larger impedance values during the first 500 cycles. During the second 500 cycles most impedance values have again decreased! The total change in most cases during the 500+500 cycles has been to larger values of impedance. Exceptions are samples N16–18 and N56–58 whose mean impedance is lower after tests.

The third series describes the total change from the beginning to the end of the reliability test cycle. Because impedance values are mostly below the initial values this emphasises the common behaviour of the thermally conductive materials; it takes quite much time (together with high temperature) to the materials to adapt themselves between the heat source and cooling surface.

When certain individual sample has exceptionally different change than the other samples of the same material then this may describe difficulties in the assemblage of material in proper way between the heat source and cooling surface.

5.9 Conclusions of the reliability tests

The reliability tests were environmental tests (cold, heat, humidity and temperature cycling), which affect on the thermal and other physical properties of the thermal interface materials.

The main criteria for evaluation of materials was the measured thermal impedance (K·cm^2/W) at power densities from 10 to 25 W/cm^2. The high power density means that samples are heated during the measurements into the high operating temperature.

All the test samples were preconditioned 12 hours at 80 ºC temperature to get same kind of initial state for the materials.

The cold conditioning at -40 ºC had quite little effect on thermal impedance, thus storage in cold does not cause problems. Impedances after the cold test were generally lower than initial.

The storage in high temperature, 500 hours at 125 ºC, decreased the measured impedances of the material samples about same amount but different materials as the cold test. The largest percentual changes are with the pads and one grease. The phase change, graphite and most of grease materials have only minimal changes of thermal impedance after the dry heat test. This means that the materials are filling the voids and gaps on the surfaces thus the impedance falls, most in those materials, which have highest impedances in initial measurements.

The composite dry heat / humidity / cold test IEC Z/AD changed a little less than the dry heat storage the thermal impedances. The main trend was decrease of thermal impedance.

As a summary the most of the changes of thermal impedance happened in the above mentioned tests.

The cyclic change of temperature -10...+125 ºC, 1000 cycles caused in the first phase mostly increase of impedances but during the second phase the trend was again slight decrease of the thermal impedance.

As a final conclusion about the thermal impedances of the materials tested most of the materials had decreasing trend of impedance through the reliability tests. Some increasing trend of impedance was visible during the cyclic test, but every material type succeeded well in the tests.

The visual inspections showed some changes in the open edges of the pads and separation of phase change material from the carrier mesh, and some creep of greases. These have probably no significant harmful effect in use.

The long term behaviour of the tested materials seems to be quite good, however the cyclic test should be continued to over 5000 cycles to get better confidence for the long life products.

Quite important observation of the tests is that the variation from sample to sample in initial and final measurements is larger than the changes during tests. The final percentual variation is about 6...120% from the minimum to maximum values depending on the material type. Therefore the user should take care of this while calculating the total variation of thermal impedance for the whole life cycle of products.

6. Mechanical properties and usability of materials

All the materials tested in this project are designed for use at high maximum power densities of 10...30 W/cm^2. Therefore the material thickness of tested materials is low, 0,1...0,4 mm. Greases have even lower thickness in use, about 0,03...0,06 mm. Tables 6.1–6.14 contain the information of the materials.

Table 6.1. Mechanical and use properties of material samples N1–5.

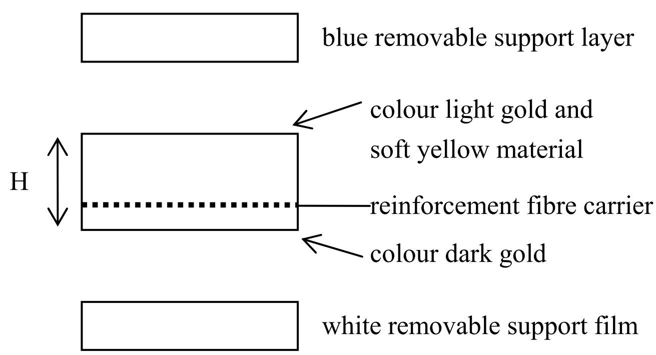
Type and code	No. 1 Pad: N1–5, tested at 15 W/cm^2
Structure and thickness	3-layer structure, thickness H = 0,381 (...3,175) mm  Soft, one side tack agent on reinforcement side
Configurations	Available as sheet, die-cut parts or roll form
Basic application	Medium thickness gaps, requires clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove white support layer, attach to the first contact surface. – Remove blue support layer, attach to the second contact surface. – Fix the clamping device. Yellow thermally conductive material is mechanically tough, soft, and flexible; endures well handling. Removing support layers requires special skills.
Reliability test properties	Free areas of material are slightly deformed and separated from the carrier mesh.
Rework	Material adheres to the contact surfaces, and needs both mechanical removal and chemical cleaning.
Electrical	Insulating material

Table 6.2. Mechanical and use properties of material samples N6–10.

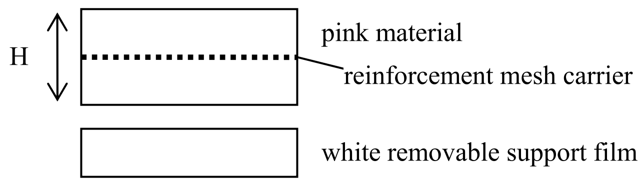
Type and code	No. 2 Pad: N6–10, tested at 10 W/cm^2
Structure and thickness	 2-layer structure, thickness H = 0,229 mm Soft material, one side tack agent on support film side
Configurations	Available as sheet, die-cut parts or roll form, with or without pressure sensitive adhesive
Basic application	Low thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove white support layer, attach to the first contact surface. – Attach to the second contact surface. – Fix the clamping device. Pink thermally conductive material is mechanically tough, soft, and flexible; endures well handling. Removing support film do not require special skills.
Reliability test properties	No change of material appearance.
Rework	Material adheres slightly to the contact surfaces, and needs both mechanical removal and chemical cleaning.
Electrical	Insulating material

Table 6.3. Mechanical and use properties of material samples N11–15.

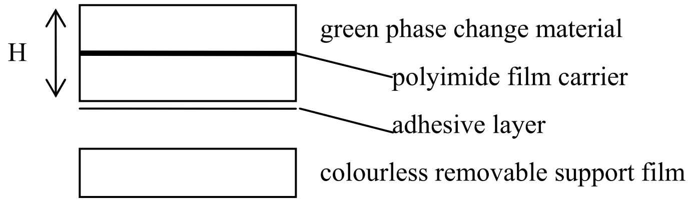
Type and code	No. 43 Phase change pad: N11–15, tested at 15 W/cm^2
Structure and thickness	 2-layer structure, thickness H = 0,102 mm Soft material, one side tack agent on support layer side
Configurations	Available as sheet, die-cut parts or roll form
Basic application	Low thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove colourless support film, then attach to the first contact surface. – Attach to the second contact surface. – Fix the clamping device. Green thermally conductive material with polyimide carrier is mechanically tough, and flexible; endures well handling. Removing support film requires special skills to preserve the adhesive layer.
Reliability test properties	No change of material appearance.
Rework	Material adheres tightly to the contact surfaces, and needs both mechanical removal and chemical cleaning.
Electrical	Insulating material

Table 6.4. Mechanical and use properties of material samples N16–20.

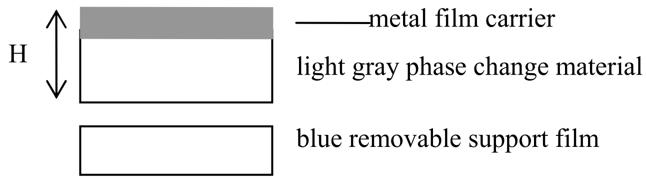
Type and code	No. 47 Phase change pad: N16–20, tested at 20 W/cm^2
Structure and thickness	 2-layer structure, thickness H = 0,089 (/ 0,15) mm Soft material, on one side (light grey polymer film) inherently tacky phase change material
Configurations	Available as sheet or die-cut parts
Basic application	Very low thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets. – First remove blue support film, then attach to the first contact surface. – Attach to the second contact surface. – Fix the clamping device. Gray thermally conductive material with metal carrier film is mechanically rather tough and flexible; endures handling. Removing support film requires good skills to preserve the thin metal/phase change material.
Reliability test properties	Material deforms and flows/creeps a little.
Rework	Material adheres on the phase change side to the contact surface, and needs both mechanical removal and chemical cleaning (e.g. isopropyl alcohol solvent). Metal side removes easily.
Electrical	Non insulating material

Table 6.5. Mechanical and use properties of material samples N21–25.

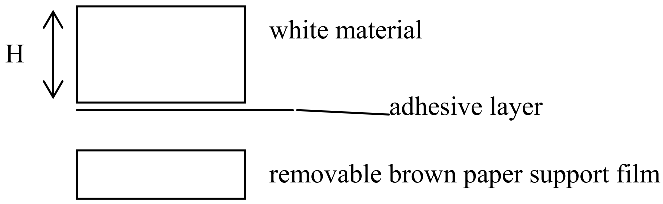
Type and code	No. 11 Pad: N21–25, tested at 10 W/cm^2
Structure and thickness	 2-layer structure, thickness H = 0,25 mm Soft material, on one side tacky adhesive material
Configurations	Available as sheet, die-cut parts or rolls
Basic application	High thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets. – First remove brown paper support film, then attach to the first contact surface. – Attach to the second contact surface. – Fix the clamping device. White thermally conductive material is mechanically rather tough and flexible; endures handling. Removing support film is easy.
Reliability test properties	No change of material appearance.
Rework	Material adheres on the tacky side to the contact surface, and needs both mechanical removal and chemical cleaning. Other side removes easily.
Electrical	Insulating material.

Table 6.6. Mechanical and use properties of material samples N26–30.

Type and code	No. 29 Graphite pad: N26–30, tested at 20 W/cm^2
Structure and thickness	 gray material, graphite and polymer 1-layer structure, thickness H = 0,13 mm Soft and brittle material, no tacky adhesive nor support film in tested material.
Configurations	Available as sheet, die-cut parts or rolls, with or without pressure sensitive adhesive
Basic application	Low thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – Attach to the first contact surface. *) – Attach to the second contact surface. – Fix the clamping device. The graphite material is mechanically very brittle, therefore handling requires special skills.
Reliability test properties	No change of material appearance.
Rework	Material adheres on the contact surfaces, and needs both mechanical removal and chemical cleaning. See material N31–35.
Electrical	Electrically conductive.

*) Use the following procedure to apply these materials (N26–30 and N31–35) if there is adhesive on it:

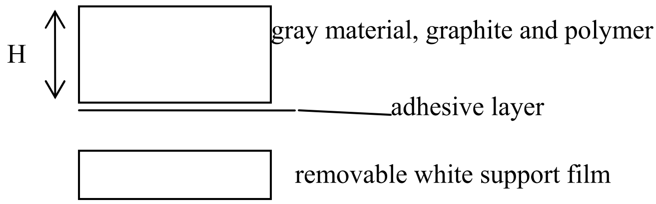
1 The mounting surface should be clean and dry prior to application. Clean the mounting surface with isopropyl alcohol applied with a lint-free wipe or swab.

2 The backing paper should be removed by peeling it away at an angle from the material. Keep the material flat while doing this. Peeling the material from the backing paper while keeping the backing paper flat will result in creasing and damage to the thermal interface materials.

3 Apply the material to the mounting surface at slight angle using finger pressure to reduce the potential for air entrapment.

Once bonded to a surface, adhesive backed materials cannot be repositioned.

Table 6.7. Mechanical and use properties of material samples N31–35.

Type and code	No. 30 Graphite pad: N31–35, tested at 20 W/cm^2
Structure and thickness	 2-layer structure, thickness H = 0,13 mm Soft and brittle material, tacky adhesive film on other side.
Configurations	Available as sheet, die-cut parts or rolls, with or without pressure sensitive adhesive
Basic application	Low thickness gaps, medium pressure requires spring type clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove white support film, then attach to the first contact surface (see N26–30). – Attach to the second contact surface. – Fix the clamping device. The graphite material is mechanically very brittle, therefore handling requires special skills. The removing of support film may brake the graphite film, because the adhesive may remove pieces of graphite.
Reliability test properties	No change of material appearance.
Rework	Material adheres on the contact surfaces especially on the tacky side, and needs both mechanical removal and chemical cleaning. The recommended method by the manufacturer for removing material is the following: 1 Apply the adhesive side of a high tack adhesive tape to the material. Press tape firmly against the material and pull away. Some material will transfer onto the tape. Repeat 4–5 times using fresh portion of tape in each application. When most of the mounting surface is exposed, stop. 2 Apply isopropyl alcohol to the remaining material and adhesive and wait 1 minute. 3 Use wooden or plastic scraper to push the material off the surface. Gather up the loose material using wiping cloth. Then soak a cotton tipped swabbing stick in isopropyl alcohol and use it to remove any lose material or adhesive. Some mild scrubbing may be necessary. 4 Perform final cleaning of the mounting surface with isopropyl alcohol applied with a lint-free wipe or swab.
Electrical	Electrically conductive.

The recommended method by the manufacturer for removing N26–30 and N31–35 means that removing of the material from interface surfaces is time consuming and requires some skills.

Table 6.8. Mechanical and use properties of material samples N36–40.

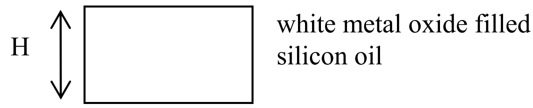
Type and code	No. 55 Grease: N36–40, tested at 25 W/cm^2
Structure and thickness	 1-layer structure, thickness H ≈ 0,03...0,06 mm Soft and low viscosity material, like grease.
Configurations	Available in syringes and bulk package.
Basic application	Low thickness gaps, medium pressure requires clamping device for pressure.
Handling in production	– First dispense material on the first contact surface. – Spread the material with proper tool e.g. roller on the first contact. – Attach to the second contact surface. – Fix the clamping device. The grease type material stains easily hands and tools, to avoid this special protection and skill is needed.
Reliability test properties	Slight creep of material.
Rework	Material adheres on the contact surfaces, opening of the interface may be difficult because the thickness is so low, and needs both mechanical removal and chemical cleaning.
Electrical	Contact is electrically conductive although the material itself is insulating.

Table 6.9. Mechanical and use properties of material samples N41–45.

Type and code	No. 56 Grease: N41–45, tested at 25 W/cm^2
Structure and thickness	 grey ceramic filled single component silicon 1-layer structure, thickness H ≈ 0,03...0,06 mm Soft and low viscosity material, grease.
Configurations	Available in bulk package.
Basic application	Low thickness gaps, medium pressure requires clamping device for pressure.
Handling in production	– First dispense material on the first contact surface. – Spread the material with proper tool e.g. roller on the first contact surface. – Attach to the second contact surface. – Fix the clamping device. The grease type material stains easily hands and tools, to avoid this special protection and skill is needed.
Reliability test properties	Slight creep of material.
Rework	Material adheres on the contact surfaces, opening of the interface may be difficult because the thickness is so low, and needs both mechanical removal and chemical cleaning.
Electrical	Contact is electrically conductive although the material itself is insulating.

Table 6.10. Mechanical and use properties of material samples N46–50.

Type and code	No. 57 Grease: N46–50, tested at 25 W/cm^2
Structure and thickness	 grey ceramic filled single component silicon 1-layer structure, thickness H ≈ 0,03...0,06 mm Soft and low viscosity material, grease.
Configurations	Available in bulk package.
Basic application	Low thickness gaps, medium pressure requires clamping device for pressure.
Handling in production	– First dispense material on the first contact surface. – Spread the material with proper tool e.g. roller on the first contact surface. – Attach to the second contact surface. – Fix the clamping device. The grease type material stains easily hands and tools, to avoid this special protection and skill is needed.
Reliability test properties	Slight creep of material.
Rework	Material adheres on the contact surfaces, opening of the interface may be difficult because the thickness is so low, and needs both mechanical removal and chemical cleaning.
Electrical	Contact is electrically conductive although the material itself is insulating.

Table 6.11. Mechanical and use properties of material samples N51–55.

Type and code	No. 61 Grease: N51–55, tested at 25 W/cm^2
Structure and thickness	 white silicon-free thermal grease 1-layer structure, thickness H ≈ 0,03...0,06 mm Soft and low viscosity material, grease.
Configurations	Available in bulk package.
Basic application	Low thickness gaps, medium pressure requires clamping device for pressure.
Handling in production	– First dispense material on the first contact surface. – Spread the material with proper tool e.g. roller on the first contact surface. – Attach to the second contact surface. – Fix the clamping device. Grease is screen printable also. The grease type material stains easily hands and tools, to avoid this special protection and skill is needed.
Reliability test properties	Creep of material.
Rework	Material adheres on the contact surfaces, opening of the interface may be difficult because the thickness is so low, and needs both mechanical removal and chemical cleaning.
Electrical	Contact is electrically conductive although the material itself is insulating.

Table 6.12. Mechanical and use properties of material samples N56–60.

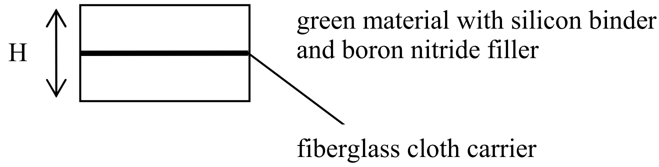
Type and code	No. 9 Pad: N56–60, tested at 10 W/cm^2
Structure and thickness	 1-layer structure, thickness H = 0,25 mm Soft material, on one side pressure sensitive adhesive available.
Configurations	Available as sheet or die-cut parts.
Basic application	Low thickness gaps, high pressure (2...3,5 MPa) requires mechanical clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets. – First attach to the first contact surface. – Attach to the second contact surface. – Fix the clamping device. Green thermally conductive material with fiberglass carrier is mechanically tough, and flexible; endures well handling. No protective film.
Reliability test properties	Some separation of carrier cloth in open areas.
Rework	Material adheres to the contact surfaces, and needs both mechanical removal and chemical cleaning.
Electrical	Insulating material

Table 6.13. Mechanical and use properties of material samples N61–62.

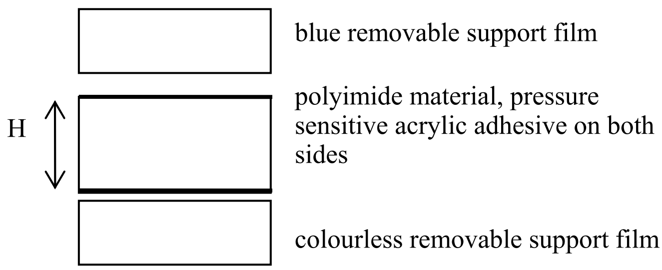
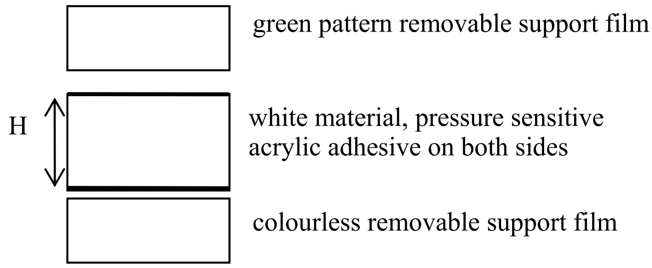
Type and code	No. 76 Tape: N61–62, tested at 10 W/cm^2
Structure and thickness	 3-layer structure, thickness H = 0,127 mm Pressure sensitive acrylic adhesive on both sides.
Configurations	Available as sheet, die-cut parts or roll form
Basic application	Low thickness gaps, does not require clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove colourless support layer, attach to the first contact surface. – Remove blue support layer, attach to the second contact surface. Beige thermally conductive tape is mechanically tough and flexible; endures well handling. Removing support layers requires special skills.
Reliability test properties	No change of material.
Rework	Material adheres well to the contact surfaces, and needs both mechanical removal (use of razor blade and spatula) and chemical cleaning (isopropyl alcohol, toluene or methyl ethyl ketone, MEK).
Electrical	Good electrical isolation.

Table 6.14. Mechanical and use properties of material samples N63–64.

Type and code	No. 69 Tape: N63–64, tested at 10 W/cm^2
Structure and thickness	 3-layer structure, thickness H = 0,23 mm Pressure sensitive acrylic adhesive on both sides.
Configurations	Available as sheet, die-cut parts or roll form
Basic application	Low thickness gaps, does not require clamping device for pressure.
Handling in production	Precutting of material when delivered as sheets or rolls. – First remove colourless support layer, attach to the first contact surface. – Remove green pattern support layer, attach to the second contact surface. White thermally conductive tape is mechanically rather tough and flexible; tape stretches easily, therefore handle carefully. Removing support layers requires special skills.
Reliability test properties	No change of material.
Rework	Material adheres well to the contact surfaces, and needs both mechanical removal and chemical cleaning.
Electrical	Good electrical isolation.

7. Measurement methods

7.1 ASTM D5470-01 method

The most used standard for measurement of thermal impedance is the ASTM D5470-01 Standard Test Method for Thermal Transmission Properties of Thin Thermally Conductive Solid Electrical Insulation Materials, December 2001.

The thermal impedance is measured with a steady state method where temperature over the material, pressure and heat flux are measured. Also the thickness of material has to be measured before the impedance measurement. The standard pressure is 3,0 ±0,1 MPa. Recommended mean temperature of the sample is 50 ºC.

This method is criticised because the used pressure 3 MPa is far over the practical pressures available in user applications where pressures only of 0,1 MPa or even less is usual. Also the repeatability of the method within laboratories has been about 20%, which is too much [Lasance 2003].

Still this method is the only generally accepted method for measurement of thermal impedance of materials.

7.2 Thermal conductivity, Hot Disk method

The Hot Disk method is an experimental technique developed from the concept introduced by the Transient Hot Strip (THS) technique (http://www.hotdisk.se/).

The Hot Disk sensor (Figure 7.1) consists of an electrically conducting pattern in the form of a double spiral, which has been etched out of a thin metal foil. The spiral is sandwiched between two thin sheets of insulating materials, functioning both as mechanical support and electrical insulation. The sensor acts both as a heat source and temperature probe.

The plane Hot Disk sensor is placed between two pieces of the sample material and is then heated by an electrical current for a short period of time. The dissipated heat generates temperature rise of both the sensor and the surrounding sample material. The average transient temperature increase of the sensor, of the order 0,5...5 K, is measured by recording the change in electrical resistance. The Temperature Coefficient of Resistivity (TCR) of the sensor material correlates the change in resistivity with the corresponding change in temperature.

The transient temperature increase in the sensor is recorded throughout the pulse duration by following the resistance increase of the double spiral and knowing the temperature coefficient of resistivity of nickel. By comparing the recorded transient temperature increase with that of the theoretical solution of the thermal conductivity equation it is possible to deduce the thermal transport properties of the sample material.

The time duration of the transient recording is normally limited by the size of the sample. In order to avoid influence from outside boundaries of the sample, the sample should be larger than the sensor diameter to ensure stable values of both thermal conductivity and diffusivity.

Figure 7.1. Hot Disk sensor.

Basic method

In the Basic method (Figure 7.2) the sensor is placed between two sample pieces, which are a least as thick as the radius of the sensor. The theory of the Basic method is based on the assumption that the sensor is placed in an infinite medium. This means that the "thermal wave" must not reach the outside boundaries of the same pieces during the pulse duration.

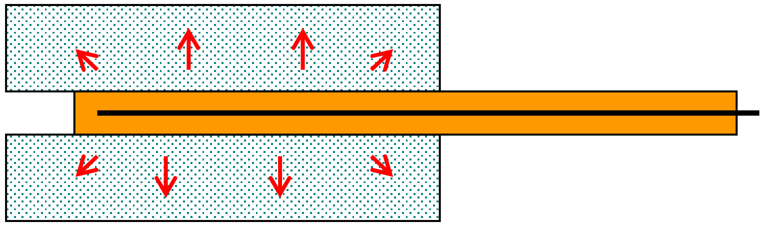


Figure 7.2. Hot Disk sensor operation.

Minimum size is a sample piece of thickness 1,5...2 mm.

Thermal conductivity range: 0,005 to 500 W/(m·K).
Reproducibility: thermal conductivity +/- 2%, thermal diffusivity +/- 5%, specific heat (per unit volume) +/- 7%.

Thin film method

The basic Hot Disk technique has been extended to thermal conductivity studies of thin films. The thickness of the films that can be studied ranges approximately from 20 to 600 micrometers.

Thermal conductivity range: 0,05 to 2 W/(m·K).
Reproducibility: thermal conductivity +/- 3%.

A specially designed sensor is sandwiched between two thin film samples and the whole assembly is clamped between two pieces of stainless steel. The measurement is performed and analysed in exactly the same way as in a basic Hot Disk measurement, and the results should give the thermal transport properties of the stainless steel. However, it is possible to extract the temperature gradient over the thin film with good accuracy. With information about this temperature gradient, the output of power, the surface area and the thickness of the sample films, the thermal conductivity of the thin film can be determined.

When performing measurements with the thin film sensors, a temperature difference develops over the insulating layer before any heating of the sample surface starts. This can be clearly seen in the recorded transient temperature increase from a measurement on a high conducting material with the basic option.

This temperature difference becomes constant after a very short time and it is also easy to estimate this temperature difference in an experiment. This observation makes it possible to measure the thermal conductivity of thin films deposited on or placed in contact with a sample material with a high thermal conductivity (10 W/(m·K) or higher). Measurements on thin films with a thickness from tens to hundreds of micrometers and a thermal conductivity less than 2 W/(m·K) are possible. Materials studied in this way include thin polymer films, deposited materials like surface barrier coatings, fabrics etc. The measurements are performed by locating the thin films between the high conducting sample pieces and a specially designed sensor having a well defined area.

7.3 Thermal impedance, ABB ASTM

The apparatus of ABB performs thermal impedance measurements according to the standard ASTM D5470-01. The pressure over sample is controllable over 0,1...0,8 MPa range. The sample diameter is 100 mm, and heat flow is 2200 W maximum thus giving maximum power density of 28 W/cm^2 (Figure 7.3).

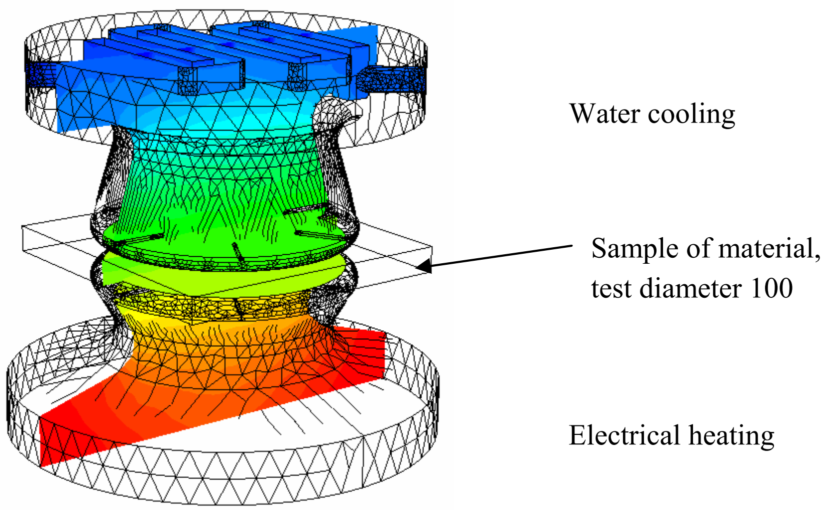


Figure 7.3. Equipment for thermal impedance measurements at ABB.

The temperatures are measured from six different places on both sides of the material sample. The calculated impedance values are mean of these six values.

This makes it possible to evaluate the area evenness of material samples and the quality of installation of the material on surfaces. The impedances are measured as a function of contact pressure. The pressure is continuously controllable, the pressures used in the test were 0,1 / 0,2 / 0,4 / 0,8 MPa.

The surface roughness is Ra = 0,4 µm. The material of the heat conducting bars is copper and the surface against the material sample has hard chrome plating. The heating resistors are in the lower bar and cooling is done with water in the upper bar.

7.4 Thermal impedance, VTT reliability tests

The apparatus of VTT performs thermal impedance measurements following the principles of the standard ASTM D5470-01. The heat flow is kept steady during the measurement and temperature differences over the material under measurement are measured.

The very first goals of the measurement system was to make it possible to

- test materials in various environmental conditions
- measure the same samples repeatedly between the reliability tests
- keep the mechanical and thermal conditions of the materials under test constant.

These goals were achieved by installation of the material samples between the test structure halves or pills (nickel-plated copper) as described in Figure 5.1 and keeping them together with specially designed springs, which have the force specified. By this way the pressure over samples is practically constant in the tests and during the measurements. Naturally there is some variation of pressure between samples and in varies situations, but these should be less than 15% absolute. In the repeated measurements of the same sample this variation is near 2% of the nominal pressure.

The pressure over sample is continuously controllable over the 0,02...0,2 MPa range. The sample diameter is 50 mm, and heat flow is 600 W maximum thus allowing about 30 W/cm^2 power density in measurements. Depending on the thermal impedance of the material sample, the temperature over material may vary approximately from 5 K to 100 K. The principal structure of the measurement system is in Figures 7.4 and 7.5. The maximum surface temperature of measured samples is limited to 150 ºC. The extra thermal contact material on heater and cooler surfaces improve contacts to the test structure (pills).

The pressure in impedance measurement over sample may be controlled with two ways:

- by using springs
- with static gravity force of weights.

The main part of the pressure is produced with springs, which were designed to have a 0,1 MPa pressure over the material samples in this test structure (Figure 5.1). The spring force changes 7,4 %/0,10 mm change in the gap around the nominal force (or change of thickness of the material under measurement). The weight of the heating system and extra weights in the measuring equipment was 10,1 kg thus giving a constant pressure of 0,05 MPa on the test structure. This additional pressure is necessary to get good thermal contact from heater and cooler to the test structure. Because the samples are spring-loaded all the time then during the impedance measurement the real pressure over material samples is ~ 0,15 MPa. Actually every material type has its own pressure, which is almost constant from sample to sample and constant from measurement to measurement.

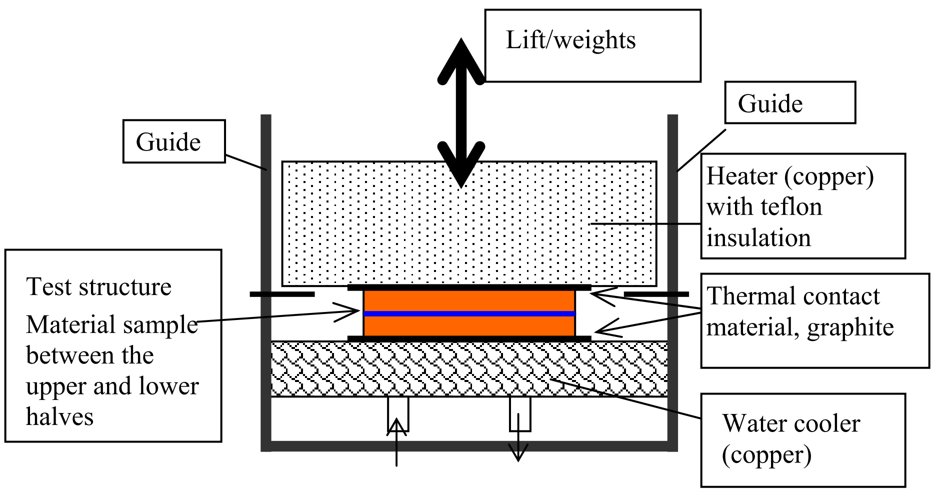


Figure 7.4. Thermal impedance measurement system in reliability tests.

The measured temperature values are taken as mean values of the front and back side temperature differences over the material sample in the pill. These values are thereafter corrected to take care of varies failure sources of the impedance measurement system and pills.

T_0–T_1 is the front side temperature difference over sample.
T_2–T_3 is the back side temperature difference over sample.

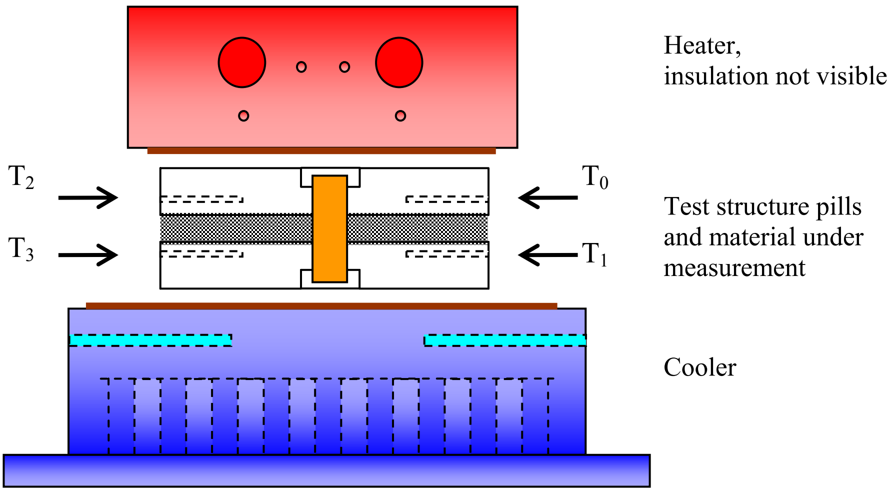


Figure 7.5. Inner structure of the impedance measurement system. The temperature measurement points over the material under measurement are T_0...T_3.

The temperatures on the hot and cool side of the material sample are measured from the holes (T_0...T_3), which are 1,0 mm from the sample surfaces (Figures 7.5 and 5.1). In handling of results the temperature difference from the holes to the pill surface is taken away from the results.

The electrical power is controlled and measured with power supply at ±1% inaccuracy (HP 6813A 300V/1750 VA AC Power source/analyzer). The heater contains two similar resistors and supplies maximum power of 600 W. Water supply for cooling is only 2 L/min. During impedance measurement temperature is measured from 14 points, four points from the test structure (T_0...T_3) and others inside and outside of heater and cooler (see Figure 7.6). Thus the heat flow can be checked and corrected in calculation of measured impedance values. The pills are placed into the test system with < 0,2 mm lateral inaccuracy.

The data logger is HP VXI connected to HP E1347A 16 channel thermocouple relay mux. The temperatures are measured with K-type thermocouples.

The time constant of the measurement system is about 5 min thus one impedance measurement is done in about 30 min.

The power/heat flow is selected before measurements individually for every material type. The used power densities have been 10, 15, 20 and 25 W/cm^2.

The maximum heater surface temperature is about 150 ºC and the minimum cooler surface temperature is about 10 ºC.

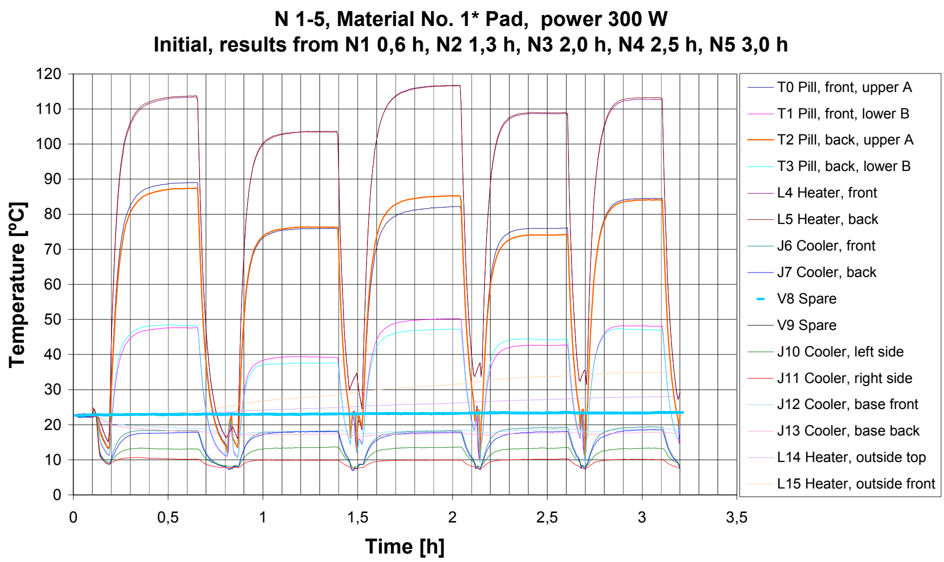


Figure 7.6. Typical temperature curves of the impedance measurement system at VTT.

The values $T_0...T_3$ (see Figure 7.5) in Figure 7.6 are used for calculation of thermal impedance values. We can see from this figure that the separate samples N1...N5 have different impedances. The times, which have been selected for result calculation are mentioned in the heading of Figure 7.6. We can see that individual samples have different properties in the evenness of impedance because the temperature measured from front area and back area of the test structure differ from each others on various samples. Also the variation of contacts on the top and bottom of the pills have some effect.

8. Specification model

The proper specification of thermally conductive materials is a natural requirement for purchase and design of electronics. These materials have important role in heat management of electronics. The designer of electronics and manufacturer of materials need a common language to understand each others, therefore some common specification model is needed.

The specifications needed differ from material type to another but, all the parameters listed here are not relevant to every material type. The detailed descriptions shall be as practical as possible to help users in understanding the properties of materials.

This specification model is grouped into the basic areas as given in the following tables (Tables 8.1–8.7). The tables give only examples of the contents of material specification on common level, the manufacturer shall select the relevant properties, and shall describe the properties more detailed when relevant. See names of the standards in Appendix 1.

*Table 8.1. Structure, material and application area of thermal interface **pad** material.*

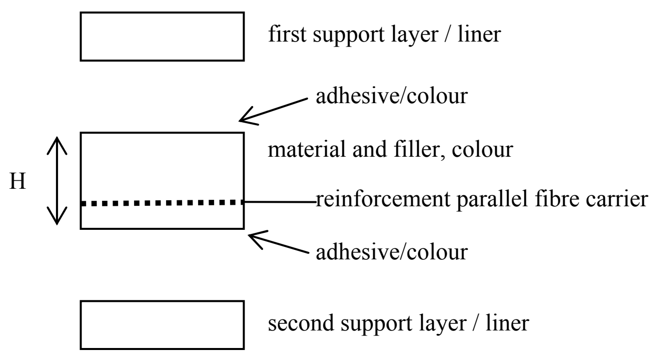
Type and code	**Pad**: Type code, manufacturer, date of specification
Structure and thickness	3-layer structure, thickness/tolerance H = (0,3...3,0) mm 
Materials	Thermally conductive materials, carrier, adhesives, support layer materials
Configurations	E.g. available as sheet, die-cut parts or roll form
Basic application	Type and thickness of gaps, type of clamping device for pressure

Table 8.2. Thermal properties.

Property	Symbol	Unit	Standard method
Operating temperature range	T	°C	..
Thermal impedance vs. pressure/ temperature *)	$\theta_{material}$	$K \cdot m^2/W$ $K \cdot cm^2/W$	ASTM D5470, modified at pressures of 0,1...0,3 MPa
Thermal conductivity	λ	W/(m·K)	ASTM E1461, ASTM C-177 $\lambda = \alpha C_p \rho$
Thermal diffusivity	α	m^2/s	ASTM E1269
Specific heat capacity	C_p	J/(kg·K) J/(g·°C)	ISO DIS 11357 Part 4 ASTM E1461, ASTM E1269
Phase change temperature	T_{phase}	°C	ASTM D3418

*) with specified surface quality of interface

Table 8.3. Electrical properties.

Property	Symbol	Unit	Standard method
Dielectric breakdown voltage		V	ASTM D149
Dielectric strength		V/mm	ASTM D149
Volume resistivity		Ωm	ASTM D4496, D257, C611
Surface resistivity		Ω/□	ASTM D4496, D257
Dielectric constant	ε_r	..	ASTM D150

Table 8.4. Mechanical properties.

Property	Unit	Standard method
Thickness	mm	ASTM 374
Coefficient of linear thermal expansion	ppm/K	ISO 11359-2, ASTM D-3386
Compression	%	ASTM C165, ASTM D575, ASTM D395, ASTM D695
Tensile modulus (strength)	MPa	ISO 527-1/ -2
Tensile strength	MPa	ISO 527-1/ -2, ASTM D412, ASTM F152, Afera 5004
Break elongation	%	ISO 527-1/ -2
Elongation (force/pressure)		ISO 527-1/ -2, ASTM D412, Afera 5004
Flexural modulus	MPa	ISO 178
Young's modulus	MPa	ASTM D575, ISO 6721-5
Peel adhesion		ASTM D3330, (IEC 60454-2, ISO 4578, ASTM B533, ASTM C906-00, ASTM D5109-99), Afera 5001
Bending strength	MPa	ISO 178
Impact strength	kJ/m^2	ISO 179
Hardness		ASTM D2240
Shear strength	MPa	ASTM D1002, Afera 5012
Viscosity, grease / gel / phase change material	Pa·s *(pascal second)*	ISO 1628, ASTM D2857

Table 8.5. Physical and chemical properties.

Property	Unit	Standard method
Specific gravity (ρ), density	kg/m^3 g/cm^3	ISO 1183 / (Micromeritics 5000 Helium Pycnometer), ASTM D792
Creep, migration		Properties in use conditions
Outgassing – corrosive agents – other agents and water	%	ASTM E595, ASTM D6375-99a (mass loss)
Chemical tolerance – detergents – solvents		Warning of materials, which may be damaging or harmful in use
Glass transition temperature	°C	ISO 11357. Part 2
Water absorption	%	ISO 62 /ISO NWI 11357. Part 8
Temperature of melting	°C	ISO 11357. Part 3
Cleaning properties		Reworkability, cleaning agents for removing material
Flammability rating		UL 94
Recyclability		RoHS, etc
Safety		UL

Table 8.6. Use in production of electronics, examples of description/requirement.

Property	Description
Storage/use conditions	Storage/use conditions and time before use and after opening the delivery package.
Handling properties of material	XXX coloured thermally conductive material is mechanically tough, soft, and flexible; endures well handling. Removing support layers requires special skills (description of requirements), tools needed, etc.
Surface quality of interface	Quality requirements of contact surfaces, preparation, possible cleaning agents.
Handling in production, pads and tapes	Precutting of material when delivered as sheets or rolls. – Remove first support layer, attach to the first contact surface. – Remove second support layer, attach to the second contact surface. – Fix the clamping device.
Handling in production, greases and gels	Applicable tools for dosing, amount of material/cm^2, method of spreading the material, cleaning.
Rework	Material adheres to the contact surfaces, and needs both mechanical removement and chemical cleaning (means and solvents described).

Table 8.7. Example of environmental and reliability test procedure.

Test type	Conditions	Duration	Standard
0 Sample preparation and stabilizing heating	80 ºC	12 h	..
1 Initial inspection	23 ºC	..	..
2 Stability at low temperature	-40 °C	168 h	IEC 60068-2-1 Tests A: Cold
3 Stability at high temperature	+125 °C	500 h	IEC 60068-2-2 Tests B: Dry heat
4 Composite temperature / humidity cyclic test	25/65°C RH 93% / -10 °C	10•24 h	IEC 60068-2-38 Test Z/AD
5 Change of temperature	-10...+125 10 °C/min	500 cycles á 1 h	IEC 60068-2-14 Modified Test N: Change of temperature
6 Change of temperature	-10...+125 10 °C/min	500 cycles á 1 h	IEC 60068-2-14 Modified Test N: Change of temperature
Final inspection	23 ºC	..	..

The test conditions and duration of tests should be agreed by the interested parties, and the test sample preparation, contact pressure during tests and measurements shall be described. The control parameters in environmental tests are typically thermal impedance and appearance.

9. Summary

The main goal of this project was to research the properties and reliability of the thermal interface materials used in electronics. The selected materials for measurements and tests contained pads, phase change materials and greases. The amount of suggested materials was 83 versions from which 36 types were selected for evaluation containing both electrically conducting and insulating materials.

The thermal impedance at pressures of 0,1...0,8 MPa was measured from the 34 materials. Also the thermal conductivity was measured with Hot Disk method, but this did not work well with the most thin sheets. From these 14 materials were selected for reliability tests.

A new measurement method for thermal impedance and a new type of test structure for material testing was developed. The new test structure for materials was designed for use in environmental tests. The test structure makes it possible to select test surface quality, test pressure and material thickness. The new method worked well and gave repeatedly stable results during all the tests.

The main criteria for evaluation of materials was the measured thermal impedance ($K \cdot cm^2/W$) at power densities from 10 to 25 W/cm^2. The high power density means that samples are heated during the measurements into the high operating temperature. The lowest impedance values had greases, phase change pads and graphite pads.

The materials were tested in various environmental conditions (high/low temperature, temperature/humidity cycle, temperature cycling) to see how their properties change in these conditions and what is the life endurance in use.

The measurement results of thermal impedance with various pressures showed that the wettability of materials have in many cases larger effect on impedance than the thermal conductivity of bulk material. The contact pressure has large effect on the impedance, therefore the impedance values should be given with the pressures (< 0,3 MPa), which are achievable in practical solutions. The environmental tests and long term temperature cycling showed that typically thermal impedance first goes down a little, and thereafter starts to raise. The tested materials (14 types) performed quite well in the tests, although there were some samples, which had quite large changes of impedance values.

The visual inspections showed some changes in the open edges of the pads and separation of phase change material from the carrier mesh, and some creep of greases. These have probably no significant harmful effect in use.

The long term behaviour of the tested materials seems to be quite good, however the cyclic test should be continued to over 5000 cycles to get better confidence for the long life products.

Quite important observation of the tests is that the variation from sample to sample in initial and final measurements is larger than the changes during tests. The final percentual variation is about 6...120% from the minimum to maximum values depending on the material type. Therefore the user should take care of this while calculating the total variation of thermal impedance for the whole life cycle of products.

Acknowledgements

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Risto Hienonen

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Appendix 1: Measurement and test standards for thermal interface materials

Afera standards

http://www.afera.com/

Afera: The European Associations for the Self Adhesive Tape Industry

Afera 4002: 1980 – Water Vapour Transmission of Adhesive Tape in a Warm Humid Atmosphere EN I2023 1996, Supplement A – Testing narrow width tapes

The test method is designed to determine the weight of the water vapour transmitted through the tape under specific test conditions.

Afera 4011: 1979 – Electric Strength of Adhesive Tape

The test method is designed to measure the electric strength of an adhesive tape under standard test conditions and, where applicable, after conditioning at high humidity, and also after conditioning by water immersion.

Afera 5001: 2003 – Self Adhesive Tapes – Measurement of Peel Adhesion from Stainless Steel or from its own Backing EN 1939: 2003

These methods cover the measurement of the peel adhesion of pressure-sensitive tapes.

Afera 5004: 2003 – Test Method for Breaking Strength and Elongation of Pressure-Sensitive Tape – EN14410:2003

The procedures in this test method describe the measurement of breaking strength, elongation at break, and energy to break, of pressure sensitive tapes.

Afera 5006: 2003 – Self Adhesive Tapes – Measurement of Thickness – EN 1942: 2003

This specification covers the determination of the thickness (calliper, gauge) of pressure-sensitive tapes at standard conditions.

Afera 5012: 2003 – Self Adhesive Tapes – Measurement of Static Shear Adhesion – EN 1943: 2002

These procedures help determine the ability of a pressure-sensitive tape to remain adhered under a constant load applied parallel to the surface of the tape and substrate.

ASTM standards

http://www.astm.org/

ASTM International, American Society for Testing and Material

ASTM B

ASTM B533-85(2004) Standard Test Method for PEEL Strength of Metal Electroplated Plastics

This test method gives two procedures for measuring the force required to PEEL a metallic coating from a plastic substrate. One procedure (Procedure A) utilizes a universal testing machine and yields reproducible measurements that can be used in research and development, in quality control and product acceptance, in the description of material and process characteristics, and in communications. The other procedure (Procedure B) utilizes an indicating force instrument that is less accurate and that is sensitive to operator technique. It is suitable for process control use.

ASTM C

ASTM C165-00 Standard Test Method for Measuring Compressive Properties of Thermal Insulations

This test method covers two procedures for determining the compressive resistance of thermal insulations.

ASTM C-177 C177-97 Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus

1. Scope

1.1 This test method covers the achievement and measurement of steady-state heat flux through flat-slab specimens using a guarded-hot-plate apparatus. The method encompasses both the single-sided and the double-sided mode of measurement. Both distributed and line source guarded heating plate designs are included, in principle, in this test method. The reader should consult the standard practices on the single-sided mode of operation and on the line source apparatus for further details on these variations of the method.

1.2 The calculations of thermal transmission properties based upon measurements using this method shall be performed in conformance with Practice C1045.

1.3 This is an absolute (or primary) method of measurement since no heat flux reference standards are required except to confirm accuracy statements and to establish traceability to recognized standards. This absolute method is contrasted with a comparative (or secondary) method, such as Test Method C518, in which the results are directly dependent on heat flux reference standards.

1.4 This test method is applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions. Special precautions in the measurement process are described for the following:

1.4.1 Specimens exhibiting appreciable inhomogeneousity, anisotropies, rigidity, or extremes of thermal flux density.

1.4.2 Measurements conducted at extremes of temperature (either high or low) or under vacuum conditions.

1.5 This test method is intended to allow a wide variety of apparatus designs and design accuracies to satisfy the requirements of specific measurement problems. Compliance with this test method requires a statement of the uncertainty of each reported variable in the report. Therefore, in the following sections, the significant error factors will be discussed.

1.6 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM C611-98 Standard Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature (referred in the GrafTech's specification of HiTherm)

This test method covers the determination of the electrical resistivity of manufactured carbon and graphite articles at room temperature.

ASTM C906-00 Standard Test Method for T-Peel Strength of Hot Applied Sealants

This test method covers a laboratory procedure for determining the PEEL strength of a hot-applied sealant, hereafter referred to as the sealant, when installed between flexible metal substrates of a T-type configuration. It also provides information on the adhesion of the sealant to the tested substrates.

ASTM D

ASTM D149-97a (2004) Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

1.1 This test method covers procedures for the determination of dielectric strength of solid insulating materials at commercial power frequencies, under specified conditions.

1.2 Unless otherwise specified, the tests shall be made at 60 Hz. However, this test method may be used at any frequency from 25 to 800 Hz. At frequencies above 800 Hz, dielectric heating may be a problem.

ASTM D150-98 (2004) Standard Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation

ASTM D257-99 Standard Test Methods for DC Resistance or Conductance of Insulating Materials

ASTM D374-99 Standard Test Methods for Thickness of Solid Electrical Insulation

These test methods cover the determination of the thickness of several types of solid electrical insulating materials employing recommended techniques. Use these methods except as otherwise required by a material specification.

ASTM D395-03 Standard Test Methods for Rubber Property – Compression Set

These test methods cover the testing of rubber intended for use in applications in which the rubber will be subjected to compressive stresses in air or liquid media. They are applicable particularly to the rubber used in machinery mountings, vibration dampers, and seals. Two test methods are covered as follows:

ASTM D412-98a(2002)e1 Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers – Tension

These test methods cover procedures used to evaluate the tensile (tension) properties of vulcanized thermoset rubbers and thermoplastic elastomers. These methods are not applicable to ebonite and similar hard, low elongation materials. The methods appear as follows:

Test Method A – Dumbbell and Straight Section Specimens

Test Method B – Cut Ring Specimens

WITHDRAWN STANDARD: D551-41 Method of Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (Withdrawn 1965).

ASTM D575-91 (2001) Standard Test Methods for Rubber Properties in **Compression**

These test methods describe two test procedures for determining the compression-deflection characteristics of rubber compounds other than those usually classified as hard rubber and sponge rubber.

ASTM D648-01 Standard Test Method for **Deflection Temperature** of Plastics Under Flexural Load in the Edgewise Position

1. Scope

1.1 This test method covers the determination of the temperature at which an arbitrary deformation occurs when specimens are subjected to an arbitrary set of testing conditions.

1.2 This test method applies to molded and sheet materials available in thicknesses of 3 mm (1/8 in.) or greater and which are rigid at normal temperature.

Note 1 – Sheet stock less than 3 mm (0.125 in.) but more than 1 mm (0.040 in.) in thickness may be tested by use of a composite sample having a minimum thickness of 3 mm. The laminate must be of uniform stress distribution. One type of composite specimen has been prepared by cementing the ends of the laminate together and then smoothing the edges with sandpaper. The direction of loading shall be perpendicular to the edges of the individual laminate.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Note 2 – The test method described as a Method B of this test method, and test methods Ae and Be of **ISO 75-1 and ISO 75-2, 1993 are technically equivalent**.

ASTM D695-02a Standard Test Method for **Compressive** Properties of Rigid Plastics

This test method covers the determination of the mechanical properties of unreinforced and reinforced rigid plastics, including high-modulus composites, when loaded in compression at relatively low uniform rates of straining or loading. Test specimens of standard shape are employed.

ASTM D734 (cancelled 1962), substitutive standards are D2219 and D2220, referred in the specification of Keratherm

ASTM D792-00 Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

These test methods describe the determination of the specific gravity (relative density) and density of solid plastics in forms such as sheets, rods, tubes, or molded items.

ASTM D1002-01 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)

This test method covers the determination of the apparent shear strengths of adhesives for bonding metals when tested on a standard single-lap-joint specimen and under specified conditions of preparation and test.

ASTM D1711 Terminology Relating to Electrical Insulation

ASTM D2219-02 Standard Specification for Poly(Vinyl Chloride) Insulation for Wire and Cable, 60 °C Operation

ASTM D2220-02 Standard Specification for Poly(Vinyl Chloride) Insulation for Wire and Cable, 75 °C Operation

ASTM D2240-03 Standard Test Method for Rubber Property – Durometer **Hardness**

ASTM D2857-95(2001) Standard Practice for Dilute Solution **Viscosity** of Polymers

1.1 This practice covers the determination of the dilute solution viscosity of polymers. There are several ASTM standards (Test Methods D789, D1243, D1601, and D4603, and Practice D3591) that describe dilute solution viscosity procedures for specific polymers, such as nylon, poly(vinyl chloride), polyethylene, and poly(ethylene terephthalate). This practice is written to augment these standards when problems arise with which the specific procedure is not concerned, or when no standard is available for the polymer under investigation.

1.2 This practice is applicable to all polymers that dissolve completely without chemical reaction or degradation to form solutions that are stable with time at a temperature between ambient and 150 °C. Results are usually expressed as relative viscosity (viscosity ratio), inherent viscosity (logarithmic viscosity number), or intrinsic viscosity (limiting viscosity number).

ASTM D3330/D3330M-02e1 Standard Test Method for Peel **Adhesion** of Pressure-Sensitive Tape

1. Scope

1.1 These test methods cover the measurement of the peel adhesion of pressure-sensitive tapes.

1.1.1 Test Method A gives a measure of the adherence, when peeled at 180° angle, to a standard steel panel or to other surface of interest for a single-coated tape.

1.1.2 Test Method B gives a measure of the adherence to the backing of a single-coated tape.

1.1.3 Test Method C gives a measure of the adherence of double-coated tape to a standard steel panel or other surface of interest.

1.1.4 Test Method D gives a measure of the adherence of the release liner to the adhesive of either single- or double-coated tape.

1.1.5 Test Method E gives a measure of the adherence of an adhesive transfer tape to a standard steel panel or other surface of interest.

1.1.6 Test Method F gives a measure of the adherence, when peeled at 90° angle, to a standard steel panel or other surface of interest for a single-coated tape.

1.2 These test methods provide a means of assessing the uniformity of the adhesion of a given type of pressure-sensitive adhesive tape. The assessment may be within a roll of tape, between rolls, or between production lots.

1.3 Variations in either the tape backing or the adhesive, or both, affect the response. Therefore, these test methods cannot be used to pinpoint the specific cause(s) of non-uniformity.

ASTM D3386-00 Standard Test Method for Coefficient of Linear Thermal Expansion of Electrical Insulating Materials

1. Scope

1.1 This test method covers determination of the coefficient of linear thermal expansion of electrical insulating materials by use of a thermomechanical analyzer.

1.2 This test method is applicable to materials that are solid over the entire range of temperature used, and that retain sufficient hardness and rigidity over the temperature range so that irreversible indentation of the specimen by the sensing probe does not occur.

1.3 Transition temperatures also may be obtained by this test method.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.5 The values stated in SI units are the standard.

Note 1 – There is no similar or equivalent ISO/IEC standard.

ASTM D3418-03 Standard Test Method for Transition Temperatures of Polymers by Differential Scanning Calorimetry

1. Scope

1.1 This test method covers determination of transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry.

Note 1 – True heats of fusion are to be determined in conjunction with structure investigation, and frequently, specialized crystallization techniques are needed.

1.2 This test method is applicable to polymers in granular form or to any fabricated shape from which it is possible to cut appropriate specimens.

1.3 The normal operating temperature range is from the cryogenic region to 600 °C. Certain equipment allows the temperature range to be extended.

ASTM D4496 Test Method for DC Resistance or Conductance of Moderately Conductive Materials

ASTM D5109-99 Standard Test Methods for Copper-Clad Thermosetting Laminates for Printed Wiring Boards

These test methods cover the procedures for testing copper-clad laminates produced from fiber reinforced, thermosetting polymeric materials intended for fabrication of printed wiring boards.

ASTM D5470-01 Standard Test Methods for **Thermal Transmission Properties** of Thin Thermally Conductive Solid Electrical Insulation Materials (thermal resistance measurement)

ASTM D6343-99 Test Methods for **Thin Thermally Conductive Solid Materials** for Electrical Insulation and Dielectric Applications

ASTM D6375-99a Standard Test Method for **Evaporation Loss** of Lubricating Oils by Thermogravimetric Analyzer (TGA) Noack Method

This test method covers the procedure for determining the Noack evaporation loss of lubricating oils using a thermogravimetric analyzer test (TGA). The test method is applicable to base stocks and fully formulated lubricant oils having a Noack evaporative loss ranging from 0 to 30 weight percent. This procedure requires much smaller specimens, and is faster, easier and safer than the standard Noack methods.

ASTM D6465-99 Standard Guide for Selecting Aerospace and General Purpose Adhesives and Sealants

1. Scope

1.1 This guide is intended to assist design engineers, manufacturing/industrial engineers, and production managers in selecting the best-fit adhesive/sealant or bonding/sealing process. The guide takes into account environmental pollution prevention and occupational health and safety factors in a selection process.

1.2 This guide is not to be considered as a database of acceptable materials. It will guide the engineers and managers through the adhesive/sealant material selection process, calling for engineers to customize their selection based on the bonding or sealing performance requirements for the specified application. A comprehensive selection process will allow for the establishment of a more efficient production process, and may eliminate unnecessary process steps. A total life cycle cost analysis or performance/cost of implementation study is recommended to compare the available alternatives.

1.3 This guide is for aerospace and general purpose operations. It is not intended to be used for automotive, carpet, construction, electronics, medical/dental, optical, or structural and nonstructural wood applications. Note that this guide is not specifically for these applications, but the general methodology may be used in the selection process for these applications.

ASTM D6551-00 Standard Practice for Accelerated Weathering of Pressure-Sensitive Tapes by Xenon-Arc Exposure Apparatus

ASTM D6604-00 Standard Practice for Glass Transition Temperatures of Hydrocarbon Resins by Differential Scanning Calorimetry

1. Scope

1.1 This practice covers determination of glass transition temperatures of hydrocarbon (HC) resins by differential scanning calorimetry (DSC).

1.2 This practice is applicable to HC resins as defined in Termninology D6440. The normal operating temperature range is from the cryogenic region to approximately 180 ºC. The temperature range can be extended.

ASTM E

ASTM E230-03 Standard Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

ASTM E595-93(2003)e1 Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from **Outgassing** in a Vacuum Environment

1. Scope

1.1 This test method covers a screening technique to determine volatile content of materials when exposed to a vacuum environment. Two parameters are measured: total mass loss (TML) and collected volatile condensable materials (CVCM). An additional parameter, the amount of water vapor regained (WVR), can also be obtained after completion of exposures and measurements required for TML and CVCM.

1.2 This test method describes the test apparatus and related operating procedures for evaluating the mass loss of materials being subjected to 125 °C at less than 7 X 10-3 Pa (5 X 10 -5 torr) for 24 h. The overall mass loss can be classified into noncondensables and condensables. The latter are characterized herein as being capable of condensing on a collector at a temperature of 25 °C.

Note 1 – Unless otherwise noted, the tolerance on 25 and 125 °C is ±1 °C and on 23 °C is ±2 °C. The tolerance on relative humidity is ±5%.

1.3 Many types of organic, polymeric, and inorganic materials can be tested. These include polymer potting compounds, foams, elastomers, films, tapes, insulations, shrink tubings, adhesives, coatings, fabrics, tie cords, and lubricants. The materials may be tested in the “as-received” condition or prepared for test by various curing specifications.

1.4 This test method is primarily a screening technique for materials and is not necessarily valid for computing actual contamination on a system or component because of differences in configuration, temperatures, and material processing.

1.5 The criteria used for the acceptance and rejection of materials shall be determined by the user and based upon specific component and system requirements. Historically, TML of 1,00% and CVCM of 0,10% have been used as screening levels for rejection of spacecraft materials.

1.6 The use of materials that are deemed acceptable in accordance with this test method does not ensure that the system or component will remain uncontaminated. Therefore, subsequent functional, developmental, and qualification tests should be used, as necessary, to ensure that the material's performance is satisfactory.

ASTM E831-05 Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis

1. Scope

1.1 This test method determines the apparent coefficient of linear thermal expansion of solid materials using thermomechanical analysis techniques. Related information can be found in Refs. (1-12)[2].

1.2 This test method is applicable to solid materials that exhibit sufficient rigidity over the test temperature range such that the sensing probe does not produce indentation of the specimen.

1.3 The recommended lower limit of coefficient of linear thermal expansion measured with this test method is 5 μm/(m·°C). The test method may be used at lower (or negative) expansion levels with decreased accuracy and precision (see Section 11).

1.4 This test method is applicable to the temperature range from 120 to 900 °C. The temperature range may be extended depending upon the instrumentation and calibration materials used.

1.5 Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may also be used.

Note 1 – Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user to determine the necessary equivalency prior to use.

1.6 SI values are the standard.

1.7 This test method is related to ISO 11359-2 but is significantly different in technical detail.

2. Referenced Documents

D3386 Test Method for Coefficient of Linear Thermal Expansion of Electrical Insulating Materials
D696 D696-03 Standard Test Method for Coefficient of Linear Thermal Expansion of Plastics Between -30 °C and 30 °C With a Vitreous Silica Dilatometer
E1142 Standard Terminology Relating to Thermophysical Properties
E1363 Standard Test Method for Temperature Calibration of Thermomechanical Analyzers
E2113 Standard Test Method for Length Change Calibration of Thermomechanical Analyzers
E228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer
E473 Standard Terminology Relating to Thermal Analysis and Rheology
ISO 11359-2 Plastics-Thermomechanical Analysis (TMA) – Part 2: Determination of Coefficient of Linear Thermal Expansion and Glass Transition Temperature

ASTM E1269-01 Standard Test Method for Determining Specific **Heat Capacity** by Differential Scanning Calorimetry

1. Scope

1.1 This test method covers the determination of specific heat capacity by differential scanning calorimetry.

1.2 This test method is generally applicable to thermally stable solids and liquids.

1.3 The normal operating range of the test is from 100 to 600 °C. The temperature range can be extended, depending upon the instrumentation and specimen holders used.

1.6 This method is similar to ISO 11357-4, but contains additional methodology not found in that method. Additionally, ISO 11357-4 contains practices not found in this standard. This method is similar to **Japanese Industrial Standard K 7123**, but contains additional methodology not found in that method.

ASTM E1461-01 Standard Test Method for **Thermal Diffusivity** of Solids by the Flash Method

1. Scope

1.1 This test method covers the determination of the thermal diffusivity of primarily homogeneous isotropic solid materials. Thermal diffusivity values ranging from 10-7 to 10-3 m 2/s are readily measurable by this test method from about 75 to 2800 K.

1.2 This test method is a more detailed form of Test Method C 714, but has applicability to much wider ranges of materials, applications, and temperatures, with improved accuracy of measurements.

ASTM F

ASTM F152-95(2002) Standard Test Methods for Tension Testing of Nonmetallic Gasket Materials (referred in the specification of GrafTech)

These test methods cover the determination of tensile strength of certain non metallic gasketing materials at room temperature. The types of materials covered are those containing asbestos and other inorganic fibers (Type 1), cork (Type 2), cellulose or other organic fiber (Type 3), and flexible graphite (Type 5) as described in Classification F 104. These test methods are not applicable to the testing of vulcanized rubber, a method for which is described in Test Methods D 412 nor for rubber O-rings, a method for which is described in Test Methods D 1414.

IEC standards

http://www.iec.ch/searchpub/cur_fut.htm

The International Electrotechnical Commission (**IEC**)

IEC environmental tests

IEC 60068-2-1 (1990-05) Tests A: Cold

IEC 60068-2-2 (1974-01) Tests B: Dry heat

IEC 60068-2-14 (1984-01) Test N: Change of temperature

IEC 60068-2-30 (1980-01) Test Db: Test Db and guidance: Damp heat, cyclic (12 + 12-hour cycle) 25 / 55 °C, RH 95% / 93%, repeated 6 times (= 6 days)

IEC 60068-2-38 (1974-01) Test Z/AD: Composite temperature/humidity cyclic test, 25 / 65 / -10 °C, RH 93%, repeated 10 times

Rate of temperature change +0,33 °C/min and during cold period -1,2 °C/min

IEC 60068-2-45 (1980-01) Test XA and guidance: Immersion in cleaning solvents

IEC 60068-2-61 (1991-07) Test Z/ABDM: Climatic sequence

IEC 60068-2-66 (1994-06) Test Cx: Damp heat, steady state (unsaturated pressurized vapour)

IEC 60068-2-67 (1995-12) Test Cy Damp heat, steady state, accelerated test primarily intended for components

IEC 60068-2-78 (2001-08) Test Cab Damp heat steady state

IEC, other standards

IEC 60216-1 (2001-07) Electrical insulating materials – Properties of thermal endurance – Part 1: Ageing procedures and evaluation of test results

Specifies the general ageing conditions and procedures to be used for deriving thermal endurance characteristics and gives guidance in using the detailed instructions and guidelines in the other parts of the standard. Simplified procedures are also given, with the conditions under which these procedures may be used. Although originally developed for use with electrical insulating materials and simple combinations of such materials, the procedures are considered to be of more general applicability and are widely used in the assessment of materials not intended for use as electrical insulation. In the application of this standard, it is assumed that a practically linear relationship exists between the logarithm of the time required to cause the predetermined property change and the reciprocal of the corresponding absolute temperature (Arrhenius relationship). For the valid application of the standard, no transition, in particular no first-order transition, should occur in the temperature range under study.

IEC 60216-2 (1990-06) Guide for the determination of thermal endurance properties of electrical insulating materials. Part 2: Choice of test criteria

IEC 60216-3 (2002-02) Electrical insulating materials – Thermal endurance properties – Part 3: Instructions for calculating thermal endurance characteristics

Specifies the calculation procedures to be used for deriving thermal endurance characteristics from experimental data obtained in accordance with the instructions of IEC 60216-1 and IEC 60216-2. The experimental data may be obtained using non-destructive, destructive or proof tests. Data obtained from non-destructive or proof tests may be incomplete, in that measurement of times taken to reach the endpoint may have been terminated at some point after the median time but before all specimens have reached end-point. The procedures are illustrated by worked examples, and suitable computer programs are recommended to facilitate the calculations.

IEC 60216-4-2 (2000-07) Electrical insulating materials – Thermal endurance properties – Part 4-2: Ageing ovens – Precision ovens for use up to 300 °C

Covers minimum performance requirements for ventilated and electrically heated precision ovens for thermal endurance evaluation of electrical insulating materials and other appropriate applications. It covers ovens designed to operate over all or part of the temperature range from 20 K above room temperature up to 300 °C. Two possible methods of achieving the required performance are described: a) where the required performance is achieved by precise control of temperature in a simple single chamber oven, i.e., upgraded versions of ovens conforming to IEC 60216-4-1, and, otherwise b) where the required performance is achieved by utilizing a second chamber (iso-box), mounted within the chamber of a single-chamber oven, the purpose of which is to reduce the magnitude of any temperature changes to an acceptable level whilst maintaining the required levels of air changes and circulation.

IEC 60216-5 (2003-01) Electrical insulating materials – Thermal endurance properties – Part 5: Determination of relative thermal endurance index (RTE) of an insulating material

Specifies the experimental and calculation procedures to be used for deriving the relative thermal endurance index of a material from experimental data obtained in accordance with the instructions of IEC 60216-1 and IEC 60216-2. The calculation procedures are supplementary to those of IEC 60216-3. Guidance is also given for assessment of thermal ageing after a single fixed time and temperature, without extrapolation. The experimental data may in principle be obtained using destructive, non-destructive or proof tests, although destructive tests have been much more extensively employed. Data obtained from non-destructive or proof tests may be 'censored', in that measurement of times taken to reach the endpoint may have been terminated at some point after the median time but before all specimens have reached end-point (see 3.1 of IEC 60216-3).

Guidance is given for preliminary assignment of a material to an insulation class, based upon the thermal ageing performance. The second edition differs from the first in that it no longer aims to provide general guidance on application of thermal endurance characteristics, but provides instructions for deriving a provisional estimate of the temperature up to which a material may give satisfactory performance in an application (by comparative thermal ageing with a material of known performance).

IEC 60216-6 (2003-12) Electrical insulating materials – Thermal endurance properties – Part 6: Determination of thermal endurance indices (TI and RTE) of an insulating material using the fixed time frame method

Specifies the experimental and calculation procedures for deriving the thermal endurance characteristics, temperature index (TI) and relative thermal endurance index (RTE) of a material using the 'fixed time frame method (FTFM)'. In this protocol, the ageing takes place for a small number of fixed times, using the appropriate number of ageing temperatures throughout each time, the properties of the specimens being measured at the end of the relevant time interval. This differs from the procedure of IEC 60216-1, where ageing is conducted at a small number of fixed temperatures, property measurement taking place after ageing times dependent on the progress of ageing. Both the TI and the RTE determined according to the FTFM protocol are derived from experimental data obtained in accordance with the instructions of IEC 60216-1 and IEC 60216-2, as modified in this standard. The calculation procedures and statistical tests are modified in relation to IEC 60216-3 and IEC 60216-5.

IEC 60454-1 (1992) Specifications for pressure-sensitive adhesive tapes for electrical purposes – Part 1: General requirements

Specifies general requirements for pressure-sensitive adhesive tapes for electrical purposes. Particular types of tape are designated by using the code letters for the form and nature of backing material given in a new table, followed by the figures for temperature index and code letters for the adhesive, as indicated in an updated table.

IEC 60454-2 (1994-11) Specification for pressure-sensitive adhesive tapes for electrical purposes – Part 2: Methods of test

Describes methods of determining the mechanical and electrical resistance and the adhesive properties of pressure-sensitive adhesive tapes, and the test methods to be used.

IEC 60454-3-1 (1998) Pressure sensitive adhesive tapes

IEC 60493-1 (1974-01)
Guide for the statistical analysis of ageing test data. Part 1: Methods based on mean values of normally distributed test results

ISO standards

http://www.iso.org/iso/en/ISOOnline.frontpage

International Organization for Standardization (**ISO**)

ISO 31-4: 1992   Quantities and units – Part 4: Heat

ISO 62:1999   Plastics – Determination of water absorption

ISO 75-1:1993   Plastics – Determination of temperature of deflection under load – Part 1: General test method

ISO 75-2:1993   Plastics – Determination of temperature of deflection under load – Part 2: Plastics and ebonite

ISO 75-3:1993   Plastics – Determination of temperature of deflection under load – Part 3: High-strength thermosetting laminates and long-fibre-reinforced plastics

ISO 178:2001   Plastics – Determination of flexural properties

ISO 178:2001/Amd 1:2004   Precision statement

ISO 179-1:2000   Plastics – Determination of Charpy impact properties – Part 1: Non-instrumented impact test

ISO 179-2:1997   Plastics – Determination of Charpy impact properties – Part 2: Instrumented impact test

ISO 179-2:1997/Cor 1:1998

ISO 527-1:1993   Plastics – Determination of tensile properties – Part 1: General principles

Several different types of test specimen are defined to suit different types of material. The methods specified are selectively suitable for use with the following range of materials: rigid and semirigid thermoplastics moulding, including filled and reinforced compounds in addition to unfilled types, sheets and films, rigid and semirigid thermosetting moulding materials including filled and reinforced compounds, and sheets including laminates, as well as fibre-reinforced thermoset and thermoplastics composites.

ISO 527-1:1993/Cor 1:1994

ISO 527-2:1993 Plastics – Determination of tensile properties – Part 2: Test conditions for moulding and extrusion plastics

The methods specified are selectively suitable for use with the following range of materials: rigid and semirigid thermoplastics moulding, extrusion and cast materials, including compounds filled and reinforced by e.g. short fibres, small rods, plates or granules but excluding textile fibres in addition to unfilled types, rigid and semirigid thermosetting moulding and cast materials, including filled and reinforced compounds but excluding textile fibres as reinforcement, and thermotropic liquid crystal polymers.

ISO 527-3:1995 Plastics – Determination of tensile properties – Part 3: Test conditions for films and sheets

Specifies the conditions for determining the tensile properties of plastics films or sheets less than 1 mm thick, based upon the general principles given in part 1. Cancels and replaces ISO/R 527:19966 as well as ISO 1184:1983, of which it constitutes a technical revision.

ISO 527-3:1995/Cor 1:1998

ISO 527-3:1995/Cor 2:2001

ISO 527-4:1997 Plastics – Determination of tensile properties – Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites

ISO 527-5:1997 Plastics – Determination of tensile properties – Part 5: Test conditions for unidirectional fibre-reinforced plastic composites

ISO 1183:1987 Plastics – Methods for determining the density and relative density of non-cellular plastics

Specification of four methods for the determination of the density of plastics except cellular plastics. Method A: Immersion method for plastics in a finished condition; Method B: Pyknometer method for plastics in the form of small particles such as powders, granules pellets or flakes; Method C: Titration method for plastics in forms as required for method A including pellets; Method D: Density gradient column method, particularly suited to measurements of small samples of products and to comparison of densities.

ISO 1183-1:2004 Plastics – Methods for determining the density of non-cellular plastics – Part 1: Immersion method, liquid pyknometer method and titration method.

ISO 1183-1 specifies three methods for the determination of the density of non-cellular plastics in the form of void-free moulded or extruded objects, as well as powders, flakes and granules:

1. Method A: Immersion method, for solid plastics (except for powders) in void-free form.
2. Method B: Liquid pyknometer method, for particles, powders, flakes, granules or small pieces of finished parts.
3. Method C: Titration method, for plastics in any void-free form.

This part of ISO 1183 is applicable to pellets as long as they are void-free.

ISO 1183-3:1999 Plastics – Methods for determining the density of non-cellular plastics – Part 3: Gas pyknometer method

ISO 1628, Plastics-Determination of Viscosity Number and Limiting Viscosity Number

ISO 2578:1993 Plastics – Determination of time-temperature limits after prolonged exposure to heat.

Specifies the principles and procedures for evaluating the thermal endurance properties of plastics exposed to elevated temperature for long periods. The study of the thermal ageing is based solely on the change in certain properties resulting from a period of exposure to elevated temperature. The properties studied are always measured after the temperature has returned to ambient.

ISO 4578:1997 Adhesives – Determination of peel resistance of high-strength adhesive bonds – Floating-roller method.

ISO 5085-1:1989 Textiles – Determination of thermal resistance – Part 1: Low thermal resistance

The method is suitable for materials up to 20 mm thick (above this thickness, edge losses become appreciable). Advice on suitable components for constructing the apparatus is given in Annex A, means of determining the thermal conductivity are described in Annex B, and numerical values for some textile materials are given in Annex C.

ISO 6721-5:1996 Plastics – Determination of dynamic mechanical properties – Part 5: Flexural vibration – Non-resonance method

Describes a flexural vibration method for determining the components of the Young's complex modulus E/Zeichen) of polymers at frequencies typically in the range 0,01 Hz to 100 Hz. Suitable for measuring dynamic storage moduli in the range 10 MPa to 200 MPa. Particularly suited to the measurement of loss factors greater than 0,1.

ISO 7345:1987 Thermal insulation – **Physical quantities and definitions**

Defines physical quantities used in the field of thermal insulation, i.a. quantity of heat, heat flow rate, density of heat flow rate, thermal conductivity, thermal resistance, thermal transmittance, heat capacity, thermal diffusivity, thermal effusivity, etc., and gives the corresponding symbols and units.

ISO 8058:1999 Air cargo – Insulated container – Thermal efficiency requirements. 9 p.

ISO 8301:1991 Thermal insulation – Determination of steady-state thermal resistance and related properties – Heat flow meter apparatus

Defines the use of the heat flow meter method to measure the steady-state heat transfer through flat slab specimens and the calculation of its heat transfer properties. Annex A forms an integral part of this standard. Annexes B, C, D and E are for information only.

ISO 8302:1991 Thermal insulation – Determination of steady-state thermal resistance and related properties – Guarded hot plate apparatus

Defines the use of the guarded hot plate method to measure the steady-state heat transfer through flat slab specimens and the calculation of its heat transfer properties. Annex A forms an integral part of this standard. Annexes B, C and D are for information only.

ISO 8510-1:1990 Adhesives – Peel test for a flexible-bonded-to-rigid test specimen assembly – Part 1: 90 degree peel

Particularly suitable for use with less flexible adherends for which a 180 ° peel test is not suitable because the adherends crack, break or delaminate. A bonded assembly of two adherends is prepared using the adhesive under test. The adherends are then pulled apart at a steady rate, starting at the open end of the bond, in such a way that separation occurs progressively along the length of the bonded adherends. The force is applied approximately normal to the plane of the bond, through the separated part of the flexible adherend. A figure shows a schematic diagram.

ISO 8510-2 1990 Adhesives – Peel test for a flexible-bonded-to-rigid test specimen assembly – Part 2: 180 degree peel

Suitable for use with less flexible adherends. The principle consists in preparing a bonded assembly of two adherends, using the adhesive under test. The adherends are then pulled apart at a steady rate, starting at the open end of the bond, in such a way that separation occurs progressively along the length of the bonded adherends. The force is applied substantially parallel to the plane of the bond, through the separated part of the flexible adherend. A figure shows a schematic diagram.

ISO 9664: 1993 Adhesives – Test methods for fatigue properties of structural adhesives in tensile shear

ISO 11339: 2003 Adhesives – T-peel test for flexible-to-flexible bonded assemblies

ISO 11339:2003 specifies a T-peel test for the determination of the peel strength of an adhesive by measuring the peeling force of a T-shaped bonded assembly of two flexible adherends. This test procedure does not provide design information. NOTE – This method was originally developed for use with metal adherends, but other flexible adherends may also be used.

ISO 11346:2004 Rubber, vulcanized or thermoplastic – Estimation of life-time and maximum temperature of use

ISO 11346:2004 specifies the principles and procedures for estimating the thermal endurance of rubbers from the results of exposure to elevated temperatures for long periods.

Two approaches are specified:

1. one using the Arrhenius relation
2. the other using the WLF equation.

The estimation of thermal endurance is based solely on the change in selected properties resulting from periods of exposure to elevated temperatures. The various properties of rubbers change at different rates on thermal ageing, hence comparisons between different rubbers can only be made using the same properties.

ISO 11357. Plastics. Differential Scanning Calorimetry (DSC).

ISO 11357. Part 1: 1997. (BS EN ISO 11357-1: 1997.) *General principles.*

ISO 11357. Part 2: 1999. (BS ISO 11357- 2: 1999.) *Determination of glass transition temperature.*

ISO 11357. Part 3: 1999. (BS ISO 11357- 3: 1999.) *Determination of temperature and enthalpy of melting and crystallisation.*

ISO DIS 11357. Part 4. *Determination of specific heat capacity.*

ISO 11357. Part 5: 1999. *Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion.*

ISO FDIS 11357. Part 6. *Oxidation induction time.*

ISO FDIS 11357. Part 7. *Determination of crystallisation kinetics.*

ISO NWI 11357. Part 8. *Determination of moisture content.*

ISO 11359-1:1999 Plastics – Thermomechanical analysis (TMA) – Part 1: General principles

ISO 11359-2:1999 Plastics – Thermomechanical analysis (TMA) – Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature

ISO 11359-3:2002 Plastics – Thermomechanical analysis (TMA) – Part 3: Determination of penetration temperature

ISO 11561:1999 Ageing of thermal insulation materials – Determination of the long-term change in thermal resistance of closed-cell plastics (accelerated laboratory test methods). 19 p.

ISO 14129:1997 Fibre-reinforced plastic composites – Determination of the in-plane shear stress/shear strain response, including the in-plane shear modulus and strength, by the plus or minus 45 degree tension test method. 9 p.

UL standards

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UL 94 (October 29, 1996) Tests for Flammability of Plastic Materials for Parts in Devices and Appliances

1 Scope

1.1 These requirements cover tests for flammability of plastic materials used for parts in devices and appliances. They are intended to serve as a preliminary indication of their acceptability with respect to flammability for a particular application.

1.2 The methods described in this Standard involve standard size specimens and are intended to be used solely to measure and describe the flammability properties of materials, used in devices and appliances, in response to heat and flame under controlled laboratory conditions. The actual response to heat and flame of materials depends upon the size and form, and also on the end-use of the product using the material. Assessment of other important characteristics in the end-use application includes, but is not limited to, factors such as ease of ignition, burning rate, flame spread, fuel contribution, intensity of burning, and products of combustion.

1.3 The final acceptance of the material is dependent upon its use in complete equipment that conforms with the standards applicable to such equipment. The flammability classification required of a material is dependent upon the equipment or device involved and the particular use of the material. The performance level of a material determined by these methods shall not be assumed to correlate with its performance in end-use application.

1.3 revised July 10, 1998

1.4 If found to be appropriate, the requirements are applied to other non metallic materials.

1.4 revised June 10, 1997

1.5 These requirements do not cover plastics when used as materials for building construction or finishing.

1.6 A product that contains features, characteristics, components, materials, or systems new or different from those covered by the requirements in this standard, and that involves a risk of fire or of electric shock or injury to persons shall be evaluated using appropriate additional component and end-product requirements to maintain the level of safety as originally anticipated by the intent of this standard. A product whose features, characteristics, components, materials, or systems conflict with specific requirements or provisions of this standard does not comply with this standard. Revision of requirements shall be proposed and adopted in conformance with the methods employed for development, revision, and implementation of this standard.

1.6 revised June 8, 2000

UL-746A (November 1, 2000) Standard for Polymeric Materials – Short Term Property Evaluations

1 Scope

1.1 These requirements cover short-term test procedures to be used for the evaluation of materials used for parts intended for specific applications in electrical end products.

1.2 Together with the requirements mentioned in Supplementary Test Procedures, Section 4, these investigations provide data with respect to the physical, electrical, flammability, thermal, and other properties of the materials under consideration and are intended to provide guidance for the material manufacturer, the molder, the end-product manufacturer, safety engineers, and other interested parties.

UL 746B (August 28, 1996) Standard for Polymeric Materials; Long Term Property Evaluations

Scope: These requirements cover long-term test procedures to be used for the evaluation of materials used for parts intended for specific applications in end products.

Together with the Standards mentioned in Supplementary Test Procedures, Section 3, these investigations provide data with respect to the physical, electrical, flammability, thermal, and other properties of the materials under consideration and are intended to provide guidance for the material manufacturer, the molder, the end-product manufacturer, safety engineers, and other interested parties.

UL 746C (November 29, 2001) Standard for Polymeric Materials – Use in Electrical Equipment Evaluations

I746C.5-H Scope

I746C.5-H These requirements cover parts made of polymeric materials that are used in electrical equipment and describe the various test procedures and their use in the testing of such parts and equipment.

I746C.5-H These requirements do not cover the specific insulating systems that are covered by the requirements contained in the Standard for Systems of Insulating Materials, General, UL 1446.

I746C.5-H Test procedures are provided herein for the evaluation of polymeric materials in specific applications. These test procedures include references to data obtained from small-scale property tests conducted under standard conditions as well as other practical means of evaluation.

UL 746D (May 28, 1998) Standard for Polymeric Materials – Fabricated Parts

1 Scope

1.1 These requirements cover a program applicable to parts that have been molded or fabricated from polymeric material and describe the material-identity control system intended to provide traceability of the material used for the polymeric parts through the handling, molding or fabrication, and shipping operations. Guidelines are also provided for acceptable blending or simple compounding operations that may affect risk of fire, electrical shock, or injury to persons.

1.2 This program is intended to provide quick verification of material identification by means of an identification marking on the part, or on the carton in which the part is shipped, or in a specification sheet placed within the shipping carton with the part. This program is intended to eliminate the uncertainty of the polymeric material identity in the end-use product and to reduce the possibility of field problems caused by the use of incorrect compounds.

1.3 The polymeric-material identity program covered by this standard is intended to provide traceability for molded finished parts (that is, enclosures, internal equipment parts, and the like) that are to be factory-installed components of other equipment where the acceptability of the combination is to be determined.

1.4 This program is not intended for manufacturing operations that add colorants or other additives to plastic materials using hot-compounding techniques that subject the material to an additional heat history and ship pellets as finished parts. This program is not intended to provide traceability for polymeric materials that are intended for field installation.

1.5 Requirements and methods for the evaluation of metallized or painted parts are contained in the Standard for Polymeric Materials – Use in Electrical Equipment Evaluations, UL 746C, and the requirements for Polymeric Materials – Short Term Property Evaluations, UL 746A. Reference should be made to the applicable individual product standard for performance requirements covering the part or assembly.

UL 746E (Deleted November 28, 2001) Standard for Polymeric Materials – Industrial Laminates, Filament Wound Tubing, Vulcanized Fibre, and Materials Used In Printed-Wiring Boards

UL 1020 (October 17, 1994) Thermal Cutoffs for Use in Electrical Appliances and Components

1 Scope

1.1 These requirements apply to thermal cutoffs intended to be embedded in windings or for freestanding use in end products.

1.2 The acceptability of a thermal cutoff in any particular device or appliance depends upon its acceptability for continued use under the conditions that prevail in actual service. For a particular application, a thermal cutoff may be affected by the requirements for the device or appliance in question, and it may be necessary to employ a thermal cutoff having features in addition to those specified in this standard. Compliance of a thermal cutoff with the requirements in this standard does not determine that the thermal cutoff is acceptable without further evaluation for use as a component in an end product.

1.3 A product that contains features, characteristics, components, materials, or systems new or different from those covered by the requirements in this standard, and that involves a risk of fire, electric shock, or injury to persons shall be evaluated using the appropriate additional component and end-product requirements to determine that the level of safety as originally anticipated by the intent of this standard is maintained. A product whose features, characteristics, components, materials, or systems conflict with specific requirements or provisions of this standard shall not be judged to comply with this standard. Where appropriate, revision of requirements shall be proposed and adopted in conformance with the methods employed for development, revision, and implementation of this standard.

1.3 revised November 10, 1998

UL 1446 (May 16, 1997) Standard for Systems of Insulating Materials – General

1 Scope

1.1 These requirements cover test procedures to be used in the evaluation of Class 120(E) or higher electrical insulation systems intended for connection to branch circuits rated **600 volts or less**. These requirements also cover the investigation of the substitution of minor components of insulation in a previously evaluated insulation system and also the test procedures to be used in the evaluation of magnet wire coatings, magnet wires, and varnishes.

1.2 These requirements do not cover a single insulating material or a simple combination of materials, such as a laminate or a varnished cloth, printed circuit boards, or planar transformers.

1.2 revised July 29, 2004

1.3 These requirements do not cover insulation systems exposed to radiation or operating in oils, refrigerants, soaps, or other media that potentially degrade insulating materials.

1.4 These requirements shall be modified or supplemented as determined by the applicable requirements in the end-product standard covering the device, appliance, or equipment in which the insulation system is used.

UL 1557 (May 29, 1997) Electrically Isolated Semiconductor Devices

1 Scope

1.1 These requirements apply to semiconductor devices of the isolated-mounting type – thyristors, transistors, diodes, and the like, and hybrid modules consisting of combinations of these devices where the voltage does not exceed 600 V ac rms or dc.

1.2 These requirements do not apply to snubber and commutation circuits associated with thyristors, transistors or other analog semiconductor devices.

1.3 These requirements cover the isolation performance of thyristors, transistors, diodes, and the like, and their combination in module packages and constructional features that are pertinent to that performance.

1.4 These requirements apply to isolated semiconductors for use as components in products. Compliance of an isolated semiconductor with these requirements does not determine that the semiconductor is acceptable for use as a component of an end product without further investigation. The acceptability of a semiconductor in any particular product depends upon its acceptability for continued use under the conditions that prevail in actual service.

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Title

Reliability of materials for the thermal management of electronics

Abstract

The main goal of this project was to research the properties and reliability of the thermal interface materials used in electronics. The selected materials for measurements and tests contained elastic pad sheets, phase change materials and greases. The thermal impedance at pressures of 0,1...0,8 MPa was measured from the 34 materials. From these 14 materials were selected for the reliability tests.

A new measurement method for thermal impedance and a new type of test structure for material testing was developed. The new test structure for materials was designed for use in environmental tests. The test structure makes it possible to select test surface quality, test pressure and material thickness.

The materials were tested in various environmental conditions (high/low temperature, temperature/humidity cycle, temperature cycling) to see how their properties change in these conditions and what is the life endurance in use.

The main criteria for evaluation of materials was the measured thermal impedance ($K \cdot cm^2/W$) at power densities from 10 to 25 W/cm^2. The high power density means that samples are heated during the measurements into the high operating temperature. Greases, phase change pads and graphite pads had the lowest impedance value.

The measured thermal impedance showed that the pressure and the wettability of materials have in many cases a larger effect on the impedance than the thermal conductivity of the bulk material. Therefore the impedance should be measured with the low pressures (< 0,3 MPa), which are achievable in practical solutions.

Quite important observation of the tests is that the variation from sample to sample in initial and final measurements is larger than the changes during the tests. Therefore the user should take care of the surface quality and contact pressure while calculating the total variation of thermal impedance for the whole life cycle of products.

Keywords
thermal interface material, electronics, measurements, thermal impedance, thermal conductivity, phase change materials, reliability, environmental testing

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Nimeke

Elektroniikan lämmönhallintamateriaalien luotettavuus

Tiivistelmä

Projektin tavoitteena oli tutkia elektroniikassa käytettyjen lämpöä johtavien materiaalien ominaisuuksia ja luotettavuutta. Tutkitut materiaalit olivat joustavia levyjä, faasimuutokseen pohjautuvia kalvoja ja rasvoja. Hankkeessa mitattiin 34 erityyppisen materiaalin lämpöimpedanssi pintapaineella 0,1...0,8 MPa. Näiden mittausten perusteella valittiin 14 materiaalityyppiä luotettavuustesteihin.

Materiaalien olosuhdetestejä varten kehitettiin uusi lämpöimpedanssin mittausmenetelmä ja siinä käytettävä testirakenne. Testirakenne mahdollistaa pinnan laadun, pintapaineen ja materiaalin paksuuden valinnan kulloisenkin tarpeen mukaan.

Materiaaleja testattiin useissa ympäristöolosuhteissa: korkea ja matala lämpötila, vaihteleva lämpö ja kosteus sekä pitkäaikainen lämpötilan vaihtelu. Tavoitteena oli saada tietoa materiaalien ominaisuuksien muutoksista ja pitkäaikaisesta stabiilisuudesta käyttöolosuhteita vastaavissa rasituksissa.

Lämpöimpedanssia käytettiin pääkriteerinä arvioitaessa materiaalien ominaisuuksia. Lämpöimpedanssi ($K{\cdot}cm^2/W$) mitattiin tehotiheyksillä 10…25 W/cm^2. Pienimmät lämpöimpedanssin arvot oli rasvoilla, faasimuutosmateriaaleilla ja grafiittikalvoilla.

Mittausten perusteella pintapaine ja materiaalien kyky mukautua asennuspintaan vaikuttivat useissa tapauksissa lämpöimpedanssiin enemmän kuin materiaalin lämmönjohtavuus. Tämän vuoksi lämpöimpedanssi tulisi mitata riittävän alhaisella paineella (< 0,3 MPa), joka on saavutettavissa käytännön jäähdytysratkaisuissa.

Materiaalien lämpöimpedanssin vaihtelu näytteestä toiseen oli saman materiaalin sisällä suurempi kuin testien aikana tapahtuneet muutokset. Tämä korostaa materiaalien huolellisen asennuksen, pintojen laadun ja pintapaineen hallinnan tärkeyttä arvioitaessa lämpöimpedanssin vaihtelua tuotteen koko elinaikana.

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The reliability of the thermal interface materials used in electronics was researched by VTT and ABB Oy Drives. The interface materials are vital for the thermal control of electronics. The materials searched were elastic pad sheets, phase change materials and greases. The thermal impedance and heat conductivity was measured first, and thereafter 14 materials were selected for the reliability tests.

A new method for the measurement of thermal impedance and a new type of test structure for material testing was developed. The new test structure for materials was designed for use in environmental tests. The test structure makes it possible to select test surface quality, test pressure and material thickness. Also a new model for specification of thermal interface materials was developed.

The materials were tested in various environmental conditions (high/low temperature, temperature/humidity cycle, temperature cycling) to see how their properties change in these conditions and what is the life endurance in use. The results showed that it is very important to design well the surface quality and contact pressure conditions of the thermal interfaces.

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