Experimental Evaluation of Glob-top Materials for use in Harsh Environments

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Abstract

This article presents results of experimental evaluation of glob-top materials for multi-chip-modules (MCM) in harsh environments. Material and process tests have been performed with the purpose to find a material which would fulfill the reliability requirements for use e.g. in military or automotive applications. Seven polymer materials, i.e. four epoxies, two silicones and one polyurethane material have been selected and evaluated in the experiments. The most critical material and process parameters for glob-top have been identified and measured. Based on the experimental results, application-based scoring of studied epoxy materials has been performed. Material evaluation results have been summarized in conclusions about the most suitable glob-top material for use in harsh environments.

Key words

Adhesion, Encapsulant, Fogging, Glob-top, Multi-attribute decision making, Multi-Chip-Module (MCM), Outgassing

1. Introduction

The use of multi-chip-modules (MCM) is of high importance for electronic products manufactured in small volumes. They give the possibility to use naked dies in combination with ordinary surface mount components. Additionally, in MCM having severe signal integrity requirements, via holes can easily be implemented. The product cost can therefore be reduced as the motherboard can be manufactured with less advanced technology. Furthermore, an upgrade of the product can be made by a simple modification of the MCM, e.g. through introduction of new functionality or die substitution.

One of the challenges with MCM is the choice of the encapsulant, which typically is a polymer material. The encapsulant can be applied on the dies by using two different methods, see Fig. 1. It describes the glob-top method and the dam and fill method. In a dam and fill solution, the dam is a barrier dispensed with a high viscosity encapsulant material in order to delimit the area to be filled with the low viscous fill material. Dam and fill gives lower total height and also poses the possibility to cover several dies. Within this study different fill materials were investigated, although referred to as glob-top.

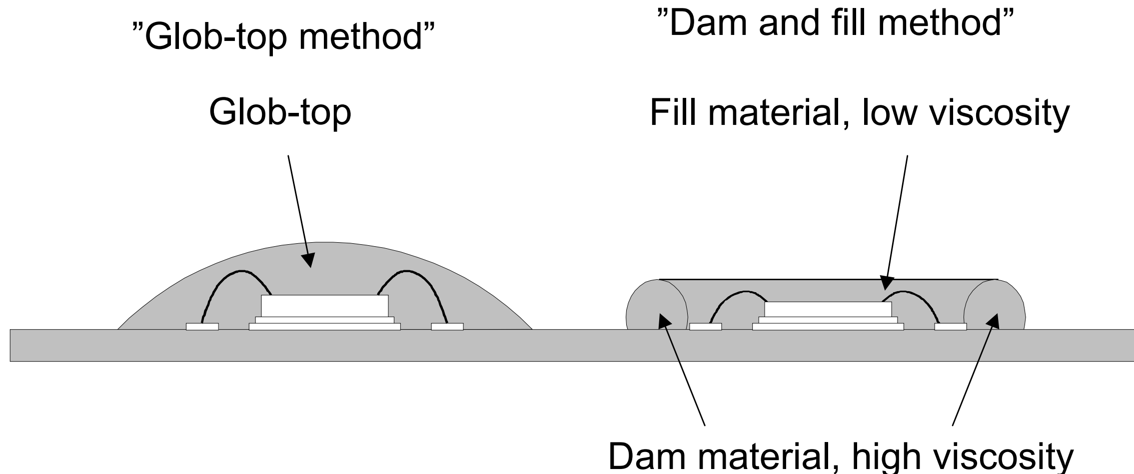

Fig. 1. Difference between the glob-top and the dam and fill methods.

By experience, it is known that it can sometimes be difficult to achieve the material properties that are presented by the material supplier. The most difficult requirement to fulfil is the adhesion between the glob-top and the die/PCB surface. Delamination can be caused e.g. by mismatch in the coefficient of thermal expansion (CTE). It is also dependent of other material properties, such as modulus of elasticity (E-modulus), glass transition temperature (T_g), etc.

This experimental study was targeted at better understanding of the material properties and process parameters that influence the reliability of the end product. Finding a reliable glob-top for use in a harsh environment was in focus. First, the most crucial material and process parameters for glob-top in a harsh environment were identified by an extensive literature study. Materials available on the market were then selected for experiments. The measured results were analyzed by two scoring methods, the simple additive weighting (SAW) method and the technique for order preference by similarity to ideal solution (TOPSIS), in order to perform application-based ranking of the studied epoxy materials.

2. IDENTIFICATION OF CRUCIAL GLOB-TOP PROPERTIES AND PROCESS PARAMETERS

Glob-top materials of interest were evaluated for use in a harsh environment that corresponded to a military or automotive under-the-hood application. In several studies moisture absorption was reported as having a large influence on the product reliability [1]-[5]. It was concluded in [2] that there was a relation between the moisture absorption and physical properties, such as T_g and CTE. In [1] and [6] opinions differ about the most suitable glob-top material for harsh environments, epoxy or silicone.

Process related questions investigated in [7] and [8] were needle- and substrate temperatures and the time for melting before application, in order to minimize the number of voids in the glob-top. Changing the curing temperature can further control the vertical position of the voids. In [9], it was claimed that the filler material in epoxy might cause breaking of the bond wires during temperature cycling. On the basis of FEM simulations, it was stated in [10] that uneven distribution of filler particles around the bond wires can cause failures during temperature cycling. The relation between die attach adhesive and encapsulant material was investigated in [5]. It was found that different combinations of die attach and encapsulant would influence the resistance to high temperature/moisture test and to high reflow temperatures.

The influence of altered curing profile on glob-top was studied in [11]-[14]. According to [11], it is interesting to investigate whether changing the curing time and curing temperature can optimize T_g. If a two-step curing profile with pre-curing is used the built-in stress can be reduced [11], [14]. Stable and controlled curing profile is therefore an important process parameter in order to obtain the optimal T_g and reduce the built-in stresses. It is also worth noting that the adhesion between the glob-top and the substrate/die can be improved by cleaning the surfaces before application of the glob-top [11], [12]. Other aspects that are important to recall in evaluation and selection of glob-top is the material of bond wires, i.e. aluminum or gold, and the type of laminate, i.e. ceramic or organic. It follows from [15] that organic laminates are preferable for

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use with an epoxy glob-top.

During thermal cycling it is necessary to consider both CTE and E-modulus according to (1), [9]:

$$\sigma_T = (\alpha_{adh} - \alpha_{sub}) * \Delta T * E_{avg} \quad (1)$$

where:

σ_T = shear stress due to encapsulant;

α_{adh} = CTE of encapsulant;

α_{sub} = CTE of substrate;

ΔT = temperature range of test;

E_{avg} = the average E-modulus of the encapsulant over the temperature range

Equation (1) shows that E-modulus has an important role in stress related failures. Large mismatch between CTE of the substrate/die and the glob-top can to certain extent be compensated by low E-modulus of the glob-top material. Thus, it is important to consider both E-modulus and CTE during material selection. When selecting e.g. an epoxy material, CTE mismatch must be minimized and T_g should be above or below extremes of the usage temperature. A controlled process is also important with respect to thawing, dispensing and curing.

3. Selection of Glob-Top Materials for Experiments

In order to find suitable glob-top materials for tests in harsh environment, material suppliers were contacted and asked to suggest materials. The parameters studied in the data sheets were E-modulus, CTE, and T_g. One of the requirements was that the T_g should not be within the temperature range of use (–40°C to +125°C). Another requirement was that the encapsulation should cover several dies, i.e. dam and fill solutions must be available. BT-laminate was used as the substrates in the experiments. The choice of materials should also be based on that the substrates in future applications will be assembled with silicon dies and wire bonded with gold or aluminum wires.

Three types of polymer materials, epoxy, polyurethane, and silicone are normally used as encapsulants due to their adequate though different material properties. Epoxy is a thermoset, i.e. after curing the molecules are irreversible cross-linked. In general, it is a hard material, if not modified, and mostly presents lower rate of moisture penetration compared to silicone materials. On the other hand, silicone materials usually have lower ion content. For the curing of epoxies several hardening systems exist. The three most usual are anhydride acids, amines, and phenols. Anhydride systems are most frequently used as hardening system in glob-top, followed by the amine system. Phenolic curing systems are often used in molding compounds. The curing of a phenol system is slow and requires prolonged post mold curing for hours. The mechanical properties of the phenol systems are the worst among the three mentioned hardening systems [16], i.e. high CTE value and low T_g.

Silicone materials are soft at room temperature with an approximate T_g of about -60°C, i.e. the material is in rubbery state in the temperature range from –40°C to +125°C. Silicone materials have good electrical properties, and provide almost no mechanical protection. Moisture quite easily penetrates silicone. However, the Si-O-Si-bonds to the substrate surface prevent corrosion. CTE of silicone materials is high, hundreds of ppm/°C. However, the strain on bond wires is low, due to the low E-modulus of the silicone. One weak point of silicones is that they are sensitive to solvents, and another weakness is the risk for silicone contamination [17].

Polyurethane is a major part of most conformal coatings. Polyurethane has good adhesion to surfaces and can also be used for stress relief. The polyurethane materials identified during the material search can be cured with UV and visible light.

It is worth noting that materials containing anhydride acids listed in Appendix 3 in [18] were rejected without further judgment on their materials properties. In order to use these anhydride acids permission from the Swedish Work Environment Authority is required. Only one glob-top material was selected from each supplier in order to be able to evaluate several suppliers. The final choice of materials was made on the basis of existing knowledge, industrial experiences and recommendations from the material suppliers.

At last, four epoxy materials, one polyurethane material, and two silicone materials were chosen for further material and process qualification. The materials were named Epoxy A, Epoxy B, Epoxy C, Epoxy D, Polyurethane A, Silicone A, and Silicone B. One of the epoxy materials (Epoxy D) contained anhydride acids and was included as a reference material since it was already used in production. The materials selected for the experiments and their CTE and T_g values are presented in Table I. Typical E-modulus values for epoxy glob-top are several GPa, whereas for silicone glop-top values are several MPa.

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Table I
Glob-top materials selected for experiments.

Material	CTE below/above T_g, (ppm/°C)	T_g, (°C)	Main substance
Epoxy A	77/-	62	Modified epoxy; Hydroxyterminated 1,3 Butadiene homopolymer and 4-(vinyloxy)butan-1-ol
Epoxy B	55/203	60	Bisphenol F epoxy resin and polyoxypropyleneamine
Epoxy C	19/-	145	3,4-Epoxycyclohexylmethyl 3',4'-epoxycyclohexanecarboxylate
Epoxy D (reference)	19/-	160	Anhydride containing epoxy
Polyurethane A	100/250	25	Modified urethane, isocyanate free
Silicone A	-/240	-	Silicone
Silicone B	Approx 300	-	Silicone, low outgassing

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4. Experiments

Environmental test specification was based on military requirements including temperature limits from –40°C to +125°C and a relative humidity (RH) of at least 85%. The experimental work on the glob-top materials included measurements of moisture uptake, adhesion, CTE, T_g, outgassing, and fogging.

Test samples with the different materials were prepared according to the scheme presented in Fig. 2. Curing profiles for each material (the actually used and the one recommended by the material supplier) are presented in Table 2. In some specific tests mentioned below other curing profiles were used. The reason for this was that the wavelength of the UV-lamp recommended could not be used in the laboratory. The suppliers were contacted and modified curing profiles were implemented.

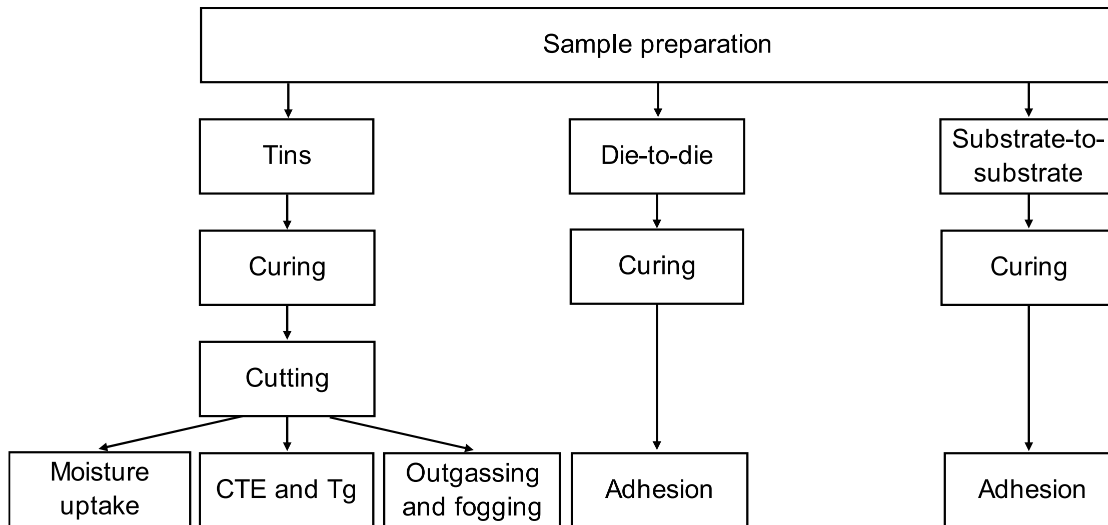

Fig 2. Scheme for sample preparation.

4.1 Moisture uptake

Moisture content in the glob-top material can cause

corrosion of the die surface, thereby affecting the reliability of the final product. The moisture uptake test included weight measurement of material samples after curing, and after treatment for 1000 hours either in 120°C environment (heat environment) or in 85°C/85%RH environment (moisture environment). Five samples were prepared with each glob-top material. Uncured samples with the weight 0.5 g - 0.7 g were cured in aluminum tins. The samples in the tins were weighed after curing. Two samples of each material were placed in heat environment for 1000 hours and the other three were treated in moisture environment during the same time. After the treatments, the samples were weighed again. Empty aluminum tins were placed in each environment as reference. The weight loss by heat treatment indicates the amount of moisture removed from the sample, whereas the weight increase after the treatment in moisture indicates moisture absorption in the sample. For this test, weight scales Sartorius BP210 ensuring an accuracy of ± 0.1 mg was used.

4.2 Adhesion

The adhesion of glob-top was tested both to the substrate (BT-laminate), see Fig. 3, and to the silicon dies. Five small pieces of substrate (Substrate B) sized to approx. 3 mm × 3 mm were glued onto a larger substrate (Substrate A) with the materials to be evaluated. The smaller substrates were sheared off, and the shear force was registered.

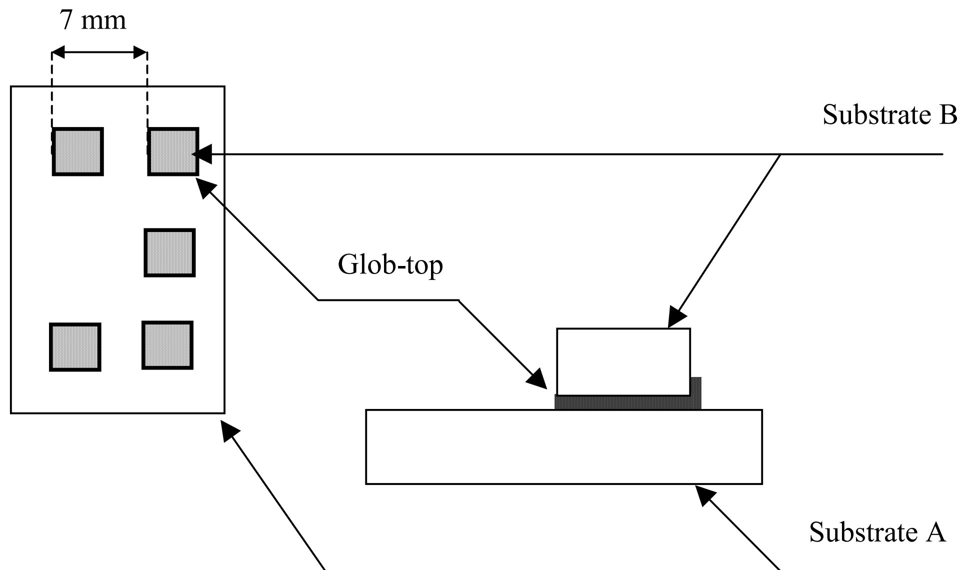

Fig. 3. Sketch of the test board for the substrate-to-substrate adhesion test.

The evaluation of the adhesion to the die surface was performed by gluing small dies (1.6 mm × 1.7 mm [1.4 mm × 1.4 mm for Epoxy C]) with respective glob-top materials on a larger die. The larger die was then glued using die attach materials recommended by suppliers to a substrate. During the adhesion test, the smaller die was sheared and the shear force was registered. The adhesion test was performed on samples after curing and after treatment during 1000 hours either in heat environment or in moisture environment. A bond tester Dage BT 2400PC was used for the adhesion tests.

4.3 Thermo-mechanical analysis

Samples were prepared by dispensing material into the aluminum tins and cured according to the actual curing profiles presented in Table 2. The Epoxy A material was found not to be cured, i.e. it was still in liquid phase after the treatment. Following discussions with the supplier the material was cured at 100°C for another hour. After completing the curing the samples were sawed in pieces to a size of approx. 5 mm × 5 mm × 3 mm. These samples were studied with thermo-mechanical analysis (TMA), Shimadzu TMA-50. The samples were heated from room temperature to 200°C with a rate of 5°C/min. The displacement was measured and plotted as a function of temperature, and from the graph CTE and T_g were determined. Two samples of each material were analyzed and each sample was analyzed during three runs. The first run for each sample was not used in the analyses as post-curing could happen during this run.

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Table 2. Curing profiles

Material	Recommended profile	Actual profile
Epoxy A	60 s UV-A 100 mW/cm^2	20 s UV-A 225mW/cm^2 + 2 h @60°C +1 h 100°C
Epoxy B	1 h @ 80°C	2 h @ 80°C
Epoxy C	3-5 min UV-A	2 min UV-A 225mW/cm^2 + 1 h @ 80°C
Epoxy D	30 min @ 125°C + 90 min @ 165°C	30 min @ 125°C + 90 min @ 165°C
Polyurethane A	10-30 s UV-A	20 s UV-A 225mW/cm^2 + 5 min @ 120°C
Silicone A	30 min @ 150°C	30 min @ 150°C
Silicone B	40 min @ 130°C	40 min @ 130°C

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4.4 Outgassing and fogging

The samples similar to those used in the previous test were prepared. The test method was according to a modified version of ASTM-E595-83 standard. First, conditioning was performed for 24 hours in approx 50% RH environment. The equipment consisted of a metal block with four channels where glass vessels with samples could be placed. The block was held at a temperature of 85°C. The lid on the metal block was cooled by water. Inside the lid metal mirrors were placed over the glass vessels. The temperature on the mirror was 10°C and the test time was 48 hours. The fogging on the mirrors was analyzed with FT-IR method. During the test, the total mass loss (TML) and the collected volatile condensable material (CVCM) were measured. After conditioning, the part of water in the total mass loss, i.e. water vapor regained (WVR), was measured by conditioning in the 50% RH environment. The actual material weight loss, which is the difference between TML and WVR of the sample, was also calculated. Target value for accepted material was TML ≤ 1% and CVCM < 0.1%. Additionally a subjective visual judgment was made on the appearance of the fogging on the mirrors (see Table 3). The judgment from experts was graded from 1 (accepted without remarks) to 4 (not approved).

Table 3. Visual judgment on fogging appearance

Grade	Comment	Judgment
1	No or insignificant fogging	Approved
2	Little fogging, thin film	Approved
3	More spread fogging (depends on whether the fogging evaporates)	Barely approved
4	Great deal of fogging	Not approved

5. RESULTS

5.1 Moisture uptake

The weight of the silicone materials was not changed after treatment in heat environment or in moisture environment. The polyurethane material showed the largest change of weight. The weight was reduced after the treatments in moisture environment and in heat environment. All the epoxy materials showed less change in weight. The reference material (Epoxy D) was least influenced and Epoxy B exhibited largest weight change among the epoxies. The results of this experiment are presented in Fig. 4. The weight percentage was calculated as the ratio of the measured weight difference to the original weight of the sample, i.e. to the weight measured after curing and before heat or moisture treatment. It was expected that the materials would loose weight during heat treatment due to moisture release. The moisture environment was suspected to saturate the samples with moisture, i.e. increase the weight. However, the polyurethane material lost in weight in both experimental environments. A possible explanation of this can be the degree of curing of the polyurethane material. The polyurethane material should therefore be studied more carefully before it can be used as glob-top for applications in harsh environments.

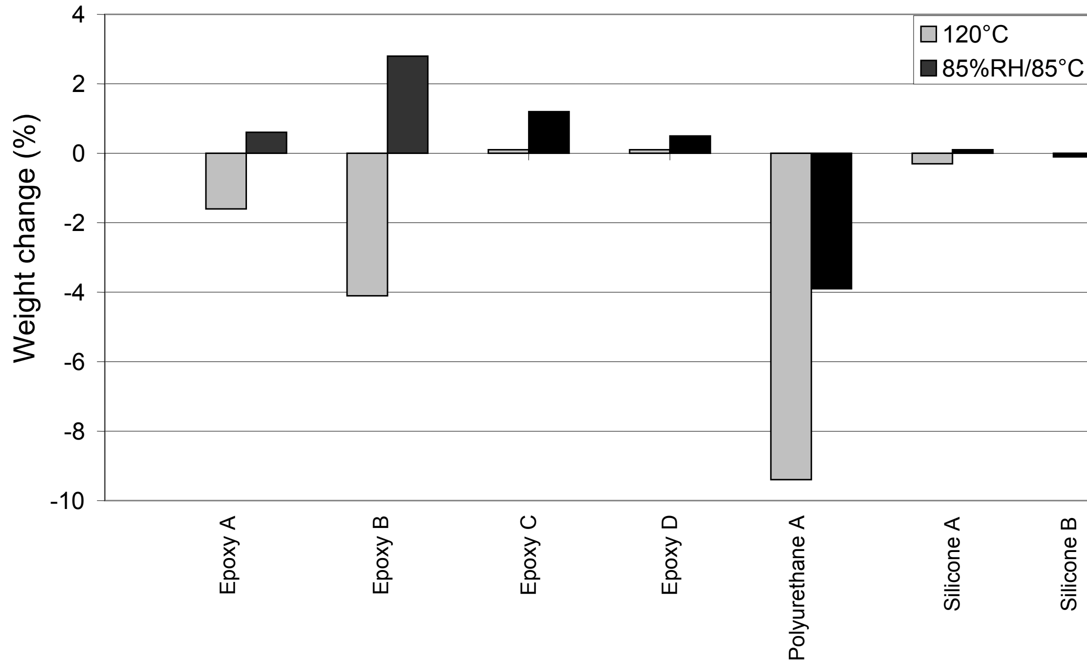

Fig. 4. Change of weight after treatment in the heat and moisture environments.

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5.2 Adhesion.

The results of the shear force measurements in the adhesion tests are presented in Fig. 5 and Fig. 6. The shear force measured for the two tested interfaces increased after heat treatment. The measured shear forces were also lower on the substrate-to-substrate samples compared to die-to-die surface. Neither heat environment nor moisture environment decreased the shear force compared to the results obtained directly after curing.

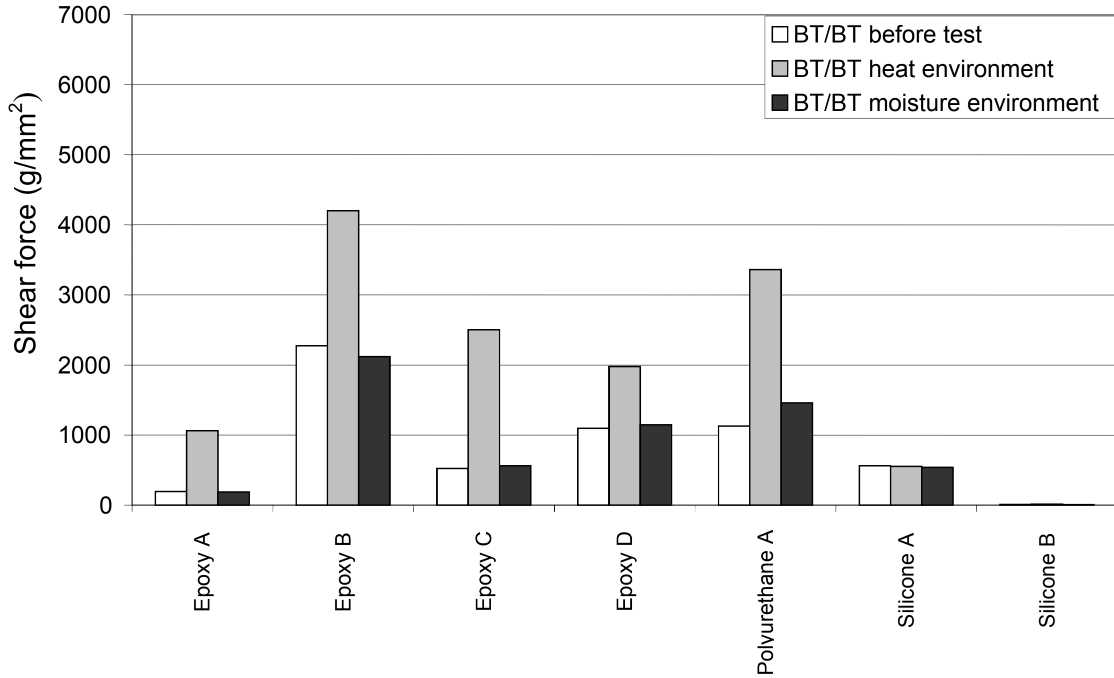

Fig. 5. Shear force measured on BT substrates.

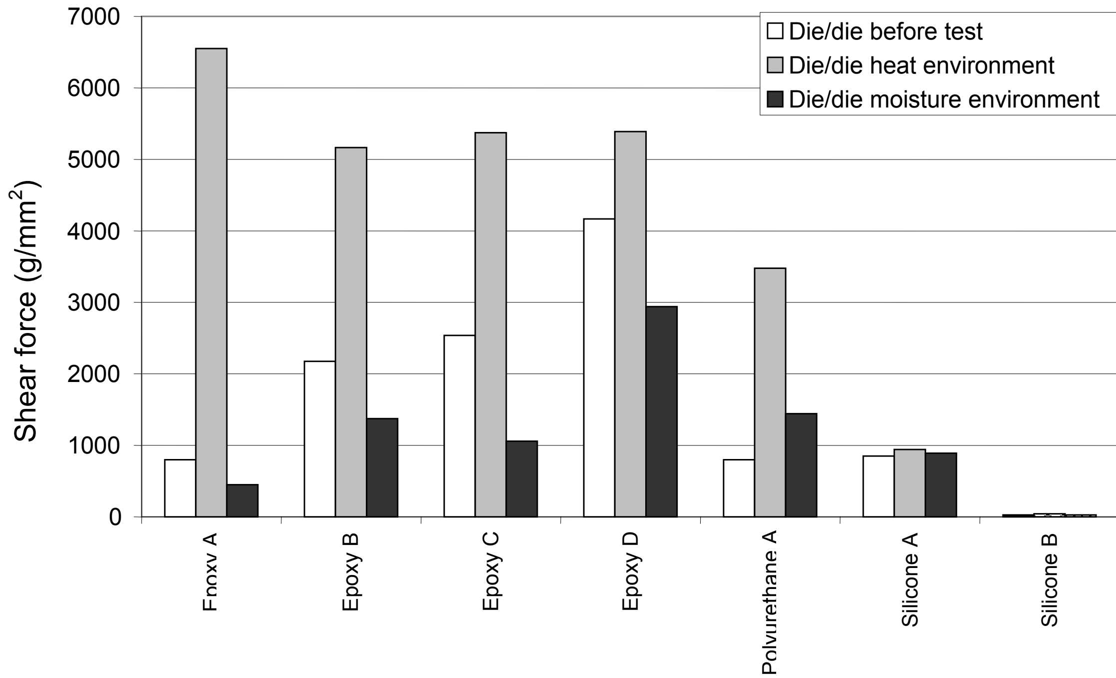

Fig. 6. Shear force measured on dies.

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5.3 Thermo-mechanical analysis

In Table 4 CTE and T_g of the glob-top materials are presented, both measured and those according to specification. The average values of the measured CTE and T_g were calculated as according to (2) and (3)

$$CTE = \frac{CTE_{run2}^{sample1} + CTE_{run3}^{sample1} + CTE_{run2}^{sample2}}{3} \quad (2)$$

$$T_g = \frac{T_{g,run2}^{sample1} + T_{g,run3}^{sample1} + T_{g,run2}^{sample2}}{3} \quad (3)$$

The T_g value for Epoxy C could not be registered, as no transition region for CTE was observed. Therefore, only the low temperature CTE is presented. The measurement results for the reference epoxy were found to be most close to the specification. Polyurethane A also followed the specification quite well, whereas Epoxy A and Epoxy B did not follow the specification. In case of Silicone B the test method was not applicable for measuring the CTE and T_g, due to the very low E-modulus. It is worth noting that measurements of CTE and T_g were performed also on dam materials suited for the selected epoxies. In fact, the measured CTE and T_g values on the dam for Epoxy C indicated results similar to the specification.

5.4 Outgassing and fogging

In Table 5, results from the outgassing and fogging tests are presented. The weight percentage (wt %) was calculated as the ratio of the measured weight difference to the sample weight before the test. The table also includes the results of visual judgment, which was based on expert knowledge.

The tests revealed four glob-top materials, Epoxy C, Epoxy D, Silicone A, and Silicone B that would be accepted for applications where low outgassing and fogging is required. Among these, only Epoxy D and Silicone B were accepted without remarks.

Table 4. Results of TMA

Material	CTE according to spec., below/above T_g (ppm/°C)	CTE measured, below/above T_g (ppm/°C)	T_g according to spec. (°C)	T_g measured (°C)
Epoxy A	77/-	-/152	62	34
Epoxy B	55/203	-/214	60	35
Epoxy C	19/-	2/-	145	-
Epoxy D	19/-	19/68	160	140
Polyurethane A	100/250	-/239	25	-
Silicone A	-/240	-/289	-	-
Silicone B	-/Approx. 300	-	-192	-

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Table 5. Results from outgassing and fogging tests

Material	TML (%)	CVCM (%)	WVR (%)	Actual weight loss (%)	Visual judgment	FT-IR spectrum
Epoxy A	0,56	0,14	0,21	0,35	4	Organic acid or ester + alcohol
Epoxy B	0,86	0,017	0,52	0,34	4	Aromatic compound + alcohol
Epoxy C	-0,003	-0,002	0,08	-0,08	2	Spectrum with noise
Epoxy D	0,20	0,05	0,14	0,06	1	—
Polyurethane A	2,34	0,16	0,80	1,54	4	Aromatic compound + alcohol
Silicone A	0,05	0,05	0,02	0,03	2	Silicone oil
Silicone B	0,08	0	0,08	0	1	—

6. Discussion and Analyses

The first part in the discussion and analyses includes interpretation of the experimental results. Based on expert knowledge in material and process technology the tested materials are compared to one another with respect to each measured parameter. The second part is based on mathematical decision-making methods where cumulative effect of the measured parameters on the material choice is taken into consideration.

6.1 Choice of glob-top material based on individual parameters.

Comparing the different material types is not easy as they exhibit quite different properties. Epoxy materials can to some extent be compared with polyurethane, whereas silicone is used in different applications.

The results from the treatment in moisture environment indicated that moisture uptake in Epoxy A and Epoxy D was quite low, whereas the moisture uptake in Epoxy B was rather high. During the treatment in heat environment the weight decreased considerably for Epoxy A and Epoxy B, whereas the weight of Epoxy C and Epoxy D was almost unaffected. Concerning the polyurethane material, it lost in weight in both experimental environments, whereas the silicones were almost unaffected. Based on the weight change results, Epoxy D would be recommended as the best choice among the epoxies.

The results from adhesion tests on BT substrates after treatment in the two environments gave the following order of preference for the epoxies: B, D, C, and A. The large change in shear force after heat treatment can most probably be related to post curing, which is the normal behavior of epoxies. The shear force measured on the polyurethane was relatively high, which allows placing this material in the preference list between Epoxy B and Epoxy D. Regarding the adhesion tests with silicones the used test methodology is not the most suitable, due to the low E-modulus. Although the results can be used in order to compare different silicone materials.

The shear force on the die surfaces was in general higher compared to the BT substrates. This is a good sign, as most of the time delamination is initiated in the die to glob-top interface. The order of preference for the epoxy materials tested on the die surfaces was different compared to the BT substrates. Epoxy D was judged to pass the test with the highest degree of preference followed by Epoxy B and Epoxy C, which could be equally preferred, due to the low difference in shear force. The shear force for the polyurethane material was independent of the surface. Epoxy B or Epoxy D would thus be recommended as the best choice of epoxies based on the adhesion test results in both environments.

According to the TMA results, Epoxy A and B were not considered as acceptable options of glob-top materials. It was due to the low T_g and therefore the large discrepancy between material specification and the measured results. The recommendation would therefore be to use Epoxy D or Epoxy C.

If sensitivity to outgassing and fogging were crucial for a particular application, the best choices would be Silicone B or Epoxy D. These would be followed by Silicone A and Epoxy C. The amount of outgassing from Epoxy A, Epoxy B and Polyurethane A should be considered too high.

It is worth noting that curing conditions were well monitored in the performed experiments. However, a general recommendation is that during qualification of glob-top materials, and maybe of other polymers as well, a verification procedure of curing degree should be performed.

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6.2 Application-based glob-top material ranking

Interpretation of the experimental results can be simplified by using scoring methods in order to decide which material is preferable for a particular application. Decision making, in this case, was complicated due to the high number of parameters or attributes that had been analyzed in the experiments. Two methods, the simple additive weighting method (SAW) and the technique for order preference by similarity to ideal solution (TOPSIS), were used in order to rank the studied materials [19]. These two scoring methods were used together for ensuring the validity of the obtained decision regarding the choice of materials at given weighting coefficients derived from expert knowledge. Scoring was implemented for the four epoxy materials, A, B, C and D (see Table 6). The silicone-type materials and polyurethane were excluded, as different material types could not be compared easily.

Table 6. Data for glob-top material scoring.

Attribute number	Attribute type	Attribute name	Attribute magnitude	Epoxy A	Epoxy B	Epoxy C	Epoxy D
1	min	Weight change @ Heat, 120°C	wt %	1.6	4.1	0.1	0.1
2	min	Weight change @ 85%RH/85°C	wt %	0.6	2.8	1.2	0.5
3	min	Difference in CTE	∆CTE, ppm/°C	73	51	15	15
4	max	Adhesion BT @ Heat, 120°C	Shear force, g/mm^2	1060	4201	2503	1978
5	max	Adhesion die @ Heat, 120°C	Shear force, g/mm^2	6551	5164	5374	5389
6	max	Adhesion BT @ 85%RH/85°C	Shear force, g/mm^2	186	2118	562	1146
7	max	Adhesion die @ 85%RH/85°C	Shear force, g/mm^2	449	1374	1058	2940
8	min	(TML)	wt %	0.56	0.86	0.003	0.20
9	min	(CVCM)	wt %	0.14	0.017	0.002	0.05
10	max	(WVR)	wt %	0.21	0.52	0.08	0.14
11	min	Visual judgment	grade	4	4	2	1

Eleven attributes were considered in the decision making process, which corresponded to the material parameters measured in the experiments. All attributes were supposed to be either minimizing (referred to as cost attributes) or maximizing (referred to as benefit attributes). For the two moisture uptake tests (attributes 1 and 2) and for the outgassing and fogging test (attributes 8, 9, 10) the attribute values were reported as weight percentage (wt %), corresponding to the ratios of the absolute value of the measured weight change to the original weight of the sample. An additional attribute (attribute 11) considered for the outgassing and fogging test was the visual judgment on fogging appearance. The four adhesion attributes were considered, with data related both to heat environment and to moisture environment. They included the shear force measured for the BT substrates (attributes 4 and 6) and for

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the dies (attributes 5 and 7). One more attribute (attribute 3), ΔCTE (difference between the silicon die CTE = 4 ppm/°C and the glob-top CTE), was included in the SAW and TOPSIS analyses. It was decided to use this attribute as the delamination most of the time appears between the die and the glob-top in the applications.

A score in the SAW method was obtained by adding contributions from each attribute, scaled to a common numerical system by applying linear normalization. The total score for each alternative was obtained by multiplication of each attribute rating by the importance weight assigned to the attribute, and then by addition of these products for all the attributes [19]. The basic concept of the TOPSIS analysis is that the selected alternative should have the shortest distance to the ideal solution and the longest distance from the negative-ideal solution in a geometrical sense. However, the ideal solution is usually unattainable. Thus, being as close as possible to such an ideal solution is the rationale of human choice [19, 20].

In both scoring methods the importance weights for the decision attributes were assigned as shown in Table 7. Being added, the weight values would give unity. The difference between the base application and the optical application is that the optical application would include mirrors and other optical systems.

Table 7. Application-based importance weights for the decision attributes

Criterion-based partitioning	Attribute weight	Base application	Optical application
Moisture uptake	w_1	0.08	0.08
	w_2	0.12	0.07
TMA	w_3	0.25	0.2
Adhesion	w_4	0.0825	0.0825
	w_5	0.0925	0.0925
	w_6	0.0825	0.0825
	w_7	0.0925	0.0925
Outgassing and fogging	w_8	0.03	0.05
	w_9	0.04	0.05
	w_{10}	0.03	0.05
	w_{11}	0.1	0.15

The weight assignment was done based on expert knowledge. The eleven attributes fell into four major criteria (moisture uptake, thermo-mechanical, adhesion, outgassing and fogging). These four criteria were given the importance values 20%, 25%, 35% and 20%, respectively, for the base application. The corresponding values for the optical application were 15%, 20%, 35%, 30%. Within each of four major criteria the weights were then assigned to the corresponding attributes. The moisture uptake attributes (attributes 1 and 2) were considered to influence the reliability. However, w_2 held a higher value due to risk for corrosion, if the glob-top would contain a high level of moisture. On the other hand, w_1 can more or less be related to outgassing. Compared to moisture uptake, the thermo-mechanical performance (attribute 3) was more important as it would influence the risk for delamination. On the other hand, delamination would also depend on the adhesion (attributes 4-7). High CTE can be compensated by a good adhesion. Therefore, the adhesion attributes together were considered more important and were given the larger importance value. The outgassing and fogging attributes (attributes 8-11) would have large influence on the reliability of optical systems, where they were assigned higher weights as compared to the base application.

Here, the steps of the SAW analysis are described in detail, and the results from the TOPSIS analysis are provided for comparison purposes. In SAW analysis, cost attributes were transformed to benefit attributes by taking inverse ratings, resulting in the following decision matrix $D = [d_{ij}]$, i = 1, ..4, j = 1, …, 11:

$$D = \begin{bmatrix} 0.625 & 1.667 & 0.014 & 1060 & 6551 & 186 & 449 & 1.786 & 7.143 & 0.21 & 0.25 \\ 0.244 & 0.357 & 0.020 & 4201 & 5164 & 2118 & 1374 & 1.163 & 58.824 & 0.52 & 0.25 \\ 10.0 & 0.833 & 0.067 & 2503 & 5374 & 562 & 1058 & 333.3 & 500.0 & 0.08 & 0.5 \\ 10.0 & 2.0 & 0.067 & 1978 & 5389 & 1146 & 2940 & 5.0 & 20.0 & 0.14 & 1.0 \end{bmatrix}$$

In order to obtain commensurable scales for each attribute the elements of the decision matrix D were normalized:

$$r_{ij} = d_{ij} / d_j^*, \tag{4}$$

where $i = 1, …, 4; j = 1, …, 11$, and d_j^* is the maximum value of the jth attribute. Thus, the normalized matrix $R = [r_{ij}]$, $i = 1, …, 4, j = 1, …, 11$ would be

$$R = \begin{bmatrix} 0.0625 & 0.8333 & 0.2055 & 0.252 & 1.0 & 0.088 & 0.153 & 0.005 & 0.014 & 0.404 & 0.25 \\ 0.0244 & 0.1786 & 0.2941 & 1.0 & 0.788 & 1.0 & 0.467 & 0.0035 & 0.118 & 1.0 & 0.25 \\ 1.0 & 0.4167 & 1.0 & 0.596 & 0.820 & 0.265 & 0.36 & 1.0 & 1.0 & 0.154 & 0.5 \\ 1.0 & 1.0 & 1.0 & 0.471 & 0.823 & 0.541 & 1.0 & 0.015 & 0.04 & 0.269 & 1.0 \end{bmatrix}$$

The value of alternative A_i was then computed as

$$V(A_i) = \sum_{j=1}^{11} w_j r_{ij}, \quad i = 1, …, 4. \tag{5}$$

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The higher the value V(A_i), the higher rank or higher degree of preference would be assigned to the alternative A_i. The alternative values and the assigned ranks (1 is the highest rank, and 4 is the lowest rank) are presented in Table 8. The results from the TOPSIS analysis can be seen in Table 8 as well as the SAW results.

It can be seen from Table 8 that the SAW and TOPSIS analyses gave similar results, and that Epoxy D (rank 1) would have the highest degree of preference, and Epoxy A (rank 4) was treated the worst material to choose in the considered applications.

Table 8. Results of SAW and TOPSIS analyses

	SAW, base	SAW, base	SAW, optical	SAW, optical	TOPSIS, base	TOPSIS, base	TOPSIS, optical	TOPSIS, optical
Material (Alternative)	Alt. value	Alt. rank	Alt. value	Alt. rank	Alt. value	Alt. rank	Alt. value	Alt. Rank
Epoxy A (A_1)	0.3289	4	0.2978	4	0.3231	4	0.2679	4
Epoxy B (A_2)	0.4379	3	0.4480	3	0.4000	3	0.4234	3
Epoxy C (A_3)	0.6848	2	0.6721	2	0.6925	2	0.6547	2
Epoxy D (A_4)	0.8122	1	0.7683	1	0.8019	1	0.7598	1

Being easy-to-use methods both TOPSIS and SAW were found appropriate to the considered problem, since they reflect the values of the decision maker. It was confirmed by similarity of the ranking results obtained by the two methods. Decisions generated by these methods were in good agreement with the unaided expert judgments based on individual parameters.

In order to draw final conclusions about the obtained scoring of the glob-top materials, a sensitivity analysis was performed. Such an analysis was aimed at checking robustness of the final decision to variation of the weights, which were the subjective parameters involved in the decision making process. The sensitivity analysis was performed for SAW results. For each fixed value of the weight $w_k \in (0; 1)$, k = 1,…,11, the other ten weights were calculated under assumptions that $w_1 + w_2 + \ldots + w_{11} = 1$, and $w_i / w_j = constant$ for i, j ≠k, and i, j = 1,…,11. It meant that the robustness of the decision was checked when varying only one weight w_k at a time, while calculating the other weights in such a way that the ratio between them would be held constant, based on the data from Table 7 for the base application. For example, if the weight $w_{11} = b_0 \in (0; 1)$ is varied, then the system of linear equations would be solved in order to find the other weights, as follows:

$$\begin{cases} w_1 + w_2 + \ldots + w_{11} = 1 \\ w_1 - 0.667w_2 = 0; \\ w_2 - 0.48w_3 = 0 \\ w_3 - 3.03w_4 = 0 \\ w_4 - 0.892w_5 = 0 \\ w_5 - 1.121w_6 = 0 \\ w_6 - 0.892w_7 = 0 \\ w_7 - 3.083w_8 = 0 \\ w_8 - 0.75w_9 = 0 \\ w_9 - 1.33w_{10} = 0 \\ w_{11} = b_0 \end{cases} \quad (6)$$

It was thus assumed that the relative weight assignment based on expert knowledge was done properly and the final decision could be influenced only by varying, say, weight w_{11} which was linked to the other weights only through the first equation in (6). Results of the sensitivity analysis for the base application are plotted in Fig. 7. The thick vertical lines in the plots indicate the weight value from Table 7 initially used by the expert. In fact, it is important that the order of preference among the alternatives would be "stable", being just in the reasonable vicinity of these vertical lines, which could compensate for possible uncertainty in the subjective judgment about a particular importance weight. It can be seen from the plots for varying weights w_8 and w_9 that the vertical line is quite close to the encircled area where the order of preference for the two best alternatives (Epoxy D and Epoxy C) is changed. Thus, the assignment of the weights for outgassing and fogging attributes 8 and 9 should be carefully verified. Similar

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analysis can be applied to weight w_2 if the order of preference among the worse alternatives (Epoxy A and Epoxy B) would be considered important for some reasons.

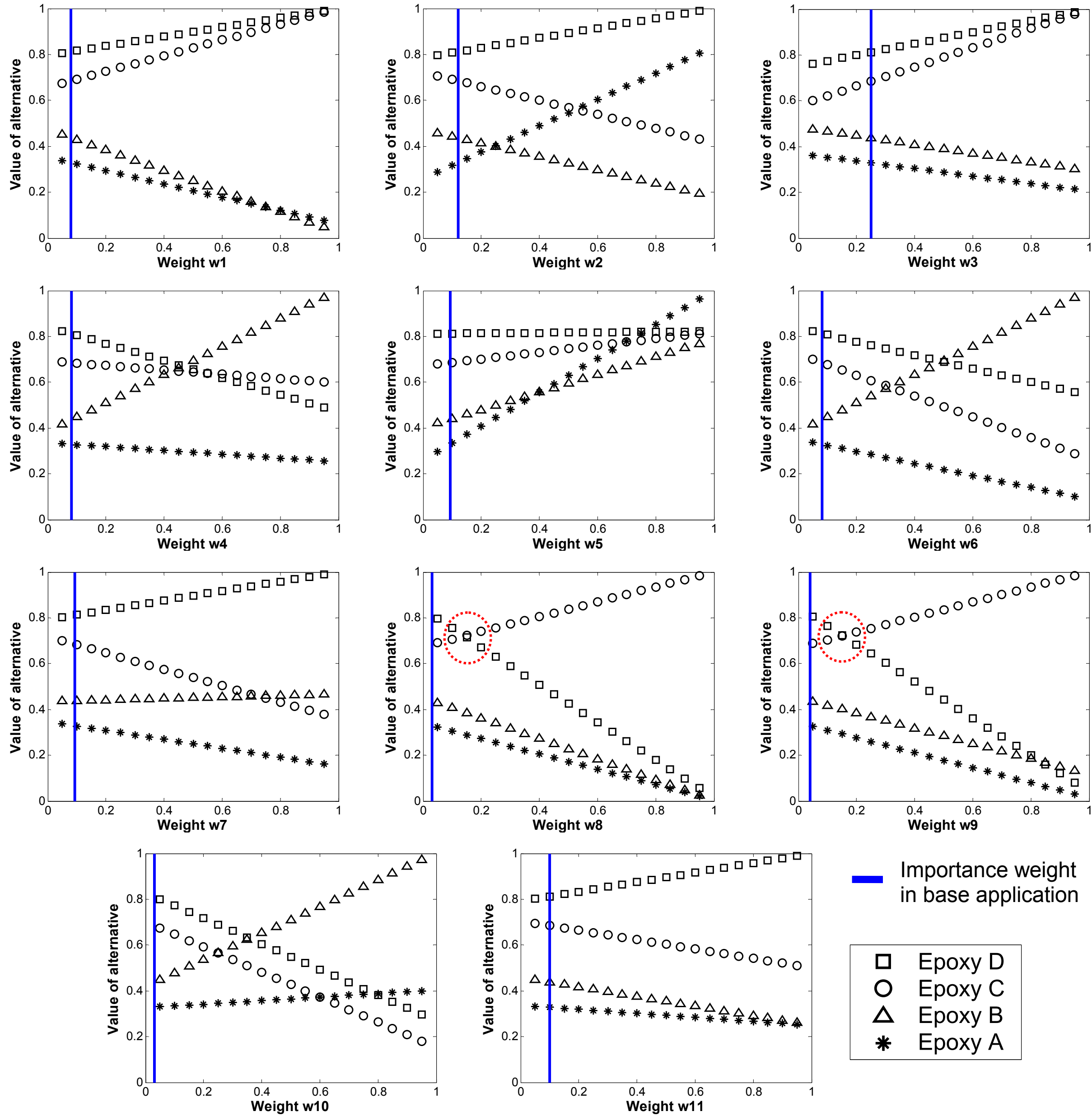

Fig. 7. Sensitivity analysis of the SAW results for the base application

Finally, the sensitivity analysis indicated the robustness of the decision to prefer Epoxy D and Epoxy C as the best alternatives for the considered applications at the available level of expert knowledge.

7. CONCLUSIONS

The experimental methodology and the experimental results presented in this paper enabled the application-based scoring of the glob-top materials for reliable service in harsh environments. The glob-top materials available on the market were initially chosen for experiments according to their E-modulus, CTE, and T_g, the properties that were

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identified as crucial for the reliability of the end product. Influence of process parameters, such as curing profile, on the product reliability was evaluated and taken into account during the experiments.

Experiments were performed on four epoxy materials, one polyurethane material, and two silicone materials. They included moisture uptake test, TMA test, adhesion test, and outgassing and fogging test. The adhesion and the moisture uptake tests were performed on the samples after treatment both in heat environment and in moisture environment. Adhesion of the glob-top materials was tested both for die-to-die and for substrate-to-substrate interfaces.

Experimental results were followed by selection of the glob-top material based on individual measured parameters. The mathematical multi-attribute decision-making methods (SAW and TOPSIS) were then used for evaluating the cumulative effect of the measured parameters on the choice of the epoxy materials for use in different glob-top applications. The following conclusions can be made about the most suitable glob-top material for use in the studied harsh environments.

If an epoxy material should be used, it follows from the above analyses that Epoxy D should be preferred when the cumulative effect of the measured parameters is taken into account. However, as this material is the reference material containing an anhydride acid, Epoxy C would be preferred as the second choice. This choice is valid both for base applications and for optical applications.

Regarding comparison between epoxy materials and the polyurethane it can be concluded that the polyurethane showed good to excellent results in the TMA and in the adhesion tests. The results, however, were worse from the moisture uptake and the outgassing and fogging tests. Therefore, further improvements of the polyurethane material or the process condition must be performed before the polyurethane can be recommended as a substitute for epoxy materials.

Silicone A can be recommended for base applications, although with a remark concerning a risk for outgassing and fogging. The same material cannot be recommended for optical applications. In case of optical applications the only silicone material option would be Silicone B, however, resulting in worse adhesion.

Acknowledgment

The authors of this paper would like to thank the companies participating in the project for a good work and interesting discussions. The participants in the project were the following companies: Kitron Electronics AB, SaabTech AB, Kitron Development AB, Ericsson Microwave Systems AB, Saab Bofors Dynamics AB. A special thanks to Swedish Defense Material Administration, Centre of Technical Expertise Sensors & Telecom and the Swedish Knowledge Foundation (Industrial Research School in Electronics Design, IRSED) for their financial support.

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