EXPERIMENTAL AND THEORETICAL STUDY ON HIGH-TEMPERATURE CONNECTION TECHNIQUES OF THERMOELECTRIC MATERIALS

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ABSTRACT

This paper deals with the structure and the connection techniques of thermoelectric materials for a thermoelectric generator for high-temperature range.
The efficiency of combustion engines can be improved by using the waste-gas with the help of thermoelectric generators. After a short introduction into the fundamentals of thermoelectric the function and structure of a thermoelectric generator will be discussed. The calculation of the thermo-diffusion voltage in dependence of the different thermoelectric materials and their physical properties will be elaborated. With the help of these values the expected performance of a thermoelectric generator can be calculated. After this, the structure of existing thermoelectric modules will be critically analyzed and their weaknesses will be examined. These include materials used, the connection techniques, temperature resistance, cyclabillity and the efficiency of selected thermoelectric modules. In conclusion the innovative structure of a thermoelectric module for high-temperature use will be discussed.

Index Terms – Thermoelectric, high-temperature connection techniques

1. INTRODUCTION

The development of fuel-efficient vehicles and driving concepts are affected by three decisive factors:

1. Regulations of the legislature, requiring manufacturers to pay high punitive duties if pollutant emission limits (e.g. maximum CO_2-value per km) are exceeded.
2. The changing customer demand towards vehicles with lower fuel consumption and a more ecological image.
3. The finite nature of fossil fuels and the dependency on fuel imports from more and more political instable regions [1].

About 1/3 of the energy released from the fuel is in the form of thermal energy in the waste gas and is currently released unused to the environment. With the help of waste heat recovery systems this heat loss can be recuperated. This can be implemented with a thermoelectric generator (Image 1). Systematically speaking, a thermoelectric generator is a kind of heat exchanger with built in thermoelectric modules. These thermoelectric modules generate a thermoelectric voltage if one side is in contact with a heat reserve (hot exhaust) and the other site is in contact with a heat sink (connection to the coolant circuit). If the circuit is closed and a load is connected (on-board power supply) the thermoelectric current flows. Therewith part of the electric power needed for the operation of the vehicle can be generated. This relieves

the alternator and the combustion engine has to provide less power. As a result fuel is saved and the vehicle can be operated more efficiently and ecologically.

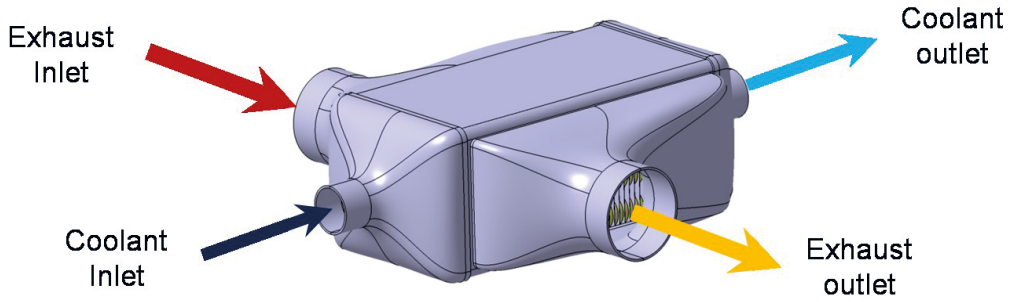


Image 1: Thermoelectric generator, source: MAHLE Behr GmbH & Co. KG

To operate a thermoelectric generator efficiently, the thermoelectric module has to bear the largest possible temperature difference. Depending on the installation location of the thermoelectric generator and the operation point of the engine, up to 900 °C hot exhaust passes through the thermoelectric generator.

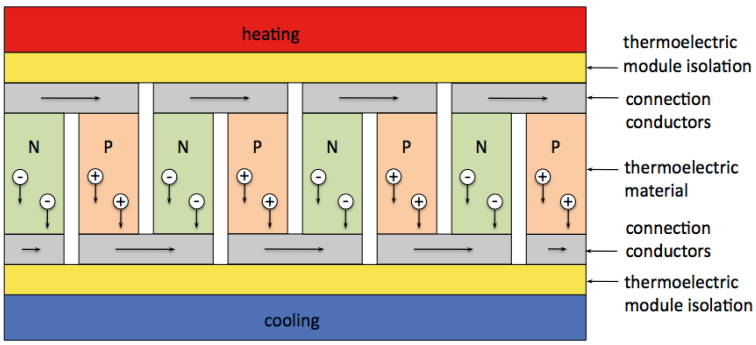


Image 2: Thermoelectric module

Inside a thermoelectric module a large number of n- and p-doped semiconductor materials are connected in a meandering shape with the help of conductor jumpers (Image 2). These are electrically insulating separated from the tubes with exhaust and coolant. The hot side of the thermoelectric module is between 300 °C and 600 °C hot. The temperature on the cold side is approx. 100 °C. This results in great thermoelectric stresses in the thermoelectric module. These have to be absorbed by the connection techniques inside the module and have to withstand a cyclic operation. For a good heat flow through the module to be possible it is important to only use cohesive connection techniques. With the help of these high temperatures the occurring thermo-mechanical stresses can be estimated. This magnitude is used as a boundary condition for the necessary shear strength of the bonded connection technique of materials inside the thermoelectric module.

2. FUNDAMENTALS OF THERMOELECTRIC

If the energy of the charge carriers in a conductor are not spatially constant, charges in higher-energy areas diffuse to areas with lower energy. This occurs when a temperature gradient is present or the material properties change. Thus an electrical field E is established [2], [5]. The relation is shown as follows as a special case of the Ohm's law.

$$E = \frac{j}{\sigma} + \alpha \cdot \frac{dT}{dz} - \frac{1}{e} \cdot \frac{d\mu}{dz}. \tag{1}$$

This equation expresses that the electric field E are determined by means of the current density j and the specific conductivity σ of the conductor. Furthermore the electric field E depends on the Seebeck coefficient α, the temperature gradient along the conductor dT/dz, the amount of the elementary charge e and the variation of the chemical potential along the conductor $d\mu/dz$.

2.1 Thermal diffusion voltage U_{TD}

The thermal diffusion of homogeneous conductors can be derived by equation (1). In an equilibrium state $(j = 0)$ [2] following applies

$$E = \alpha \cdot \frac{dT}{dz} - \frac{1}{e} \cdot \frac{d\mu}{dz} = \left(\alpha - \frac{1}{e} \cdot \frac{d\mu}{dT} \right) \cdot \frac{dT}{dz}. \tag{2}$$

A temperature gradient in a conductor leads to the diffusion of charge carriers from the hot to the cold site of the conductor. Throughout the conductor the electrons moving from the hot to the cold end have higher speed. This results in a directed electron motion. A charge displacement is created and an electric field E is established. The strength of the electric field is defined by two proportions: the Seebeck term and the chemical potential. The Seeback term dominates the system of equation, but is somewhat weakened by the chemical potential. The thermal diffusion voltage $U_{TD}^{w \to c}$ leading from the hot to the cold side can be calculated by using the equation as follows

$$U_{TD}^{w \to c} = \int_A^B \vec{E} \cdot d\vec{z} = -\int_{T_1}^{T_2} \alpha \cdot dT + \frac{1}{e} \cdot \int_{z_c}^{z_w} d\mu. \tag{3}$$

In semiconductors the thermo-diffusion leads to the accumulation of the majority-carriers at the cold end, thus defining the sign of the Seebeck coefficient. The Seebeck coefficient α of an n-semiconductor is less than 0. The Seebeck coefficient α of a p-semiconductor is greater than 0.

In a closed circuit, the amounts lift due to the variation of the chemical potential. As a result, the measured thermoelectric voltage is determined solely by the Seebeck proportion of the thermo diffusion voltage

$$U_{TD} = \int_{T_1}^{T_2} (\alpha_B - \alpha_A) \cdot dT. \tag{4}$$

2.2 Performance of a thermoelectric module

The electric performance P_{el} of a thermoelectric module can be calculated with the maximum degree of efficiency $\eta_{\max}$ approach. The Carnot degree of efficiency and the material specific parameters are substantial [3], [4].

$$P_{el} = \dot{Q}_H \cdot \eta_{max} \tag{5}$$

$$\eta_{max} = \frac{T_H - T_c}{T_H} \cdot \frac{\sqrt{Z\,\overline{T} + 1} - 1}{\sqrt{Z\,\overline{T} + 1} + \frac{T_c}{T_H}} \tag{6}$$

$$Z\,\overline{T} = \frac{\alpha^2 \cdot \sigma}{\lambda}\,\overline{T} \tag{7}$$

$$\overline{T} = T_H - T_c \tag{8}$$

To calculate the electric performance P_{el} the heat flow $\dot{Q}_H$ through the thermoelectric module, both surface temperatures (T_H and T_c) on the hot and cold side, the ZT-value, the Seebeck coefficient α, the electric conductivity σ and the thermal conductivity λ have to be known.

The equation (6) signals through the influence of the Carnot degree of efficiency alone the importance of a high temperature on the hot side of the thermoelectric module. Furthermore the specific thermoelectric materials only work efficiently in a certain temperature range. This is illustrated through the progress of the ZT-values in the image 3.

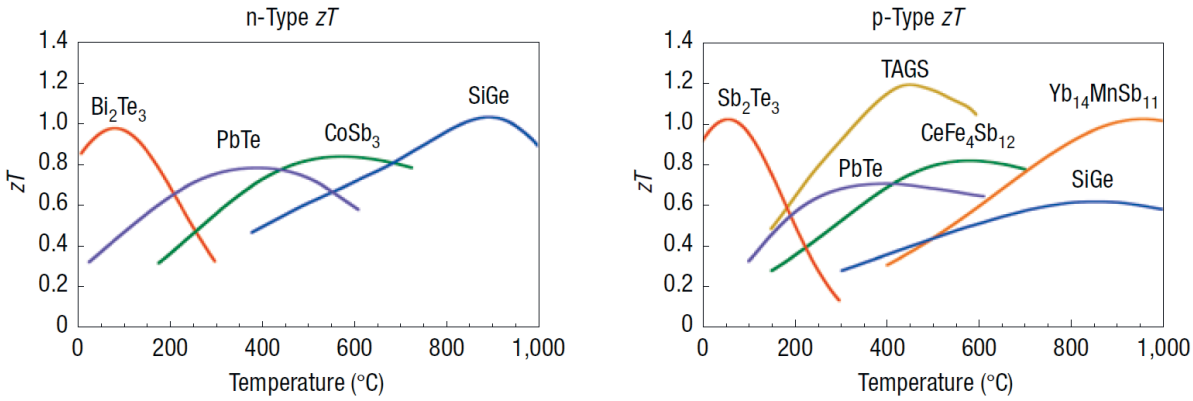


Image 3: Exemplary performance of the ZT value selected for n-and p-doped semiconductor materials [4]

For the intended temperature range for the hot side of the thermoelectric module Snyder and Toberer recommend $CoSb_3$ for the n-doped semiconductor. For the p-doped semiconductor TAGS.

3. EXPERIMENTS AND DISCUSSION

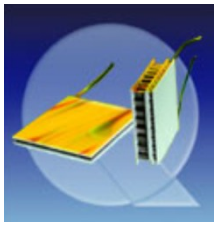
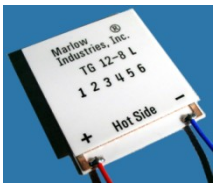
Thermoelectric modules which are used in a heat exchanger in a thermoelectric generator that are developed for the car industry have to meet a variety of boundary conditions:

- The thermoelectric materials must present high ZT-values in the temperature range between 300 °C and 600 °C
- Thermoelectric material must present sufficiently good mechanical strength at this operating temperature

- The modules need to be installed free of pressure
- Only cohesive bonding techniques can be used in the entire module to ensure an ideal heat transport through the thermoelectric module
- The module must be encapsulated to prevent any oxidation damage
- The module must be designed for cyclic operation

3.1 CONSTRUCTION VATIANTS OF THERMOELECTRIC MODULES AND THEIR LIMITATION OF OPERATION

Table 1 compares the technical details of selected thermoelectric modules such as operational temperature, used thermoelectric material, size of module, different concepts in the cover plates, possible encapsulation or cycling.

Manufacturer	Quick-Ohm Küpper & Co. GmbH	Marlow Industries, Inc.	Quick-Ohm Küpper & Co. GmbH	GMZ Energy	
Photo					Target
Type	QCG-18-5.0-1.3	TG12-8	High-temperature thermo generator	TG8-1.0	
T_{hot} [°C]	175	230	570	600	300 - 600
T_{cold} [°C]	50	50	20	100	100
dT [°C]	125	180	550	500	200 - 500
Performance [W]	6,5	7,95	76	7,2	
Size of module (without electrical connections) [mm x mm]	40 x 40	40 x 40	171 x 92	40 x 40	
Performance per area [W / cm²]	0,41	0,49	0,48	0,45	> 1
Thermoelectric material	Bi_2Te_3*	Bi_2Te_3*	Bi2Te3 / PdTe	Half Heusler	e.g. Half Heusler, Silicide, Skutterudite
Cover plate material	Ceramics	Ceramics	Steel	Steel	Metal
Encapsulation (Oxidation protection)	No	No	Yes	Yes	Yes
Cyclability	Yes	Yes	No	Yes	Yes
Pressure-free application possible	Yes	Yes	No	No	Yes
source	[6]	[7]	[8]	[9]	

* The datasheet of the manufacturer does not describe the used thermoelectric material. Due to the range of use and performance, however, it can be assumed that Bi_2Te_3 was used.

Table 1: Comparison of the technical details of selected thermoelectric modules such as operational temperature, used thermoelectric material, size of module, different concepts in the cover plates, possible encapsulation or cycling.

When comparing these different thermoelectric modules, it is found that they can be divided into two different classes. The first two modules in the table (manufacturers Quick-Ohm and Marlow) are laid out for low temperature ranges. They often have a ceramic cover plate. The conductor jumpers are made of copper and connected to the cover plates with the direct copper bonding process. Bi_2Te_3 is the thermoelectric material used. This material is ideal for operation temperature between 100 °C and 200 °C (see image 3). Soft solder is used as connection technology between conductor jumpers and the thermoelectric cubes. This solder

must have a melting point above the operational temperature and below the decomposition point of the thermoelectric material. The modules are often open laterally or sealed with a silicone-seam. This seam, however, is just a protection against contamination or moisture. It cannot serve as an oxidation barrier. This kind of thermoelectric module is being built annually by the millions. They cannot, however, be used in temperatures above 300 °C. The used thermoelectric material Bi_2Te_3 decomposes at these temperatures. The copper conduction jumpers would oxidize, since the casing does not offer an oxygen barrier. Furthermore the total efficiency of these modules is very low. This is due to the influence of the Carnot efficiency degree as shown in equation (6).

The third and fourth module in table 1 (manufacturers Quick-Ohm and GMZ Energy) represent modules laid out for high-temperature ranges. They have a closed metallic casing, which is probably filled with inert gas or evacuated. This information is not available. As a result it prevents a possible oxidation and destruction of the conduction jumpers and the thermoelectric materials at temperatures above 300 °C. The GMZ Energy module uses Half Heusler and the Quick-Ohm module uses PdTe as a thermoelectric material on the hot side. Thus the modules can be used in temperatures up to 570 °C or 600 °C respectively. The performance per area of the modules is, however, very similar in all four modules. (In addition it must be mentioned here that the GMZ Energy module has a performance per are approximately twice as high as the other modules, if for this comparison not the large side of the module is taken into consideration, but the smaller side with an edge length of 27 mm x 27 mm). This is at first sight surprising, since a significant increase in performance can be expected due to the better Carnot efficiency degree alone. Both modules for high temperature range need a minimum contact pressure during application. The module from Quick-Ohm needs 30 bar and the module from GMZ Energy needs 6 bar. This allows conclusion on the structure of the thermoelectric module to be drawn. There was probably no cohesive connection techniques used inside the module, but only press fits, thus significantly degrading the thermal conductivity through the module. This may contribute to the relatively low performance per area. In conclusion it can be said that no thermoelectric module which can be used in a waste heat recovery system for the car industry currently exists.

3.2 ASSEMBLY POSSIBILITIES OF FIRMLY BONDED CONNECTIONS MODULES

Two different variants for the implementation of the firmly bonded connection modules are pursued: through the use of full-surface ceramic substrates or metal substrates with separate ceramic areas.

Ceramic-based substrates

There is a variety of ways for ceramics to be metalized. If it is important for the application that the metallization has a good electrical conductivity, bulk materials should be used. If a more massive strip of copper is connected to the ceramic, there are two common methods: the direct copper bonding and the active metal bonding. The direct copper bonding is specially recommended for oxide ceramics, but it is less complex and expensive. The weld seam, however, has a higher strength. Furthermore there are different ceramics which can vary considerably in their properties such as thermal conductivity, the expansion coefficient and strength. The differences are summarized in table 2:

Ceramic	Al_2O_3	AlN	Si_3N_4
Expansion coefficient CTE [10^{-6} $^1/_K$]	7,0 – 8,0	4,5 – 5,0	2,5 – 3,5
Thermal conductivity [$^W/_{mK}$]	24 - 30	100 - 180	15 - 45

Table 2: properties of technical ceramics [10], [11]

Aluminum oxide is the most cost-effective of the three ceramics, since oxide ceramics can be produced more easily. Aluminum nitride has a much higher thermal conductivity. Silicon nitride has a higher strength, which is of importance for the longevity of the module in cyclical operation.

To compare the strength and the quality of the different ceramics and connection techniques a test layout was compiled. Each ceramic plate was metalized on both sides. Thus, it corresponds to a three-ply structure in order to prevent a curvature of the substrate during cycling. Table 3 contains images of segments of the test layout.

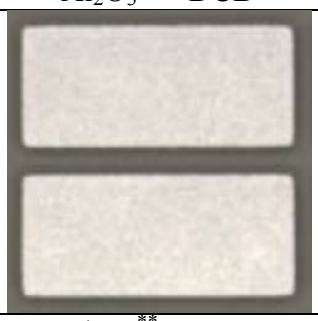
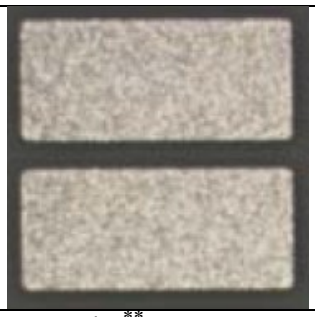
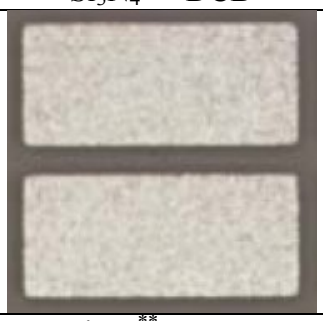
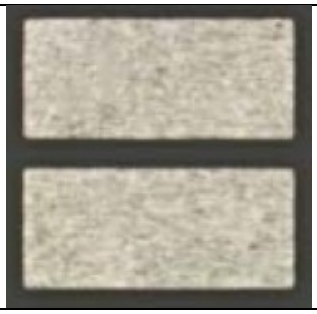
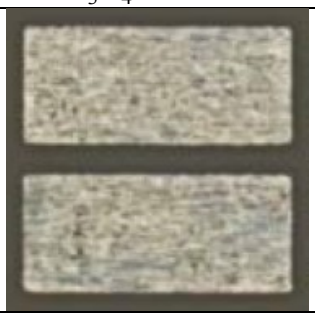
Al_2O_3** – DCB	AlN** - DCB	Si_3N_4** - DCB
		
Al_2O_3** - AMB	AlN** - AMB	Si_3N_4** - AMB
		

Table 3: Display of six different structure (three different ceramic, two different connection techniques), **The description is not the chemical composition of the ceramic. It is the main component of the ceramic. The ceramic may have been optimized by further ingredients.

These samples were tested on a defined test cycle, based on intensified framework conditions to simulate a longer use in reality. The cycle has a high heating and cooling phase (see image 4) and was repeated 100 times on each sample.

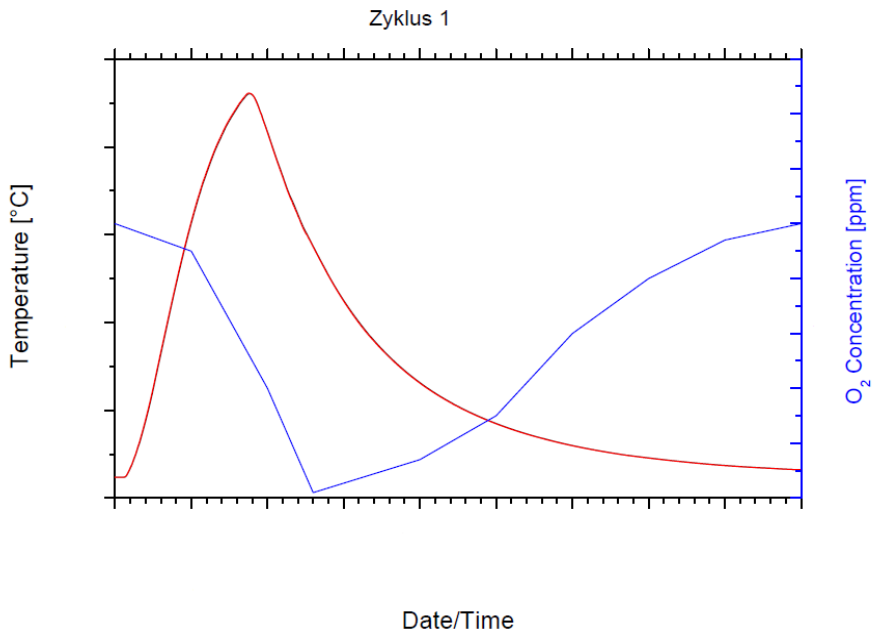


Image 4: Temperature curve over time from the test of the connection techniques in combination with different ceramics and the oxygen level in the atmosphere

The results of the cycle tests are shown in table 4 and 5.

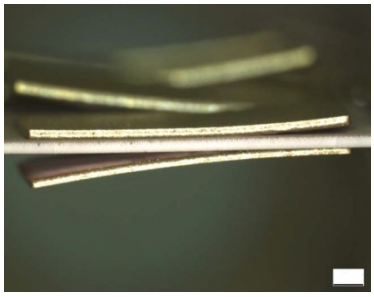
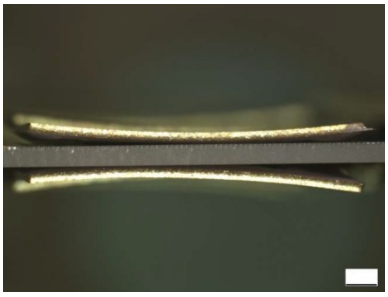
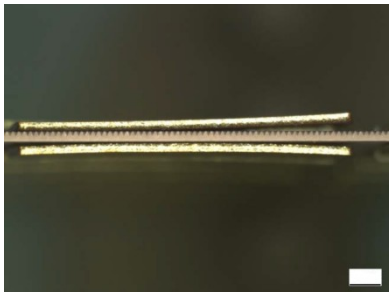
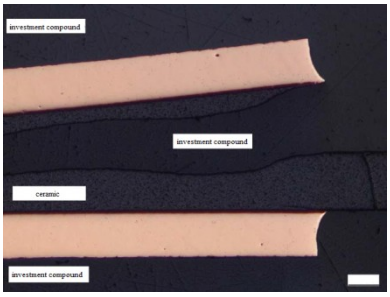
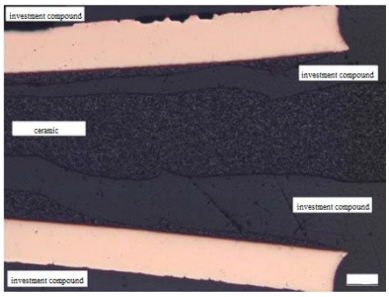
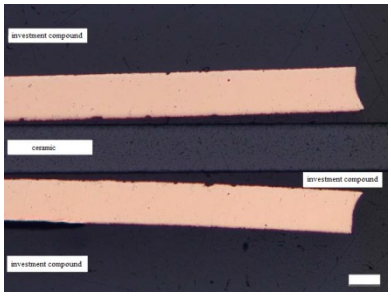
Al_2O_3** – DCB	AlN** - DCB	Si_3N_4** - DCB
		
		

Table 4: The results of the cycle tests (connection techniques: DCB), **The description is not the chemical composition of the ceramic. It is the main component of the ceramic. The ceramic may have been optimized by further ingredients.

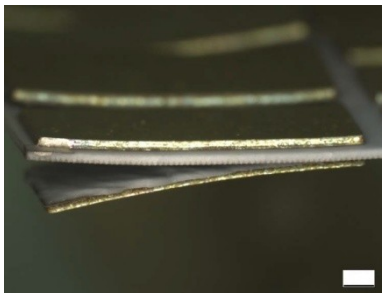
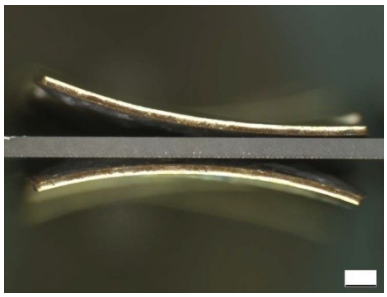
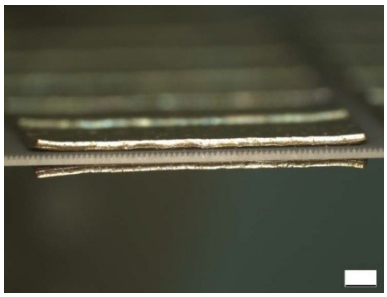
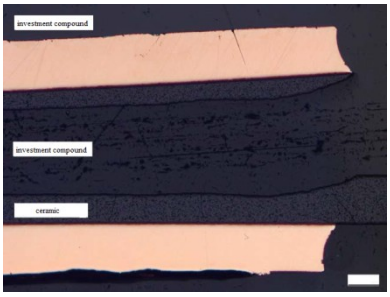
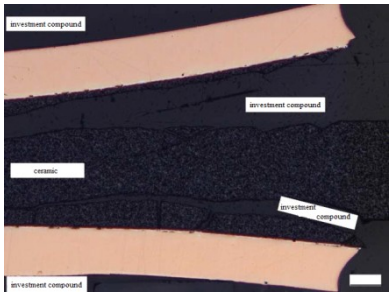
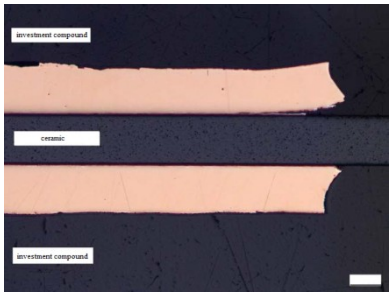
Al_2O_3** - AMB	AlN** - AMB	Si_3N_4** - AMB
		
		

Table 5: The results of the cycle tests (connection techniques: AMB), **The description is not the chemical composition of the ceramic. It is the main component of the ceramic. The ceramic may have been optimized by further ingredients.

At first sight the samples seem to have been destroyed after 100 cycles. It should however not be forgotten, that the samples were cycled under intensified framework conditions. The damage to the connection techniques is due to the different thermal expansion coefficients of the different materials. High thermo-mechanical stresses arise from the different expansions at a rapid warming of the materials. The failure of the component comes at the spot of least resistance. The greatest stress thus occurs in the edge region of the connected parts. When observing the crack formation in dependence of the number of cycles of the six substrate-samples, it is evident that the cracks with all samples always start occurring at the edge region and then run to the center. At first the edges of the conductor jumpers rise slightly, this is clearly visible in the image of the Si_3N_4** - AMB sample. In a real module there is a thermoelectric cube here, which is firmly bonded to the conductor jumpers. As a consequence the edges of the conductor jumpers are pressed on the subtract reducing substantially the shearing effect.

Substrates with an Al_2O_3 - ceramic

While comparing the Al_2O_3 - substrate produced with the DCB process with the Al_2O_3 - substrate produced with the AMB process many similarities are discernible. The weakest spot is the ceramic. A crack always spreads throughout the ceramic. This type of failure is called clam-shell marked fracture. The connection between the conductor jumpers and the ceramic is stronger than the strength of the ceramic. The AMB connection is slightly better than the DCB connection. This difference, however, can be disregarded for Al_2O_3 - substrates. Al_2O_3 based substrates cannot be used on the hot side of the modules. If Al_2O_3 based substrates should be used on the cold side, a DCB joined conduction jumpers suffice.

Substrates with an AlN - ceramic
While comparing the AlN-substrate produced with the DCB process with the AlN-substrate produced with the AMB process many similarities are also discernible. The weakest spot of the AlN-substrates is, as already shown with the Al_2O_3-substrates, the ceramic. This is especially noticeable on the AMB version. Cracks form on both versions underneath the conductor jumpers, with a residual ridge of AlN remaining. Due to the lower strength of AlN the ceramic on these substrates are approx. twice as thick as the ceramic on the other substrate versions. This facilitates the forming of two parallel cracks within the ceramic. Significantly more AlN residue adhered to the conduction jumpers in the AMB version than in the DCB version. This is again due to the greater adhesive strength of the soldered variant. The conduction jumpers on the AlN-substrates start to loosen with considerably fewer cycles than on the Al_2O_3-substrates. Furthermore the curvature of the conduction jumpers is also much more pronounced. On these grounds the use of AlN-based substrates for the production of thermoelectric modules is not recommended, even though they have a better thermal conductivity.

Substrates with a Si_3N_4 - ceramic
While comparing the Si_3N_4-substrate produced with the DCB process with the Si_3N_4-substrate produced with the AMB process there are many more differences in comparison to the aluminum based subtracts discernible. The Si_3N_4-ceramic shows a very high performance. There are no crack formations in both AMB and DCB versions. The ceramic has the highest strength in this combination of materials. The weakest spot on the DCB version is the connection technique. The conduction jumpers start to loosen at the edges after only 20 cycles. The loosened area increases with each cycle. Neither the ceramic nor the conduction jumper is damaged. The AMB version looks much more promising. The bonding of some of the conduction jumper partially starts to marginally loosen from the edges of the ceramic only after 100 cycles. If the cracks are examined closely, it can be ascertained that the soldered seam does not loosen from the ceramic. The cracks form along the interdiffusion zone of the conduction jumper. Therefore the weakest point is the solder joint of the conduction jumpers. This is a very good result. Due to the excellent connection of the conduction jumper through active metal brazing with the high strength of the Si_3N_4-ceramic the AMB version of Si_3N_4-based substrates is recommended for the structure of thermoelectric modules on both the cold and the hot side. A DCB version of the substrates in combination with the high quality Si_3N_4-ceramic is not recommended.

Metal-based Substrates
It is more difficult to integrate a thermoelectric module with ceramic cover plates into a metallic heat exchanger. If such a module has an edge length of several centimeters, thermo-mechanical stresses occur at the seam of the heat exchanger, which are much greater than the ones in the, by comparison much smaller, conduction jumpers. One approach to avoid this are metal-based substrates with isolated brazed ceramic strips. These, in turn, must have a one- or two-sided metallization. If the metal substrate is applied 3-dimensionally, the same can close the module. This, for example, has been applied to the GMZ Energy module in the form of a tub. If the inside of the module is filled up with inert gas or evacuated, the oxidation protection is also given. From the tests with the ceramic-based substrates it is known that the Si3N4-ceramic with soldered conductor jumpers is the most suited for high temperature range cyclic operation. This combination is used for the metal-based substrates. The substrates are constructed in two different variants. In the first variant, the ceramic strips are metalized on both sides and are brazed by means of hard solder to the sheet steel. The second variant has

ceramic strips metalized on only one side brazed by means of an active brazing solder to the sheet steel.

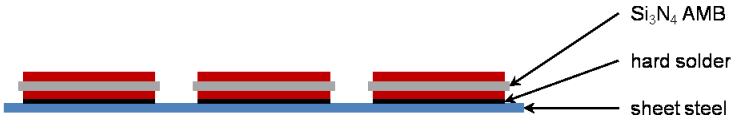


Image 5: Sheet of steel with single brazed two-sided metalized silicon nitride strips by means of hard brazing

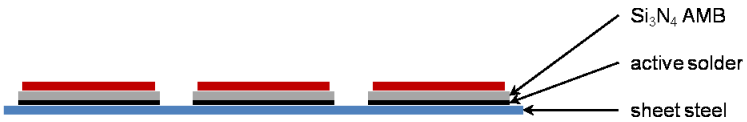


Image 6: Sheet of steel with single brazed one-sided metalized silicon nitride strip by means of active metal brazing

It is difficult to ensure the evenness of the substrates after the soldering process. When the solder hardens, the steel sheet and the metalized ceramic strip are firmly bonded to each other. During the cooling process the individual layers pull together to varying degrees due to the different expansion coefficients. Since the metalized ceramic strips are soldered to the steel sheet on only one side, the substrate behaves like a bimetal and curves. Furthermore, the soldering temperature and time have to be chosen in order to ensure that the contact surface is wetted sufficiently good but the diffusion zone remains in the desired thickness.

Through a variety of test series a brazing curve for the hard solder and active solder could be developed in order to achieve optimum wetting. Additionally the wetting behavior was influenced by various metallization of the conductor jumpers. An adjusted placement of weights during the soldering process improved the evenness of the substrates. By adjusting the solder composition and the amount of solder a good solder seam could be developed. A cross section of such a structure is shown in table 6.

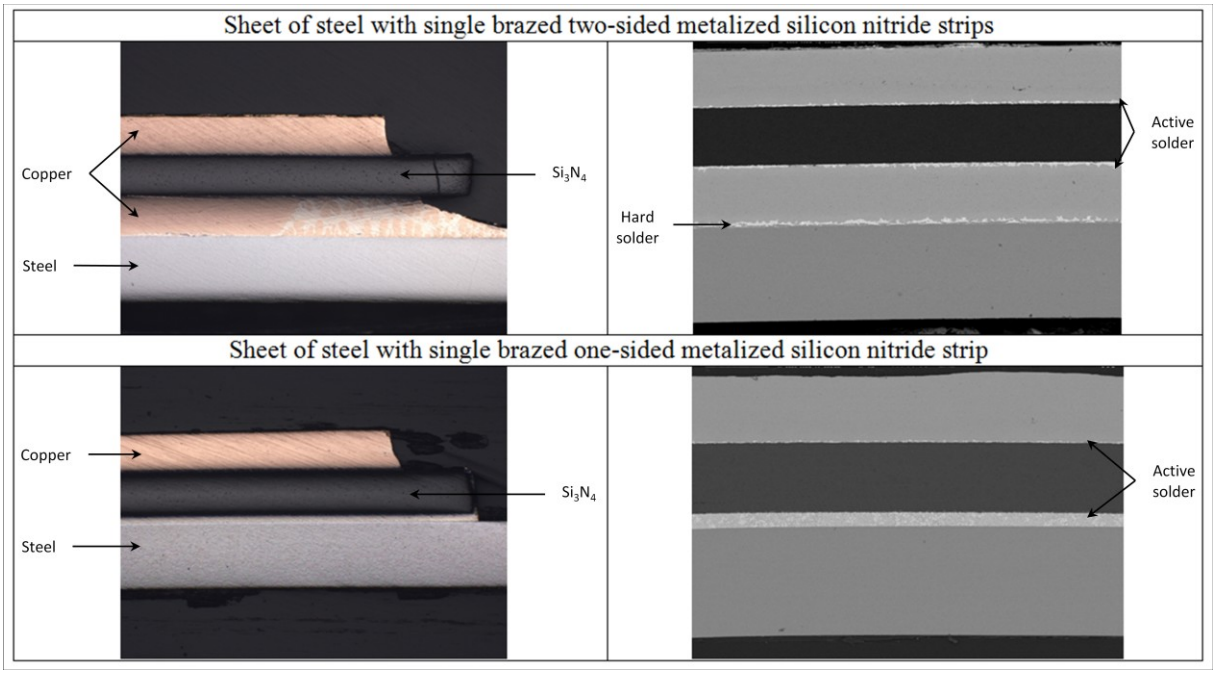


Table 6: Cross section of a steel-based substrate with soldered scattered metalized ceramic strip

The first two images in table 6 show a steel-based substrate produced by hard brazing. The other two show a steel-based substrate produced by active metal brazing. On both cross sections on the left, the structure of each substrate is clearly visible. The images on the right are from the analysis from the scanning electron microscope. These clearly show how the solder effectively wets the steel plate. Furthermore, all solder layers are non-porous. The diffusion zones are equally pronounced. In the peripheral area of the hard sold (first image) there was a slight accumulation of solder. In the subsequent tests less solder will be used here. The evenness of the substrates is also satisfactory. Hereinafter, the thermoelectric material is firmly bonded on these substrates to obtain a complete thermoelectric module for high temperature range.

4. SUMMARY AND OUTLOOK

The aim of this study was to increase the efficiency of thermal combustion engines for the car industry through thermoelectric generators. It has been shown that thermoelectric generators are a type heat exchanger fitted with thermoelectric modules. Hereafter a short introduction into thermoelectric displayed the structure of a thermoelectric module and how it works physically. Next it was explained that a thermoelectric module can only be laid out for a specific temperature range. This primarily depends on the used thermoelectric materials. The complete module has to be subsequently adjusted to this operational temperature range. In addition the structure and function of four current thermoelectric modules and their weaknesses were discussed. Thereafter various ceramic-based substrates (cover plate including conduction jumpers) were assembled and subjected to an application-related test. Based on the results a second generation of steel based substrates was developed an analyzed. With this substrates the high demands from the car industry (possible encapsulation of the module in order to prevent oxidation, firmly bonding techniques to optimize the electrical performance of the module, pressure free installation, cyclability of the module) can be met. The first analyses of the steel-based substrates show promising results. On the next step theses steel-based substrates will be subjected to the same tests as the ceramic-based substrates. Furthermore functioning thermoelectric modules will be assembled, thermally cycled and their electric performance depending on the temperature measured.

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