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RESEARCH ARTICLE

A Novel Method for Monitoring the Skinning Process of Sealing Silicones

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Abstract

Tight and faultless sealing is very important, when joining different internal-combustion engine parts (cylinder head – cylinder block – crankcase – oil sump). For this purpose, silicones are widely used. However, the curing process of silicones varies in a wide range – according to assembly line experiments. Environmental conditions as well as the properties of the actual silicone material can strongly affect the curing. Consequently, a local qualification possibility of the supplied silicone at the assembly line would be favourable. We evolved a novel testing method, which give us real-time information about the curing process of a silicon sample. According to this method a machine was built and installed at the motor assembly plant of AUDI HUNGARIA MOTOR Kft. at Győr (Hungary) and patent claim was announced for it at DPMA (German Patent Office).

Keywords

sealing silicone · motor assembly · curing time · skinning process

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1 Introduction

Sealing silicones are commonly used in automobile industry (Dawir [2]) A crucial application is to ensure the tight sealing between the joined motor parts (cylinder head – cylinder block – crankcase – oil sump, see Fig. 1.). The strong, faultless adhesion of the silicone to each mounted surface is essential for avoiding oil leak. The curing process of silicones during motor assembly is not controlled directly; factories use the recommendations of the silicon manufacturers. However, the real curing process depends highly on environmental conditions, as temperature and humidity (Lin et al. [4]) It means that the curing time differs fundamentally beside summer and winter conditions. Moreover, sometimes there are evident differences between the silicone materials of the same type from the same supplier (Bokor [1]) These statements agree with the requirements of the production line: for ensuring maximum quality, an objective testing method of the curing time of silicones is required.

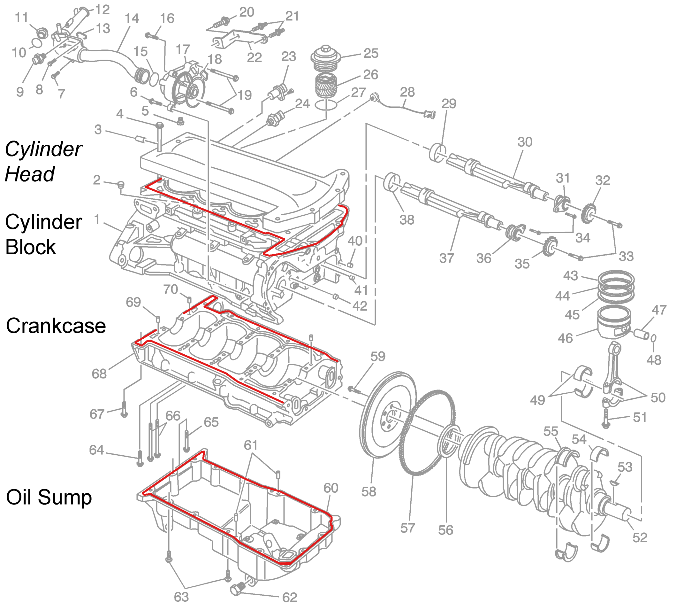


Fig. 1. Main parts of a combustion engine (`http://data.motor-talk.de/data/galleries/`). Silicone sealing is placed between each part.

2 Basic properties of silicones

Silicone is an inorganic polymer, built up from a silicone-oxygen (siloxane) chain with attached methyl- and other reactive groups (marked with X on Fig. 2.). The curing process of one-component silicones – used for sealing in vehicle industry – is called Room Temperature Vulcanization (RTV-1), which is a polycondensation process. During this process the silicone take up H_2O (water), from which the H-atoms bond to two reactive groups which release, while the oxygen atom establish a new binding between two Si-O chains (see Fig.2.) On the whole, the curing is initiated by the humidity of air atmosphere, starts on the surface and move toward the bulk material, and a by-product release. It is usually acetic acid (at acetoxy silicones) or ethyl alcohol (at alkoxy silicones). The by-product can also be primary amine (eg. methylamine) or a ketoxime (Fink [3]).

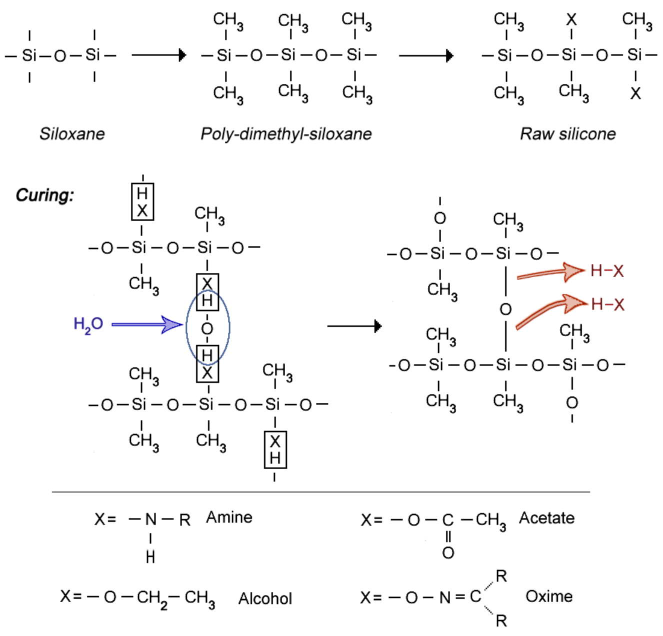


Fig. 2. Silicone chemical construction and curing (on the basis of `http://www.silicones-science.com/chemistry/chemical-reactions-on-the-finished-silicone`)

When applying silicone as sealant, two basic requirements are the good bulk mechanical properties and the appropriate adhesion to the part surface. These properties are influenced by many parameters beside the nature of the base silicone: humidity of the atmosphere, temperature, presence of oil, base surface roughness, time between silicone exposure to air and fitting the two parts etc. These factors influence the curing rate too.

The two main data of curing time are the skin-forming time and the time of full curing. The former is important for assembly: for the best adhesion, the parts have to be fixed before skin forming on the surface of the silicone; while the latter shows the period, after which the assembled parts can be exposed to designed requisition.

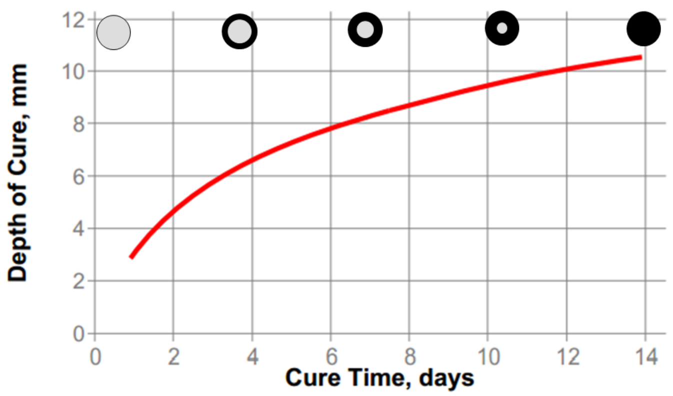


Fig. 3. Depth of curing as a function of time and visualization of cured surface layer (on the basis of `http://www.henkelna.com/`)

3 Objective

The expectation of AUDI HUNGARIA MOTOR Kft. was a testing machine, which give real-time information about the progress of skin formation on the silicone surface. On the one hand, the tests executed beside the same, standard conditions are suitable for qualifying the supplied silicone. On the other hand, the tests at the assembly line – executed at the actual temperature and humidity – give real time information for the maximum time, in which the parts have to be fitted after silicone sealing has been exposed to air – in order to ensure maximum adhesion (before skin formation).

Skin formation process

Skin formation on silicone surface does not take place in a definite time, as it is a continuous process: a thin cured layer starts being formed immediately after exposition to air; thereafter this skin layer thickens continuously. Thus, the time – after which the adhesion should be considered as having deteriorated significantly – could not be determined exactly, we can only make a convention. Practically, we have to choose a property of the silicone which changes during the curing process and which can be measured easily, and define a threshold value. After reaching this value we can say, that a significant skin layer has been formed on the surface, and the adhesion will not be suitable.

Possibilities of measuring skin formation

There is a wide variety of measuring methods, which are suitable for reaching our goal. We can separate them into two groups: measurements with and without physical contact. For example: optical or chemical measurements can be executed without contact, while penetration or sticking force measurements necessitate physical contact. There are even other methods for measuring surface stiffness, like blowing the surface and investigate its deformation.

For the final choice we considered several aspects, like:

- explicit response to skin formation
- real-time result

- continuous or discrete measurement
- the simplest possible setup
- good repeatability
- suitability for different silicones
- ability for measuring production-like samples
- low sensibility to ambient disturbances
- the least possible impact to curing process
- exact and accurate interpretability
- no expensive consumable parts

Sticking force measurement

Taking into account the abovementioned considerations, we have chosen an indirect sticking force measurement method. The basic idea was: push an examination stick into the surface of the silicone, and let it torn out from the surface by its own weight. According to theoretical considerations we expected four typical phases, which was confirmed by subsequent examinations (see Fig. 4.):

1 Soft tacking: In the first period of curing the silicone adheres to the examination stick, but the soft material could not hold the weight of the stick for long.

2 Soft skin: After the formation of a thin skin layer the surface of the silicone is still adhesive, but the cured film becomes strong enough to hold the stick for longer. Finally the stick tears out a small part from the skin layer.

3 Tough skin: This period starts, when the examination stick is not able to tear up the skin layer. The sticking time decreases significantly.

4 No tacking: After a while the surface becomes tack-free, the stick will stick to the surface for no time, practically.

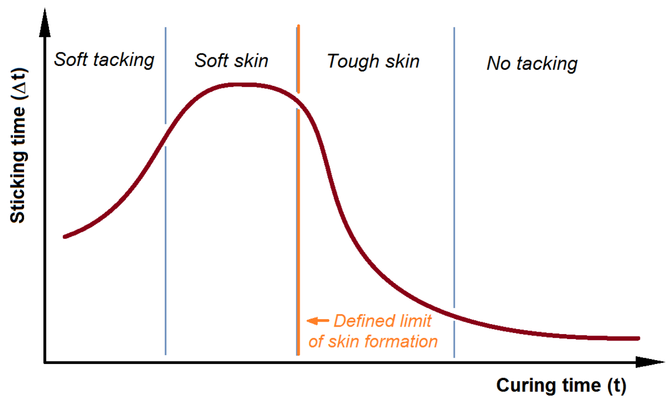
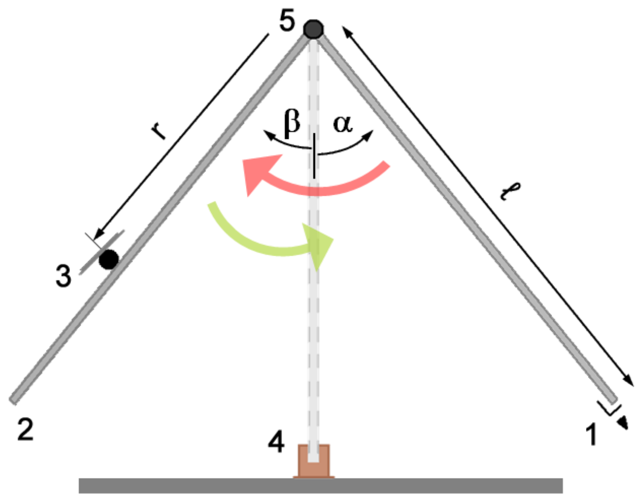
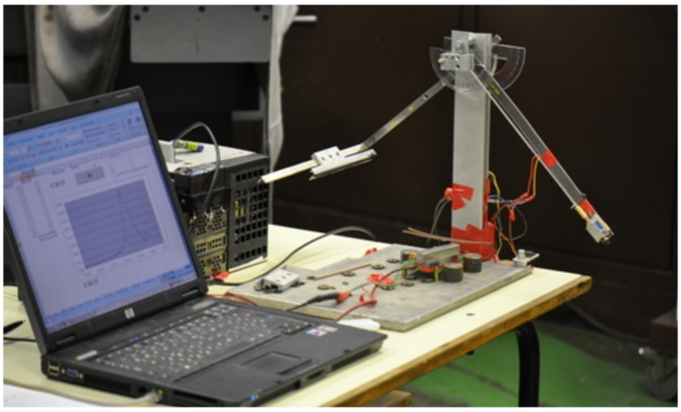


Fig. 4. Sticking time as a function of curing time

In agreement with our partners we decided to define the “skinned state” at the verge of the second and third phase: appropriate adhesion can be expected as long as the examination stick is able to tear up the surface skin layer.



(a)



(b)

Fig. 5. Theoretical model (a) and first built model capable of calibration measurements (b)

Preliminary and calibration experiments

When we started to design the test machine, two main parameters were declared: the basic method of measurement, and the form of the silicone sample: a cylindrical silicone stripe with a diameter of around 3 mm on a flat metallic plate – according to manufacturing conditions. Additionally, the measurement method determines discrete sampling. On the other hand, we have to choose the shape, size and material of the examination stick, its geometrical position relative to the silicone sample, the technique and force to push the stick into the silicone and the method for measuring sticking time. Finally, a pendulum-like setup was chosen, shown by Fig. 5. The basic parts are the starting position (1) defined by the starting angle (α) and stick length (ℓ), the arriving position (2) defined by arriving angle (β), the sample position (3) defined by the distance from the axis if rotation (r), the time measuring unit (4) determines the interval between the first and second passing of the stick, and the rotation point (5) which ensures the lowest possible frictional loss. According to this model, we created a calibration apparatus, by which sticking time was measurable beside different parameters (α, β, r) during the curing of the sample silicone. The difference between α and β results the energy of impact, angle β determines the pulling force after impact while r influences the impact and pulling torque.

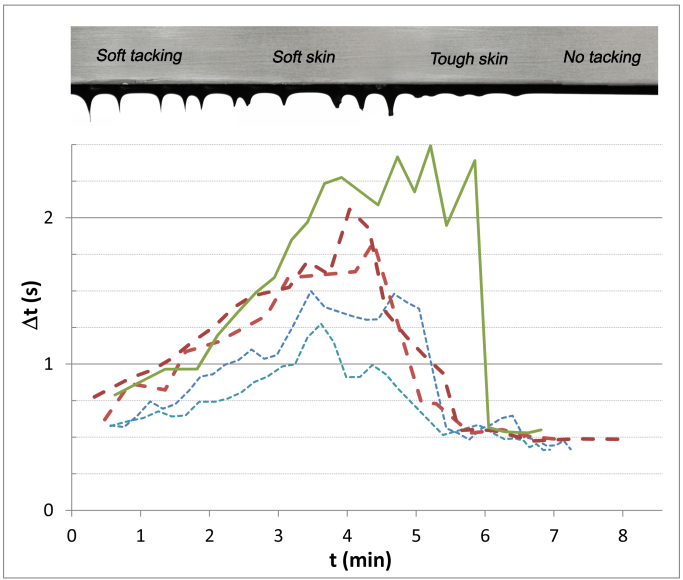


Fig. 6. Sticking time as a function of curing time beside different settings, and the surface of silicone after measurement

After several calibration measurements beside different settings, the optimal geometry was specified, and we continued the tests with different silicones. It was found, that the measured graphs shows exactly the skin formation time – the transition between soft skin and tough skin state – for all the tested silicones.

4 Apparatus setup

Using the knowledge reached from laboratory experiments we designed and built a unique machine for determining skin formation time. The scheme of patented apparatus is shown in Fig. 7.

The main parts of the apparatus are the control unit (under) and the measuring place (over). The control unit contains the power supply, a measurement control PLC, and other control and measuring electronics. It ensures an USB connection to control PC, where the measuring software runs. The measuring place consist of the measuring sticks (15 pieces) placed into a special stick holder, the stick starter stage, the optocoupler stage for sticking time measurement and the sample holder, which is a removable door facilitating the silicone onlay. Supplementary components are: main power switch, operation LED, leveling, measurement place illuminator, adjustable feet, operation monitoring microswitches, humidity and temperature sensors.

Measuring program

To execute a measurement, you have to connect the control PC via USB cable and start the measurement program. It connects automatically to the control PLC, get the temperature and humidity information and log them beside other information: operator, date and time. Then you have to choose a preset measurement or give the measurement details manually. In the meantime, the test silicone strip should be laid onto the sample holder. When you finished it, press RUN immediately, and insert the sample holder door to its place. If both doors are closed, the program let the sticks swing into the sample one-by-one according to the measurement and ambient parameters. When the last stick swings back, the measuring program stops, indicates the *sticking time – curing time* diagram and give the skin formation time. It shows estimation too for the skin formation time beside "base conditions" (22°C, 60% RH). After the measurement you have to clean or change the examination sticks and the silicone sample holder.

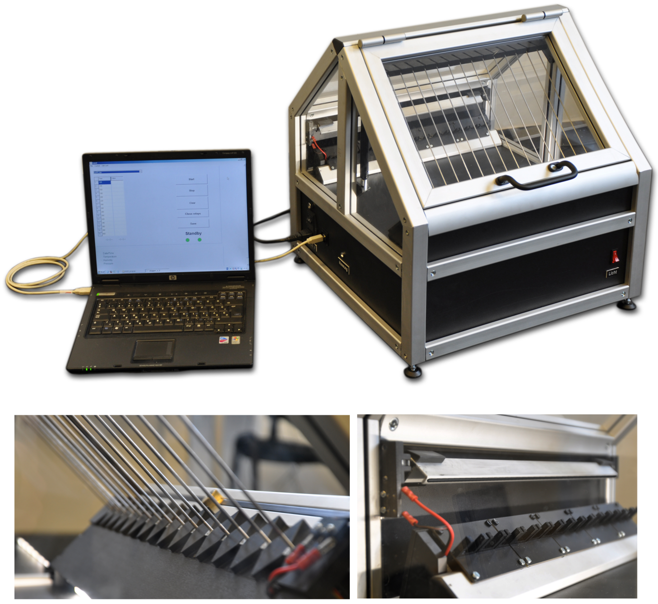


Fig. 8. Skin formation time measurement apparatus with control PC (up), stick starting stage (down left), sample holder and optocoupler stage (down right)

5 Conclusion

Silicone – used for different sealing purposes in vehicles – can ensure appropriate adhesion only if mounting is performed before surface skin is formed. Assembly plants have to rely on the recommendations of silicone manufacturers. However, many parameters influence skin formation time, which were not taken into account at the sealant manufacturers.

AUDI HUNGARIA MOTOR Kft. decided to ensure maximum quality sealings via monitoring the curing properties of silicones used for motor assembly. It ensures the observation of accidental low-quality silicone supplies, as well as assembly line can adapt to the changing properties of silicones due to the varying ambient conditions.

In cooperation with AUDI HUNGARIA MOTOR Kft., our department (the present Department of Automobiles and Vehicle Manufacturing) creates a unique apparatus which fulfils the requirements of assembly line. The patenting procedure is in progress at DPMA (German Patent Office, ref. no. 102012017572.8) for the construction and operation of the testing machine.

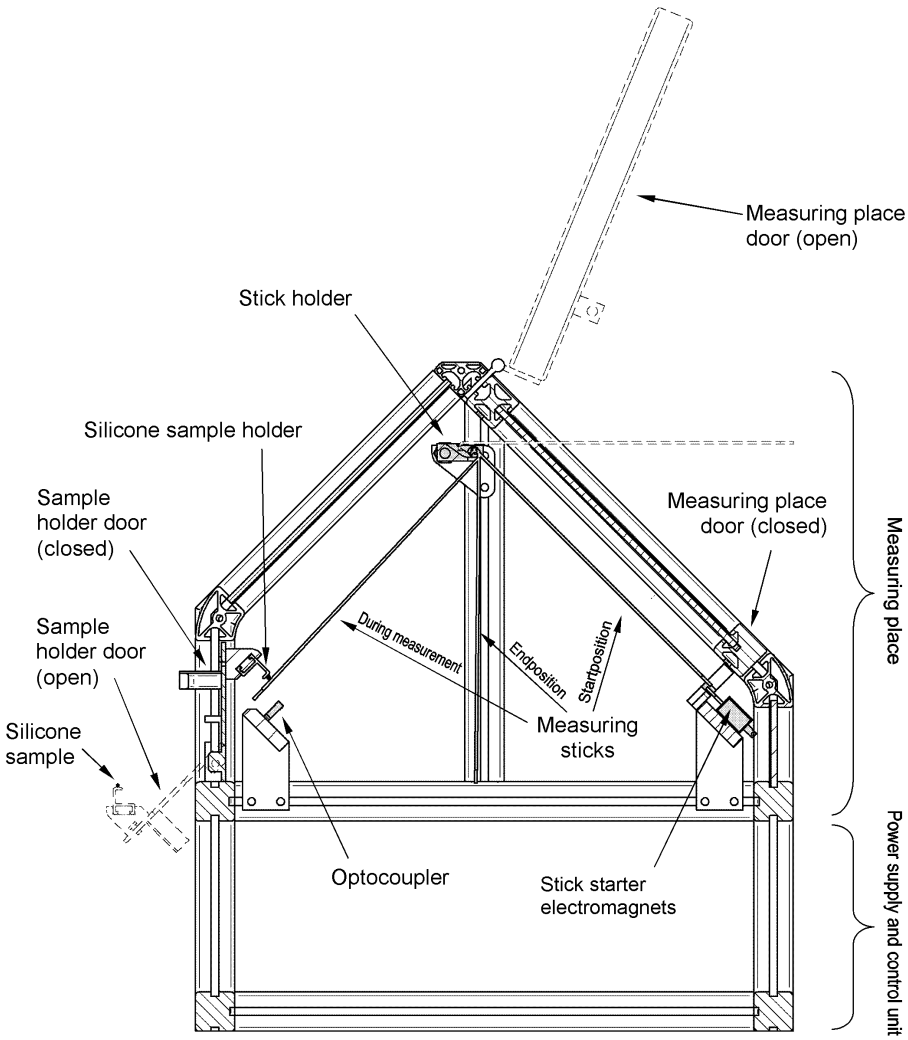


Fig. 7. Outline of the patented silicone skin formation measuring apparatus

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