Calculating the temperature and degree of cross-linking for liquid silicone rubber processing in injection molding

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Funding information
Baden-Württemberg Stiftung, Grant/Award Number: Biofunctional materials surfaces

Abstract
Processing of liquid silicone rubber (LSR) in the injection molding process has a high economic potential. Since there are some fundamental differences compared to classical thermoplastic injection molding, up to now there is a lack of well-founded knowledge of the process which allows an estimation of the cycle time. Because, in addition to reverse temperature control, LSR processing also involves an irreversible temperature- and time-dependent chemical reaction. In this paper, the complex cross-linking reaction is first modelled phenomenologically using dynamic differential scanning calorimetry (DSC) measurements and the well-fitting Kamal-Sourour model. Afterwards, a temperature and cross-linking simulation is set up, which reliably simulates the time- and travel-dependent temperature profile and degree of cross-linking in the mold. Therefore, the released exothermic cross-linking heat is also taken into account. The simulated temperature values are verified with measurements in the cavity during the injection molding process. The measured values correspond very well with the simulated values at different mold temperatures. It is shown that the influence of the cross-linking heat on the overall temperature profile in the LSR component during the injection molding process is relatively low. Nevertheless, the model is necessary to determine the degree of cross-linking - it can be used to calculate the cycle time at which the component of a certain cross-section can be ejected at a known tool temperature and is fully cross-linked. With this knowledge, existing processes can be optimized in terms of mold temperature and curing time, but also new components can be calculated economically.

Keywords
cross-linking, cure kinetics, injection molding, Kamal-Sourour, kinetic modeling, liquid silicone rubber (LSR), processing
1 | INTRODUCTION

The importance of liquid silicone rubber (LSR), developed in the 1970s, has increased continuously in recent years. The reasons for this are its unchanging mechanical and electrical properties over a wide temperature range, good resistance to aging, UV, ozone and chemicals, and biocompatibility. This allows LSR to be used for seals in the automotive and aerospace industries, as cable insulation for the electrical industry, baking dishes and pacifiers for the food industry, and even medical implants and devices. Added to this are the excellent optical properties that allow its use in LEDs and matrix headlights. In addition to the outstanding application properties, there are also advantages in processing - LSR can be precisely processed in the injection molding process in short cycle times, sometimes without postcuring, and yet has a long pot life at room temperature.11

LSR is produced from two components, A and B, in a platinum-catalyzed addition reaction. The base polymer is poly(dimethyl)siloxane, PDMS, shown in Figure 1, which can be adapted to the requirement profile by substitution of various functional groups. One of the two components, usually component A, contains polymer and the catalyst (platinum complex) necessary for the reaction, the other component B contains polymer and the cross-linking agent (H-Siloxan). The crosslinking reaction of LSR can be described by the Chalk-Harrod catalysis cycle. The advantage of the addition reaction compared to the peroxide cross-linking also used is that no low-molecular, volatile components are released. The degree of cross-linking can be determined by differential scanning calorimetry (DSC) measurements. Since the curing normally takes place exothermically due to the high degree of cross-linking, the curing process is extremely accelerated when it touches the hot mold wall and the viscosity increases with the degree of cross-linking. This poses a particular challenge for the fast-curing LSR because the cross-linking reaction is extremely accelerated when it touches the hot mold wall and the viscosity increases with the degree of cross-linking. There is already experience in molding microstructures in thermoplastics, but there is hardly any experience in the field of LSR processing. Investigations into cure kinetics of LSR have already been carried out by Haberstroh, Lopez, Hernandez-Oritz, Hong, Guan among others.

This paper aims to investigate and numerically model the heat transfer and degree of cross-linking after injecting cold LSR compound into the hot cavity. The calculated values shall be verified experimentally with precise temperature measurements in the cavity during the injection-molding process. The results are to be presented in a clear way, which makes it possible to determine the necessary process times based on the given mold temperature and wall thickness. With this knowledge, existing processes can be optimized and new LSR components can be calculated realistically and economically.

2 | MATERIALS AND METHODS

2.1 | Theoretical approach of cross-linking

To simulate the cross-linking behavior of LSR as precisely as possible for different mass- and mold temperatures, it is necessary to model the reaction mechanism. For this purpose, first, the degree of curing in a two-component system has to be considered, then the reaction kinetics based on this curing is calculated and finally, a kinetic model is developed.

The degree of curing can be determined by differential scanning calorimetry (DSC) measurements. Since the curing normally takes place exothermically due to
forming of covalent bonds, the reaction heat released can be detected as an increase in the specific heat flow of the sample mass. The amount of heat released corresponds to the area of the completely exothermic peak of the heat flow curve. If the amount of heat released at time \( t \) is set in relation to the maximum amount of heat released, the reaction conversion \( \alpha \), hereinafter also referred to as the degree of cross-linking, can be determined. \(^{28}\)

To kinetically model the cross-linking of a two-component system, as it is the case with LSR curing, usually the phenomenological approach is used. It describes the curing process utilizing metrologically ascertainable turnover curves. Here, the assumption always applies that the reaction rate is a function of the degree of curing \( \alpha \), which in turn is equated with the reaction conversion.

The cross-linking process is composed as a combination of nth-order reaction and autocatalytic behavior. At nth-order reaction, the reaction rate decreases with increasing degree of cure. The autocatalytic reaction accelerates itself during cross-linking, since reaction intermediates act as catalysts for further reaction steps. \(^{27}\) This means that the system already has an initial reactivity at the beginning, for \( \alpha = 0 \), which accelerates itself during the reaction by autocatalysis. \(^{23,29,30}\) Kamal and Sourour \(^{31,32,33}\) summarized the two models in the empirical Kamal-Sourour model as follows, Equation (1):

\[
\frac{d\alpha}{dt} = (k_1(T) + k_2(T) \cdot \alpha^m) \cdot (1 - \alpha)^n. \tag{1}
\]

where \( m, n = \text{reaction orders} \)

\( k_1(T), k_2(T) = \text{temperature dependent constants} \)

The temperature dependent constants follow the well-known Arrhenius dependence, Equation (2).

\[
k_i(T) = a_i \cdot e^{-\frac{E_i}{R \cdot T}} \tag{2}
\]

where: \( a_i = \text{temperature-independent frequency factor} \)

\( E_i = \text{temperature-independent activation energy} \)

\( R = \text{general gas constant} \)

\( T = \text{temperature} \)

With Equation (2) inserted into Equation (1), the following Equation (3) is obtained, with \( a_1 = b_1 \) and \( a_2 = b_1 \cdot b_2 \), for which corresponding values must be found to fit the experimental data:

\[
\frac{d\alpha}{dt} = b_1 \cdot (1 - \alpha)^n \cdot \left( e^{-\frac{E_1}{R \cdot T}} + b_2 \cdot \alpha^m \cdot e^{-\frac{E_2}{R \cdot T}} \right). \tag{3}
\]

where: \( b_1 = \text{preexponential factor} \)

\( b_2 = \text{preexponential factor for the autocatalytic part of the reaction} \)

Since cross-linking takes place above the maximum glass transition temperature \( T_g \) of about - 130°C to - 120°C, \(^{34}\) the consideration of diffusion processes can be dispensed with in the kinetic modeling of LSR. \(^{35}\)

To calculate the degree of cross-linking and temperature profile within an LSR component, the results have to be combined with a suitable thermal conductivity model. According to Baehr, \(^{36}\) the approach shown in Equation (4) can be used to describe one-dimensional heat conduction problems, taking into account temperature-dependent material values:

\[
c \cdot \rho \cdot \frac{\partial \vartheta}{\partial t} = \frac{1}{r^m} \cdot \frac{\partial}{\partial r} \left( r^m \cdot \lambda \frac{\partial \vartheta}{\partial r} \right) + W(r, t) \cdot \vartheta. \tag{4}
\]

where \( c = \text{specific heat capacity} \)

\( \rho = \text{density} \)

\( \vartheta = \text{temperature} \)

\( \lambda = \text{heat conductivity} \)

\( W = \text{heat source} \)

\( r = \text{specifies position depending on given geometry} \)

\( m = \text{exponent, which describes the described geometry} \)

To calculate the temperature profile of LSR in the mold, the heat transfer is considered in a simplified way via the thickness of the component, that is, a flat layer system since this is where most of the heat is introduced.

Consequently, Equation (4) with \( m = 0 \) for cartesian coordinates and \( r = x \) can be written as follows, Equation (5):

\[
\frac{\partial \vartheta}{\partial t} = \frac{1}{c \cdot \rho} \frac{\partial}{\partial x} \left( \lambda \frac{\partial \vartheta}{\partial x} \right) + \frac{W(x, t) \cdot \vartheta}{c \cdot \rho}. \tag{5}
\]

To take into account the exothermic cross-linking reaction described before (Equation (3)), this can be coupled with the heat source described in Equations (4), Equation (6):

\[
\frac{d\alpha}{dt} = \rho \cdot q_{ges} \frac{d\alpha}{dt}. \tag{6}
\]

If nonconstant material values \( \lambda \) (thermal conductivity), \( \rho \) (density), \( c \) (specific heat capacity) or \( a \) (thermal diffusivity) are used in Equation (5), because they are dependent on temperature, location, time or turnover, a closed solution of the heat conduction equation is usually no longer possible. \(^{20,36}\) One possibility for the numerical solution of the transient, geometrically one-dimensional Equation (5) is the explicit difference method. \(^{37}\) The time variable \( t \) and the local variable \( x \) are discretized and a grid over space and time is formed, with \( t \geq t_0 \), a step size of \( \Delta t \) and the counting index \( k \) as well as \( x \in [x_0, x_n] \), the
step size $\Delta x$ and the counting index $i$. The temperature in each grid point is calculated using the approximate solution of the difference equation by linking each temperature to the temperatures of the neighboring grid points. The discretization error that occurs should approach zero with a reduction of the mesh size $\Delta x$ or $\Delta t$. After the boundary conditions, the starting temperature profile at time $t_0$ and the temperature profile over time of the grid edges $x_0$ and $x_n$, have been determined, the temperature $\theta_i^k$ at the times $t_k > t_0$ at the locations $x_i \in [x_0, x_n]$ can be calculated iteratively according to Equation (7):

$$
\theta_{i}^{k + 1} = \theta_{i}^{k} + \frac{2 \cdot a_i^k \cdot \Delta t}{\Delta x^2} \cdot \left( \frac{1}{1 + \frac{x_i}{\Delta x_{i+1}}} + \frac{1}{1 + \frac{x_i}{\Delta x_{i-1}}} \right) + W_i^k \cdot c \cdot \rho.
$$

For the explicit difference method to be stable, the following convergence criterion must be met, Equation (8):

$$
\frac{a_i^k \cdot \Delta t}{\Delta x^2} \cdot \left( \frac{1}{1 + \frac{x_i}{\Delta x_{i+1}}} + \frac{1}{1 + \frac{x_i}{\Delta x_{i-1}}} \right) < \frac{1}{2}.
$$

With Equation (8), the maximum timestep size $\Delta t$ can be calculated for given substance data and a desired spatial resolution $\Delta x$. For strongly fluctuating material values it is recommended to recalculate the maximum permissible time step value for each time step.\[20,36,37\]

Aiming to be able to determine not only the temperature course but also the turnover course, the kinetically modeled cross-linking of LSR $a_i^k$ must be calculated for each grid point $t_k > t_0$ in the range of $x_i \in [x_1, x_{n-1}]$, Equation (9):

$$
a_{i}^{k + 1} = a_{i}^{k} + b_1 \cdot (1-a_i^k)^n \cdot e^{-\frac{x_i}{\Delta x}} + b_2 \cdot a_{i}^{k} \cdot e^{-\frac{x_i}{\Delta x}} \cdot \Delta t_k.
$$

As start condition it is assumed that the cross-linking reaction has not yet started at the start time $t_0$ and therefore $a_i^0$ is valid for $i \in [0, n]$.

2.2 | Experimental part

2.2.1 | Material

In this study, we used a commercial two component LSR material by Dow (SILASTIC RBL-9200-50 LSR) with a hardness of 50 Shore A. The components A and B were mixed in a 1:1 ratio.

2.2.2 | Characterization of the material properties

Various analysis methods are available for determining the thermal behavior. For a kinetic modeling of the cross-linking reaction of LSR, for example, a thermal analysis method such as DSC measurement or rheological methods with a rheometer can be used. Harkous et al.\[11\] found that thermal analysis methods are better suited for LSR than rheological methods to determine the kinetic behavior of LSR. Furthermore, Hulder\[27\] discovered that dynamic rather than isothermal measurements are particularly suitable for DSC measurements to determine the reaction kinetics.

DSC measurements

The calorimetric measurements of the LSR mass were carried out with the DSC 214 Polyma dynamic differential calorimetry analyzer from NETZSCH-Gerätebau GmbH. The dynamic measurements were performed for sample masses of 10 mg at four different heating rates (1 K min$^{-1}$, 2.5 K min$^{-1}$, 5 K min$^{-1}$ and 10 K min$^{-1}$) in sealed and once pierced aluminum pans. As a reference, an also pierced and sealed empty pan was used. First, heating was carried out at the corresponding heating rate from -70°C to 220°C, then cooled down again to -70°C, this temperature was held for 15 minutes to allow thermal equalization, and then heated up again to 220°C. The second heating curve is therefore recorded to determine whether the sample has cross-linked completely in the first heating. Each measurement was performed at least twice. The material under investigation was stored at -20°C to prevent precross-linking. For the preparation of the LSR samples, 20 minutes were observed in each case until the start of the DSC measurement. During all measurements, the samples were purged with nitrogen.

For the evaluation of the DSC curves, only the two heating curves are used. Figure 2 shows an example of a typical course of the curves. The green curve describes the first heating. At about 90°C the cross-linking begins, which is noticeable in the measured data as an exothermic peak. Because the second, red heat flow curve runs without an exothermic peak, it is clear that the material is completely cross-linked after the first heating.

For lower heating rates, the peak shifts towards lower temperatures, and for faster heating rates it shifts towards higher temperatures, Figure 3. The total amount of heat released was determined by drawing a straight line before and after the peak, connecting the baselines and
integrating the resulting area under the peak, see Figure 2. For all heating rates, the cross-linking area remains largely the same. The average amount of heat released during cross-linking was determined to be $6922 \text{ J/kg}^{-1}$.

2.2.3 | Experimental setup

The LSR was processed on an Arburg 420 C 1000 injection-molding machine, which is specially equipped for processing liquid silicone rubber. By means of a special dosing system, the pumpable, pasty components A and B are transported separately to the static mixer, shortly before the dosing unit of the injection-molding machine. Only immediately before the actual processing, the two components are mixed, dosed and injected into the hot mold via the injection unit, which is temperature-controlled to $25\degree C$. The usual mold temperatures range from $140\degree C$ to $220\degree C$. The material samples for further investigation of the LSR were taken directly from the dosing unit of the injection-molding machine after production. This ensured that the material properties corresponded to the actual production conditions.

2.3 | Setup simulation

To simulate the cross-linking behavior of LSR, simplifying assumptions were made. It was assumed that for injection molding, the injection process of the low-viscosity LSR is so fast that the cross-linking reaction is not started until the mold has been completely filled. The component
investigated, shown in Figure 4 on the left, has a component volume of about 16 ccm. In the injection molding process, an injection speed of 100 ccm s\(^{-1}\) is used, so that the silicone is in the mold in 0.16 second. In addition, the cross-linking process is only considered via the component thickness (5 mm). This simplifies the structure to a system of mold - component - mold, as shown in Figure 4 on the right. The following parameters for the two molds (nozzle and ejector side) and the LSR component, listed in Table 1, were used for simulation. The material data for LSR, such as density, heat capacity and heat transfer coefficient were determined experimentally.

### 2.4 Setup verification with measurements

To verify the simulated data, the component was injected with the simulated parameters and the temperature curve was measured during the entire injection process in the component. For this purpose, disposable thermocouples of type J were used.\(^{38,39}\) The thermocouples are made out of two different wires. For Type J, these are iron (Fe) and copper-nickel (CuNi), clamped into the mold cavity at different measuring heights, see Figure 5 for the mold and Figure 4 on the left for the molded part.\(^{40}\) The measuring point was always in the center of the component. Using a slide mold, developed especially for this purpose, a few shots can first be produced in a “production cavity” without thermocouples to establish thermal equilibrium and real production conditions in the tool. Then, without any major interruption, the slider mold is converted to the “measuring cavity”, which is equipped with the disposable thermocouples at different measuring heights. When the next shot is injected into the mold, the thermocouples record the temperature profile during the entire cycle. After this shot into the measuring cavity, the production process must be interrupted, because the measuring cavity must be equipped with new thermocouples again. Then the procedure starts again.

### 3 RESULT AND DISCUSSION

#### 3.1 Kinetic model

From the data of the dynamic DSC measurements described in Chapter 3.1, the kinetic constants of the
Kamal-Sourour model presented in Chapter 2.1 were calculated. The model parameters are adjusted until they describe the cross-linking process for all measured heating rates as accurately as possible. The left diagram of Figure 6 shows the measured DSC data and the calculated data according to the Kamal-Sourour model for the corresponding heating rates. For the mathematical modeling, the parameters listed in Figure 6 on the right side were used, to which no physical significance can be attributed. The calculated model is highly temperature-dependent and sensitive to small changes in initial conditions.35

3.2 Simulation results

With the appropriate mathematical modeling of the cross-linking reaction, the heat transfer during the injection molding process can be modeled as described in chapter 2.1.4. The MATLAB software package was used for the iterative numerical calculation. Table 2 shows the temperature profile and the degree of cross-linking in the LSR component, plotted over time and position for different mold temperatures. The data used for the simulation are listed in Table 1.

3.3 Verification of simulated results

To check whether the simulated results correspond to the actual temperature curve, the temperature curve in the cavity was measured during the injection molding of a LSR component, described already in chapter 2.4. The disposable thermocouples used for this purpose were positioned at different measurement heights, see Figure 4.

Figure 7 shows the measured and simulated temperature curve at the five different measuring positions in the LSR component. For the simulation, the actually measured surface temperatures of the ejector and nozzle side were used as mold temperature. At the set mold temperature of 160°C with the temperature control unit, just 143°C was measured on the ejector side and 148°C on the nozzle side. The positions of the five measuring points in the component were measured subsequently for each part. For every position in the component, the measured values agree very well with the simulated temperature curves.

3.4 Influence of reaction heat

The investigations have shown that the influence of the released exothermic cross-linking heat of the analyzed LSR has a minor effect on the temperature profile in the LSR component. Thus, the difference between the simulated model with and without cross-linking, for example, in a measurement position of 1.2 mm after 45 seconds is only 1°C, shown in Figure 8. Nevertheless, the simulated data with the heat of cross-linking are more consistent with the actual measured values than without. The results also demonstrate that the data with and without cross-linking show hardly any deviation at the beginning (1 second-30 seconds) and the influence of the heat of networking only becomes noticeable towards the end when the temperature of the LSR rises above 120°C.

Despite the low influence on the temperatures in the mold, the model is needed for the temperature and time-dependent determination of the degree of cross-linking. This allows cycle time estimates to be made for the efficient and economical calculation of LSR components. A prerequisite for this is knowledge of the minimum permissible degree of cross-linking at the thickest component point at which the component can be ejected without hesitation. This is because it is not necessary for the LSR component to already cross-link fully in the mold - the residual heat when the component is ejected

**FIGURE 6** A, Measured DSC data and calculated data according to the Kamal-Sourour Model. B, Parameters used for the mathematical modeling of Kamal-Sourour Model. DSC, differential scanning calorimetry
TABLE 2  Simulated temperature and cross-linking curves for different mold temperatures (140°C, 160°C, 180°C, 200°C, 220°C) and 60 seconds cycle time

| Mold-temperature | Time- and travel-dependent temperature profile: mold - LSR component - mold | Time- and travel-dependent crosslinking-degree in the LSR component |
|------------------|---------------------------------------------------------------------------|------------------------------------------------------------------|
| 140°C            | ![Temperature profile](image1) | ![Crosslinking degree profile](image2) |
| 160°C            | ![Temperature profile](image3) | ![Crosslinking degree profile](image4) |
| 180°C            | ![Temperature profile](image5) | ![Crosslinking degree profile](image6) |
| 200°C            | ![Temperature profile](image7) | ![Crosslinking degree profile](image8) |
TABLE 2 (Continued)

| Mold-temperature | Time- and travel-dependent temperature profile: mold - LSR component - mold | Time- and travel-dependent crosslinking-degree in the LSR component |
|-------------------|--------------------------------------------------------------------------|-----------------------------------------------------------------|
| 220°C             | ![Image](image1.png)                                                     | ![Image](image2.png)                                             |

Abbreviation: LSR, liquid silicone rubber.

FIGURE 7  A-E, Temperature curve at the individual measuring points, measured and simulated in five different height positions (1.2 mm, 1.6 mm, 3.35 mm, 3.8 mm, 4.6 mm). F, LSR component with overmolded thermocouples. LSR, liquid silicone rubber

is sufficient to cross-link it completely until it cools down. Postcross-linking by residual heat is shown in Figure 9. At a mold temperature of 180°C the component was ejected after 32 seconds. At this point, there is only a degree of cross-linking of 6% in the center of the component, whereas the component is already fully cross-linked.
**FIGURE 8** Measured data at certain height position (1.2 mm) in the LSR component compared with simulated data with and without concerning the exothermal heat resulting from cross-linking. A, Temperature curve over 50 seconds. B, Enlarged temperature curve in the area of maximum deviation at the end of the injection molding cycle (30 seconds to 50 seconds). LSR, liquid silicone rubber

**FIGURE 9** Simulation of postcuring a 5 mm thick component at room temperature (25°C) with 180°C mold temperature and a curing time in the mold of 32 seconds. After 100 seconds the component is 100% cross-linked. A, Temperature curve at specific positions in the component (0.05 mm, 0.5 mm, 2.5 mm). B, Distance- and time-dependent temperature course over the component thickness. C, Degree of cross-linking in the center of the component. D, Distance- and time-dependent course of the degree of cross-linking over the component thickness.
in the areas contacting the tool. Due to the residual heat present in the part, the component is nevertheless completely cross-linked after further 68 seconds at air (25°C). Plotted for different mold temperatures (120°C to 220°C) and different wall thicknesses (1 mm to 6 mm), Figure 10 shows the minimum cycle time at a given mold temperature, based on the maximum wall thickness.

4 | CONCLUSION

In this paper, the temperature profile within an injection molded LSR component was simulated. In addition to the usual material parameters, the exothermic cross-linking heat generated during cross-linking was also taken into account. To obtain correct stock data for this purpose, dynamic DSC measurements at various heating rates were carried out, from which a mathematical model could be derived using the Kamal-Sourour model. The gained kinetic model demonstrably reproduces the cross-linking process at different heating rates. Using the explicit difference method, it was then possible to simulate the temperature curve inside an LSR component in the hot mold. The simulated results were verified with temperature measurements in the mold. For this purpose disposable thermocouples were placed in different component heights in a slider tool. After the mold had been brought into thermal equilibrium in a production cavity during several production cycles, it could be switched to the measuring cavity equipped with sensors. Then, in the next production cycle, the temperature curve within the component is recorded. The measured results agree very well with the simulated temperature curves over the entire temperature range. Furthermore, it could be shown that the influence of the released exothermic cross-linking heat has a minor effect on the temperature profile in the component. Nevertheless, the model is needed to describe the cross-linking to be able to make statements about the degree of cross-linking in the component. From the simulation data, a graphic was developed with which the minimum cycle time required for complete cross-linking in the LSR component can be read for given wall thickness and mold temperature. In order to apply the knowledge gained to more complex geometries, the relevant data on heat transfer and exothermic reaction could be integrated into a fluid dynamic simulation software. Such software could even be used to model the injection process. In comparison to mostly very expensive licensed and less flexible plastics simulation software, this is a comparatively cheap and flexible alternative. Furthermore, the correlation between cross-linking and viscosity must be investigated. Coupled with reliable simulation data, the next step is to investigate the edge area directly on the mold wall. If, for example, fine structures such as nanostructures are to be molded, knowledge of the temperature curve can be used to predict the point at which they can no longer be molded, since the gel point has been reached through progressive cross-linking.

ACKNOWLEDGMENT

D. Weißer would like to acknowledge the support of Baden-Württemberg Stiftung gGmbH within the scope of “Biofunctional materials and surfaces” research program.

AUTHOR CONTRIBUTIONS

Dennis Weißer: Conceptualization; data curation; formal analysis; investigation; methodology; project administration; resources; software; validation; visualization; writing-original draft; writing-review and editing. Daniel Walz: Data curation; formal analysis; investigation; writing-review and editing. Johannes Schmid: Data curation; funding acquisition; investigation; writing-review and editing. Dennis Mayer: Data curation; investigation; writing-review and editing. Matthias Deckert: Conceptualization; funding acquisition; methodology; project administration; software; supervision; writing-review and editing.

CONFLICT OF INTERESTS

Neither the author nor the co-authors have any conflicts of interest.

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How to cite this article: Weißer DF, Walz D, Schmid J, Mayer D, Deckert MH. Calculating the temperature and degree of cross-linking for liquid silicone rubber processing in injection molding. Journal of Advanced Manufacturing and Processing. 2021;3:e10072. https://doi.org/10.1002/amp2.10072