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Title: Enhanced thermal control in casting processes using thermally sprayed resistive heating coatings

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Abstract

Thermally sprayed resistive heating coatings based on semiconducting TiO_x offer a promising solution to compensate for uneven cooling and premature solidification in casting processes. A deep understanding of how spray parameters influence coating properties is essential. Statistical analysis of input variables enables the design of tailored heating coatings for both simple and complex geometries, tested in casting processes. Spray parameters fundamentally shape the phase composition of TiO_x and resulting temperature homogeneity. Optimized coatings withstand 20.000 heating cycles at a power density of $p = 40\ W/cm^2$ and achieve a heating rate up to $\dot{T} = 28\ K/s$. The system transfers effectively from simple to free-form geometries and model tools, reducing cycle times to $t = 16\ s$ and reducing part warpage by a factor of four in injection molding. TiO_x enables a novel heating and sensor system with direct melt contact, enhancing precision in casting processes.

Keywords

Thermal compensation, thermal spray, heating coatings, casting, injection molding

1 Introduction

In casting processes, precise thermal control is essential for achieving high part quality. Non-uniform cooling rates can lead to e.g. warpage. Although elevated mould temperatures may be beneficial for part quality, they also increase cooling times and thus reduce throughput, making it economically unfeasible for processes such as injection moulding. As part of the research program SFB 1120, this project investigates the use of thermally sprayed heating coatings to dynamically control mould temperatures and compensate detrimental effects based on inhomogeneous temperature fields.

With thermal spraying (TS), resistive heating elements based on metallic or ceramic conductive layers in between ceramic insolating layers can be applied directly onto tool surfaces (Fig. 1). The use of thermally sprayed heating elements in casting processes was introduced by Deckert for injection moulding in 2012 for a NiCr20 based metallic heating coating [Dec12]. Ceramic based, sub-stoichiometric titanium oxides (TiO_x) on the other hand have gained attention as heating coating materials due to their low thermal expansion coefficient, which matches that of insulating ceramics such as Al_2O_3 [FFK+10]. To improve the stability of the semiconducting system, Cr_2O_3 is commonly added to the TiO_x powder at concentrations of 20 wt. % [TBT+11].

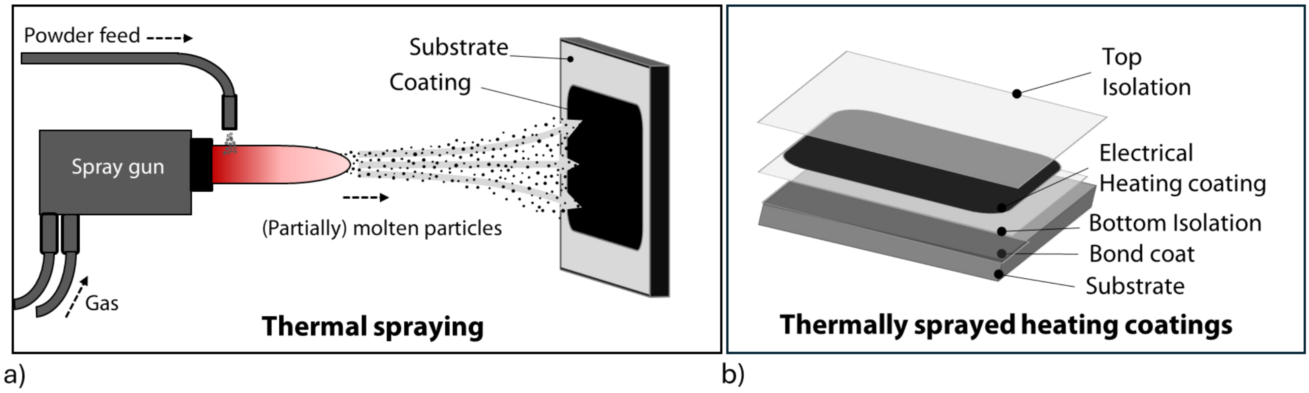

Figure 1: a) Schematics of the thermal spraying process b) explosion drawing of thermally sprayed heating coatings

Owing to its significantly higher electrical resistance compared to metal conductors, which typically need to be structured in meander geometries, $TiO_x/20Cr_2O_3$ allows full-area deposition of the heating coating [BWK+20], thereby easing the fabrication of heating elements. Furthermore, the temperature-dependent resistance of the material exhibits negative temperature coefficient (NTC) behaviour and can be approximated with an Arrhenius-type equation [BWK+20]. This property may also enable the use of the actuator coating in sensing applications.

2 Aim of the Investigation

Aim of this work is the advancement of thermally sprayed ceramic heating coating systems based on $TiO_x/20Cr_2O_3$ in casting systems for advanced thermal control (Fig. 2). During the two phases as part of the SFB 1120, the following research areas are adressed:

- Determining the influence of spray parameters on electrical properties (Phase II, III)
- Increasing the durability of the coatings (Phase II)
- Transferring the multilayer system onto freeform geometries (Phase III)
- Evaluate temperature control based on the electrical resistance of the coatings (Phase III)
- Applying the heating coatings in casting processes (Phase II/III)

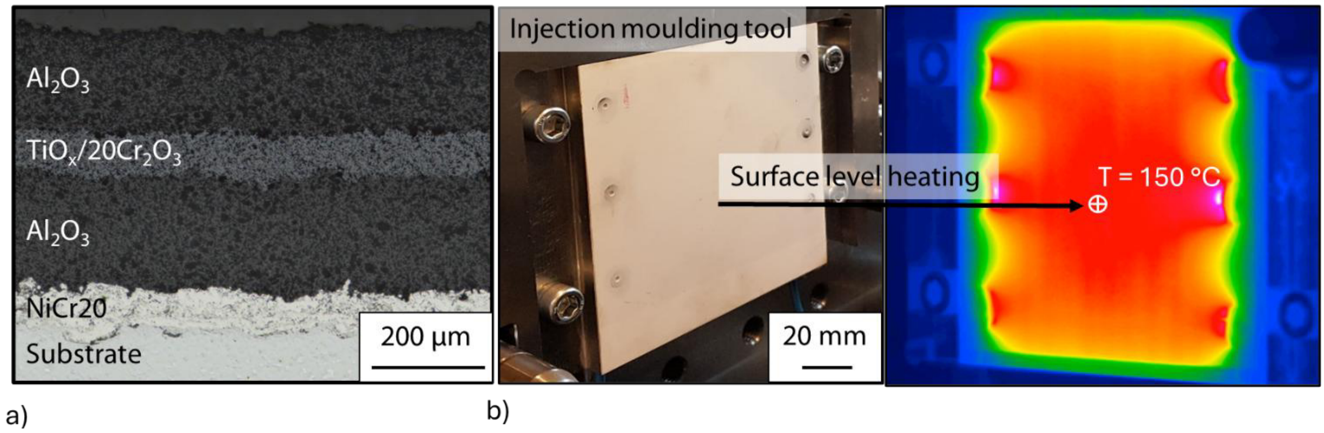

Figure 2: a) cross section of TS-heating coatings based on $TiOx/20Cr_2O_3$ b) Injection moulding tool with heating coatings. Electrical connection is obtained by isolated screws from the backside of the tool geometry.

3 Materials and Experimental Details

Atmospheric plasma spraying (APS) is conducted using a TriplexPro™-210 plasma generator (Oerlikon Metco, Wohlen, Switzerland) for the NiCr20 bond coat, the Al_2O_3 insulation layer and the $TiO_x/20Cr_2O_3$ heating coating. Spray powders are obtained from commercially available suppliers: NiCr20 (GTV Verschleißschutz GmbH, Luckenbach, Germany), $TiO_x/20Cr_2O_3$ (Ceram GmbH, Albbruck-Birndorf, Germany) and Al_2O_3 (Oerlikon Metco). Coatings are applied using a KR16 robot handling system (KUKA, Augsburg, Germany), with the spray gun moved over the surface in a meander pattern. The substrate materials, unalloyed carbon tool- (C45U) and structural steels (S235JR), are cleaned and sandblasted before coating. The spray parameters are listed in Table 1.

Table 1: Parameter set for the heating coating systems

Parameter	APS		
Function	Bondcoat	Insulation	Heating coating
Material	NiCr20	Al_2O_3	TiO_x/Cr_2O_3
Particle size distribution / µm	-53 +25	-45 +22	-25 +5
Injector diameter / mm	2	2	2
Primary gas Ar / SLPM	70	60	60
Secondary gas H_2 / SLPM	0	6	6
Current / A	400	450	450
Stand-off distance / mm	120	120	120
Meander width / mm	5	5	5
Robot velocity / $mm \cdot s^{-1}$	800	1,000	1,000
Carrier gas Ar / SLPM	4.5	5.5	7.5
Feeding rate / $g \cdot min^{-1}$	31	21	24

Heating experiments are conducted using a custom-built electrical test bench (RST Rostock System Technik GmbH, Rostok, Germany) equipped with a LabView-based PID-controller to regulate the power supply. The temperature levels are selected according to the intended application of the coatings. Electrical resistance is measured using a four-point probe method with a TM-508A device (Isothermal Technology, Southport, United Kingdom). Sample cross-sections are polished and examined by scanning electron microscopy (Phenom XL, Thermo Fisher Scientific Inc., MA USA) and optical light microscopy (Carl Zeiss AG, Oberkochen, Germany). Phase composition was determined by X-ray diffraction (XRD) using a XRD 3000 (GE Energy Germany GmbH, Ratingen, Germany)

4 Results and Discussion

Influence of spray parameters on the conductivity of $TiO_X/20Cr_2O_3$

To determine a stable process window with homogenous and robust coating properties, a design of experiments (DoE) is chosen around the current and gas flow for the APS process, using C45U as the substrate material. The influence of these parameters on the electrical resistivity is evaluated using the response surface method (RSM) with a second order correlation model combining linear and non-linear terms. A statistically significant influence on the resistivity (p = 0.05) is identified for the investigated parameters (Fig. 3) [BWH+21a].

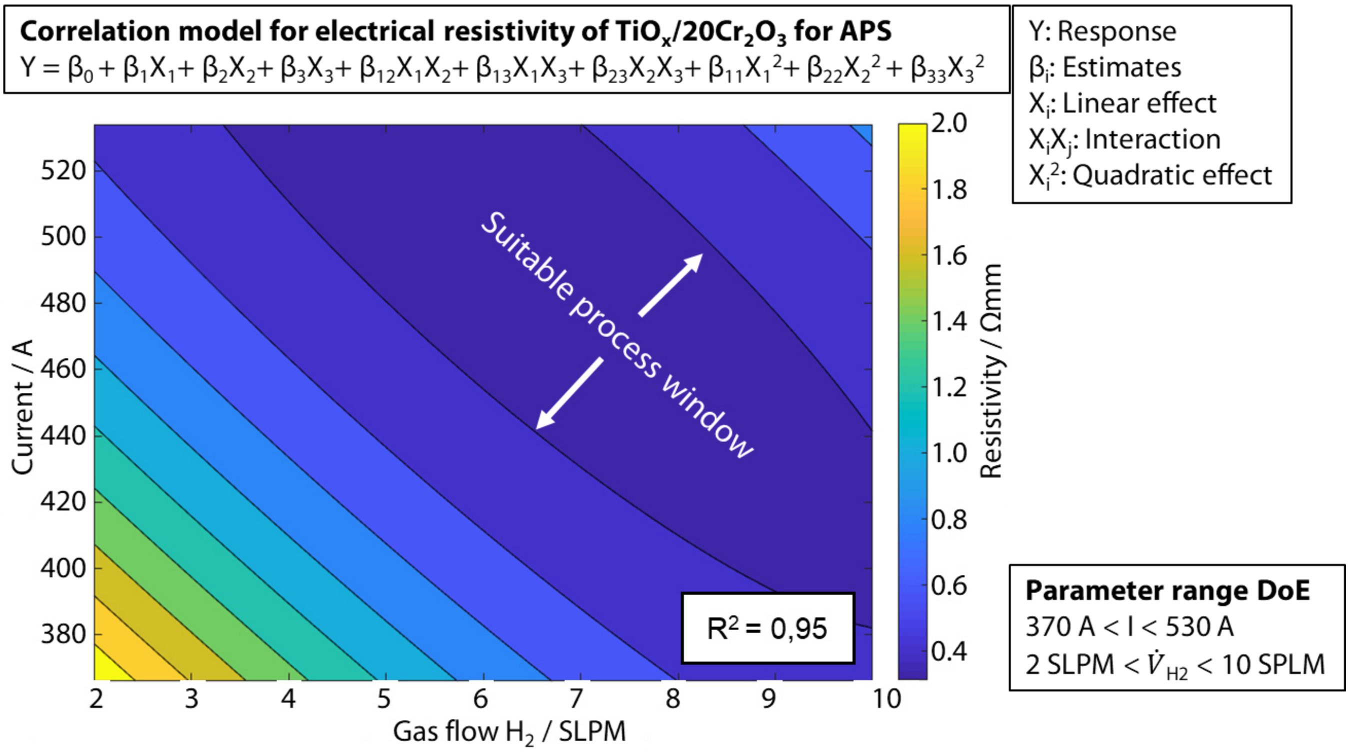

Figure 3: Correlation between plasma current, H_2 gas flow and resistivity for $TiO_x/20Cr_2O_3$. Values for the correlation coefficients can be found in [BWH+21a]. Modified after [BWH+21a].

As shown in Fig. 3, high currents and increased H_2-flow rates result in a wide window with homogenous properties, which corresponds to the lowest measured resistance of R ~ 0.5 Ωmm. Measurements of particle temperatures using SprayWatch 4S (Oseir Ltd., Tampere, Finland) indicate that this process window is associated with relatively homogeneous particle temperatures above T = 2,500 °C. Lower current values and reduced H_2 flow lead to lower particle temperatures below T = 2,500 °C and increased scatter in the measured resistance. This behavior may be attributed to a higher fraction of unmelted particles, which is also associated with increased coating porosity in the coatings [Sch24].

Measurable variations between powder batches prior to coating have little influence on the phase composition of the final coating [BWH+21a]. This might be explained by the oxidative effects of the atmosphere during spraying, which are countered by the reductive properties of H_2 secondary gas. The resulting equilibrium likely governs the formation of the sub-stoichiometric phases, which do not significantly vary in between different coatings [BWH+21a].

Durability of the heating coatings

Heating coatings must withstand thousands of heating cycles for economic viability. It was found, that the electrical connection to the heating coating represents a significant point of failure [BWH+21b]. A substantial increase in the number of heating cycles can be achieved through an improved contact design based on isolated contact screws, which is also better suited for in-mold applications [Sch24]. Using the improved electrical connection and the parameter set derived from the correlation model (Table 1), cyclic heating tests are conducted (Fig. 4).

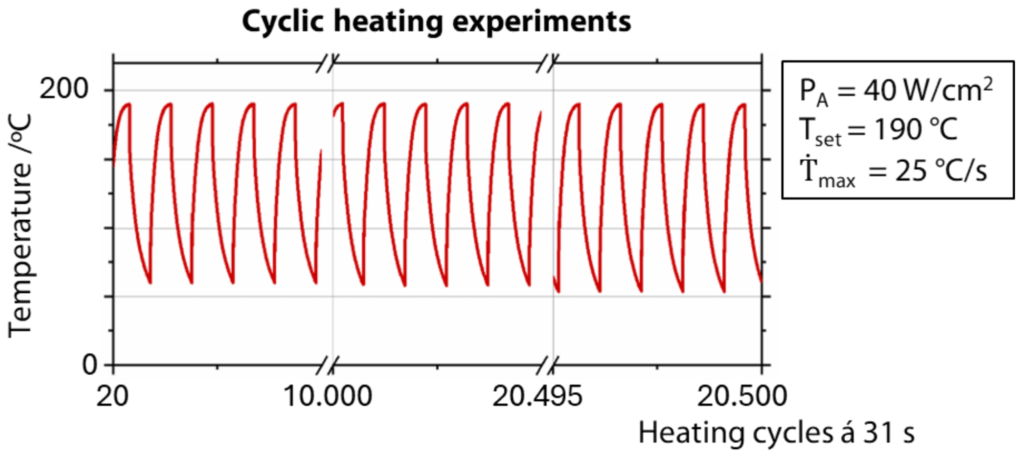

Figure 4: Cyclic heating experiments with optimized heating coatings for flat sample geometries. Modified from [Sch24].

Ceramic $TiO_x/20Cr_2O_3$ heating coatings can withstand more than 20.000+ heating cycles in the range of T = 50 °C to T = 190 °C, with a cycle time of t = 31 s. The cycle number was limited by experimental time rather than sample failure. At a power density of P_A = 40 W/cm^2, a maximum heating rate of $\dot{T}_{max}$ = 25 °C/s can be achieved. These findings underline the suitability of the $TiO_x/20Cr_2O_3$ heating coatings for applications with rapidly changing temperature levels, such as variothermal injection moulding.

Complex geometries

In cooperation with the project partners of the plastic processing institute (IKV), geometries with pronounced warpage are identified. Simulation data obtained using GOM-Inspect indicate that in box geometries, corners are in danger of warpage, as the outermost and innermost corners experience different cooling rates. While the outer corner have already solidified, the inner corners may remain molten, resulting in steep gradients in specific volume [HBK+23]. To homogenize temperature distributions in corners, triangular and strip-like heating coating designs were investigated. The simulation results show, that strip-like heating coatings are superior for warpage compensation, achieving up to an eightfold reduction in warpage [HBK+23] (Fig.5a). The application of multilayer heating coating systems on freeform geometries, such as parts with radii, is challenging because the spray angle can deviate from the ideal 90° angle. To investigate these effects, the spray angle is gradually reduced by an angle α, down to α = 45° and the electrical properties of the coatings were measured (Fig 5b). At a deviation of α = 45°, the resistance of the $TiO_x/20Cr_2O_3$ heating coating increased significantly. This can be partially attributed by the reduced coating thickness resulting from decreased deposition efficiency, as well as increased porosity. Furthermore, the increase in resistance may also be caused by prolonged particle in-flight oxidation, as the particles traverse longer paths at higher spray angle deviations geometrically [BHE+24].

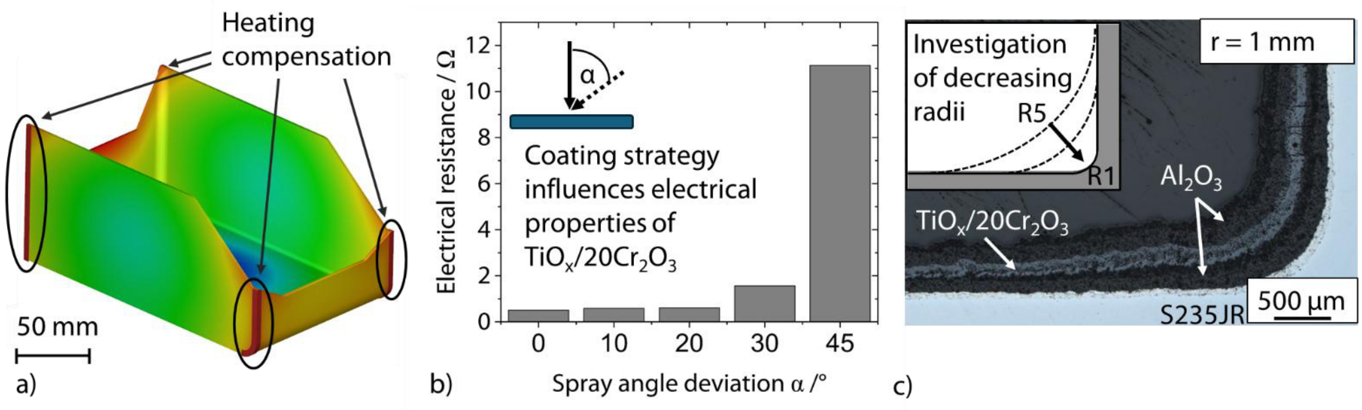

Figure 5: a) Simulative data on the requirements of heating compensation in box shaped geometries [HBK+23] b) Electrical resistance in dependence to the spray angle. Modified from [BHE+24] c) Cross section of sample with a radii of r = 1 mm

Based on these findings, coating strategies are developed for the coating of radii (Fig 5c). With a vertical coating strategy, the radii are coated in one continuous step, while with the orthogonal strategy, each side is coated in a 90° angle. This affects the temperature distribution during heating: the orthogonal approach results in higher temperatures at the vertex of the coating, while the vertical approach produces higher temperatures adjacent to the vertex [BHE+25a]. Using these strategies, radii up to r = 1 mm can be coated and withstand over 3.000+ heating cycles in the temperature range of T = 70 °C to T = 220 °C (Fig. 6a) [BEJ+26].

Cooling represents a major limitation on achievable cycle times. When applied to a model tool with internal cooling channels (Fig.6b), active cooling of the geometry enabled cycle times as low as t = 16 s within the temperature range of T = 70 °C to T = 150 °C [BEJ+26]. These results demonstrate, that the developed heating coatings can meet the requirements predicted by simulation data [HBK+23] for warpage compensation in box-shaped geometries.

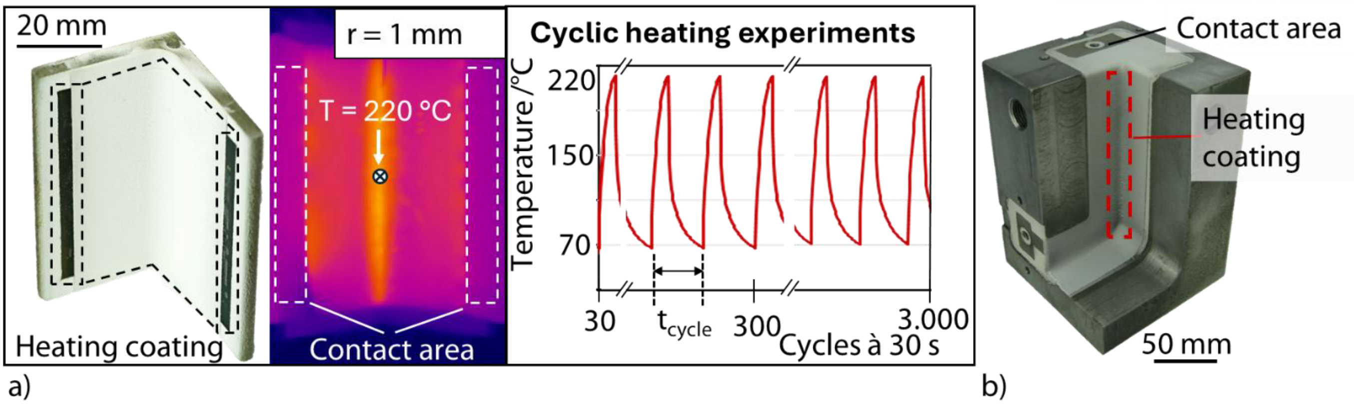

Figure 6: a) Cyclic heating experiments for a sample prepared by the orthogonal coating strategy b) Model of tool geometry.

Temperature control

As a semiconductor, thermally sprayed $TiO_x/20Cr_2O_3$ coatings exhibit negative temperature coefficient (NTC) behavior which means that the electrical resistance of the coating decreases with increasing temperature. In this case its relation is in a non-linear manner, which can be described by an Arrhenius-equation (Fig. 7b) [BWK+20].

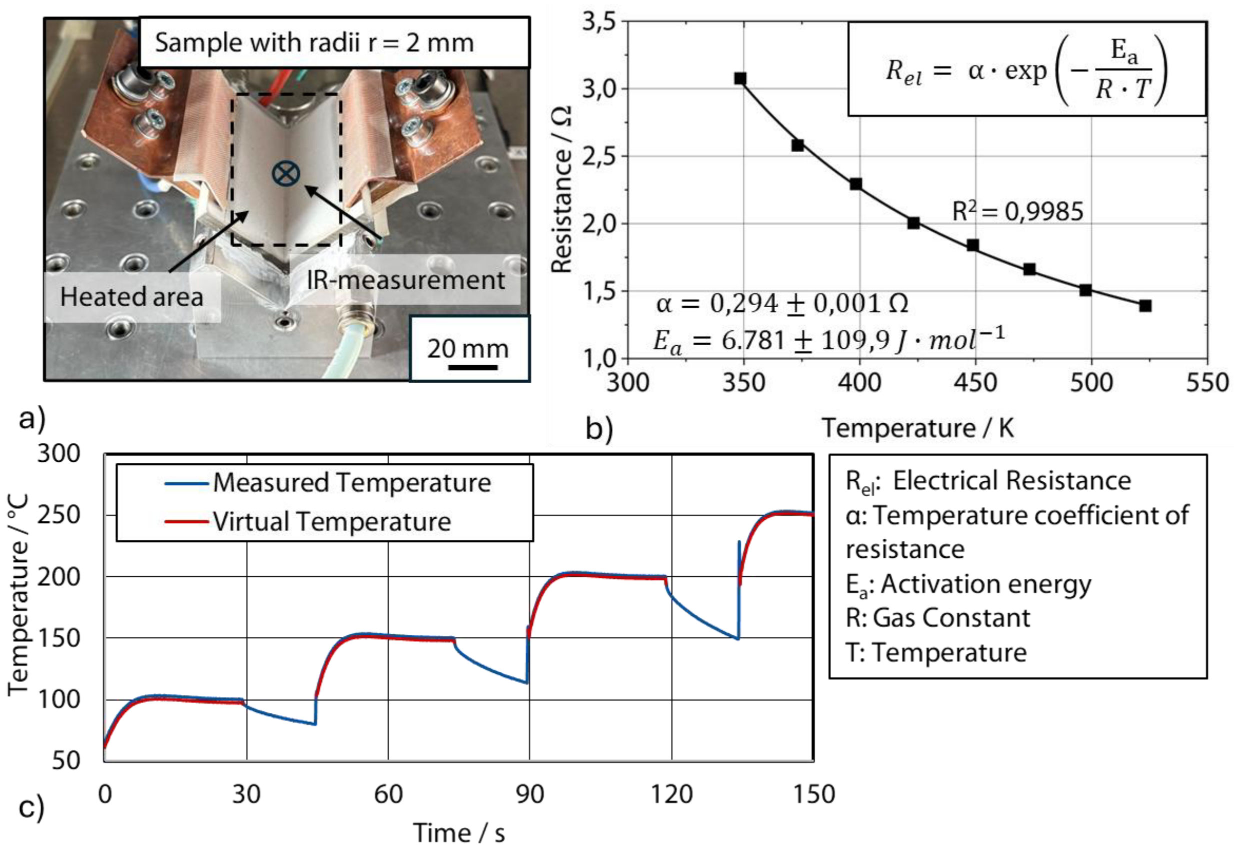

Figure 7: a) Experimental setup inside the test bench b) Temperature dependent resistance for the $TiO_x/20Cr_2O_3$ heating coating on the L-shaped sample c) Thermal control using the temperature dependent resistance

By determining the parameters of this Arrhenius-Equation, sensor applications of the coating become feasible [BWK+21]. Furthermore, temperature control of the heating coating through its temperature dependent resistance can be achieved, as demonstrated for plate geometries [BHE+25b]. This property is particularly relevant for applications in mold geometries, where space for external sensors is often limited. To investigate this, the application on free form geometries is tested using samples with a radius of r = 2 mm (Fig 7a). The activation energy E_a of the charge carriers was determined as E_a = -6.781 J/mol in good agreement with previous reports of E_a = - 7.072 J/mol [BHE+25b]. However, the temperature coefficient α differs between free form and flat geometries by a factor of two. This difference may arise from variations in overall resistance or from microstructural differences. This warrants further investigation, as it may allow for thermal control without the need for prior calibration. Good agreement was observed between temperatures measured with external IR-sensors and the virtual temperature derived from the sensor properties (Fig 7c), demonstrating the viability of this approach for tool geometries.

Application in injection molding

To verify the performance of the thermally sprayed heating coatings in casting processes, coated tool inserts were integrated into an industrial injection mould. (Fig .8a) [FHK+21]. The investigated part geometry exhibits warpage due to structural ribs on the backside of the sample, which locally increases part mass and reduces the cooling rate.

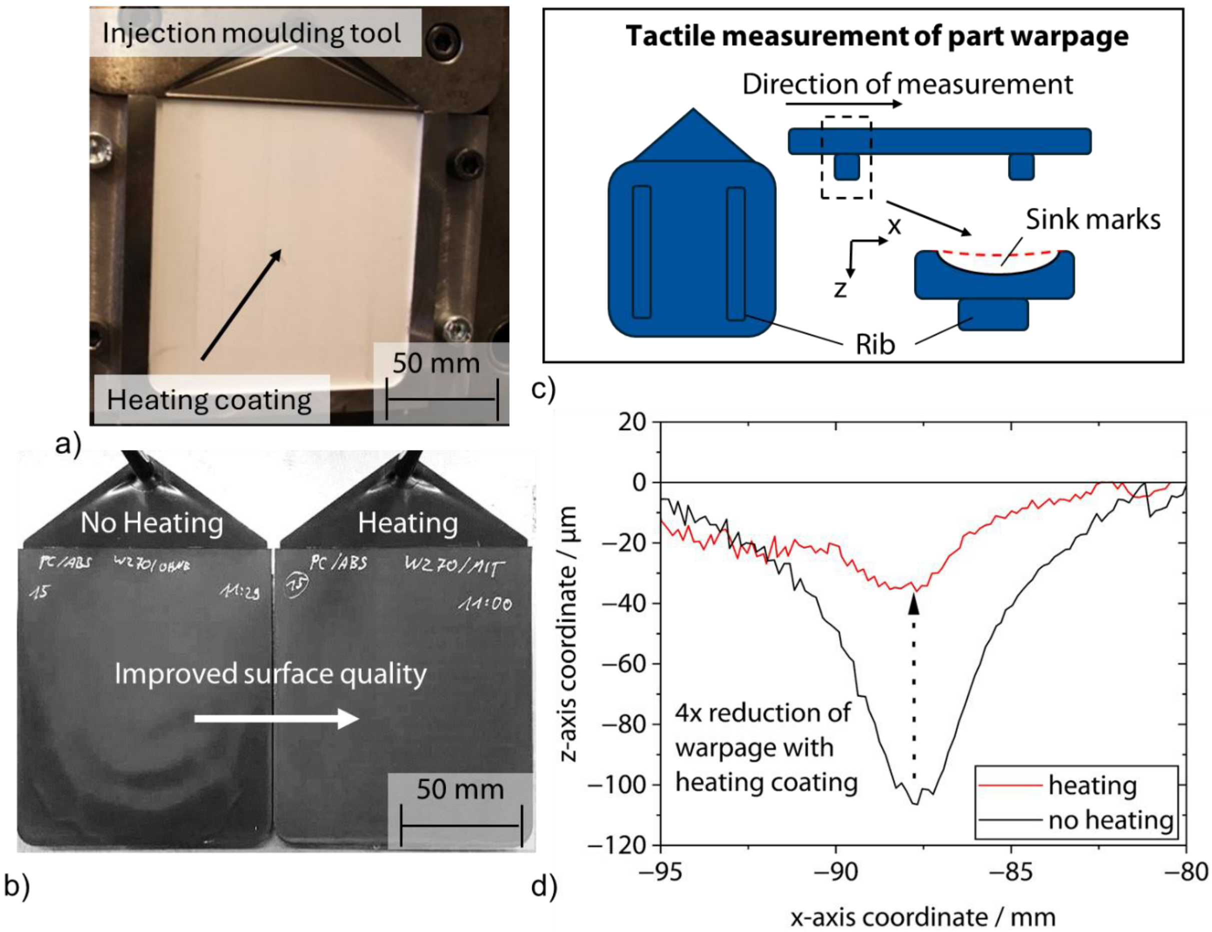

Figure 8: a) Injection moulding tool with integrated heating coating. Taken from [Sch24]. b) Improved surface quality in parts produced with active heating coatings c) Schematics of part geometry and tactile measurement d) Tactile measurements of parts fabricated with heating coatings. Data from [FHK+21]

The use of the heating coating results in an overall improved surface quality for the tested samples (Fig. 8b). For polypropylene (PP), pronounced sink marks are observed, caused by the uneven cooling between the tool sides, resulting in reduced part accuracy (Fig 8c) [FHK+21]. To compensate for this, active heating with thermally sprayed coatings is applied up to T = 120 °C. Warpage is reduced by a factor of four compared to samples without active heating. More than 400 parts can be produced using the active heating coatings without significant changes in heating behaviour [FHK+21].

In the next steps, the obtained knowledge on the coating of free-form geometries is applied on the box-shaped demonstrator shown in Fig. 5a. Using four coated segments (Fig. 6b), the warpage compensation is tested for more advanced tool geometries to advance the application field of the coatings.

5 Summary

Thermally sprayed heating coatings based on $TiOx/20Cr_2O_3$ enable rapid surface heating, making them suitable for applications where thermal compensation is needed. The properties of the coatings are influenced by the process parameters in the plasma spraying process. Ceramic coatings exhibit high durability, withstanding multiple thousands of rapid heating cycles. Multilayered coatings can be applied to freeform geometries, such as box-shaped parts with radii. $TiO_x/20Cr_2O_3$ provides thermal control through its temperature-dependent resistance. By using active heating coatings in injection moulds, warpage can be reduced significantly.

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Conflict of Interest

The author declares no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request on http://hdl.handle.net/ using the persistent identifier (PID): 21.11102/63d2e243-6847-4ce5-961b-c9b0d78605ee

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