

Control System Design by Multicriteria Selection in Microwave Sintering Processes^{*}

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Abstract: Microwave sintering has emerged in recent years as a non-conventional method for sintering materials with significant advantages against conventional procedures. By microwave sintering are achieved higher density ($\sim 99\%$), hardness (18 GPa) and fracture toughness ($6.3\text{ MPam}^{\frac{1}{2}}$) properties and homogeneous micro-structure compared to conventional heating. Reason why, the design of microwave systems, including adaptive control schemes, becomes a very important topic to be performed. The present work describes the microwave cavity control system design for sintering alumina-zirconia nanocomposite. The samples were sintered in a multimode microwave furnace (2.45 GHz) at different temperatures ($1200\text{--}1400\text{ }^{\circ}\text{C}$) in air. The main challenge from the control point of view is to guarantee an small tracking error, when a temperature profile is required, due to the nonlinear dynamical behaviour of nanocompistes.

Keywords: Microwave ovens, Adaptive control, Nanomaterials, Ceramics; Microstructure, Multicriteria Design, PID.

1. INTRODUCTION

The application of microwave energy to the processing of various materials such as ceramics, metals and composites offers several advantages over conventional heating methods. These advantages include unique micro-structure and properties, improved product yield, energy savings, reduction in manufacturing cost and synthesis of new materials (Sutton (1989)). Microwave heating is fundamentally different from the conventional one in which thermal energy is delivered to the surface of the material by radiant and/or convection heating that is transferred to the bulk of the material via conduction and also, the non-conventional sintering technique as spark plasma sintering (SPS), where it is possible to consolidate powder compacts by applying an on-off dc electric pulse.

In ceramic materials, the high temperatures required to fully densify ceramic powders result in large grain sizes due to Ostwald ripening when traditional sintering techniques are used. This makes it extremely difficult to obtain dense materials with nanometric and sub-micrometric grain sizes (Anselmi-Tamburini et al. (2006)). To overcome the problem of grain growth, microwave sintering (MW) has been proposed in this work with an efficient technique for hin-

dering the grain growth as well as producing a homogeneous micro-structure. Microwave radiation for sintering of ceramic components has recently appeared as a newly focused scientific approach (Binner et al. (2008); Borrell et al. (2012)). Recent works have proved the performance of the sintered materials either in rectangular (Borrell et al. (2012)) or cylindrical (Guyon et al. (2011)) cavities, providing really good mechanical properties (Vleugels et al. (2011); Borrell et al. (2012)).

However, the sintering of materials requires an accurate heating process, in fact, each material has an optimal heating profile where the properties of the resulted nanocompistes are maximized. For that reason, the heating process has to be managed by an advanced control system which could guarantee an optimal tracking behaviour. Moreover, the control system has to deal with strong nonlinear dynamical behaviours produced by structural changes of the sinterized materials during the heating process. Therefore, the control system scheme and the control parameters adjustment are determinant from the control point of view. This work presents the control system design for a rectangular cavity, based on the TE_{101} mode, to accurately track a predefined temperature profile, obtaining $Al_2O_3 - ZrO_2$ nanocomposites with high properties. The main algorithm included in the control system is the well known PID; however, the selection of the different parameters of that classical control scheme have been selected using multicriteria methodologies.

The research work done is summarized in six sections. First, sections 2, 3 and 4 describe the sintering process using microwave cavities, a brief description of nanocom-

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posites and the control scheme. Section 5 describes in detail the proposed design process to set up the PIDs using multicriteria design methods. Finally, section 6 shows the results obtained and some concluding remarks.

2. PROCESS DESCRIPTION

Figure 1 shows the system designed to sinter the materials. From right to left we can see: the 1 kW magnetron, including the feeding system, the circulator to prevent the power source from reflected power (including a coupling system to measure the reflected power with the polymer), the TE_{101} cavity, which will be described in detail in the next paragraphs, and a motorized short-circuit to tune the cavity and connected to a laptop to allow an automatic control.

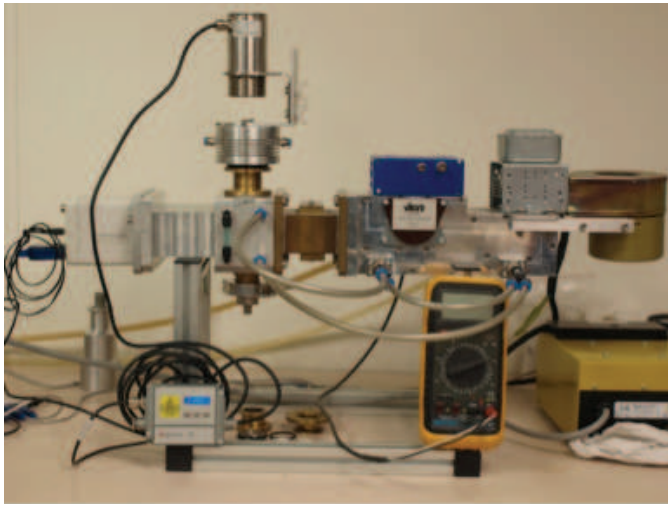


Fig. 1. Microwave system designed for material sintering.

The cavity, with rectangular shape, is used to excite the TE_{101} mode, based on a $WR340$ waveguide and excited through a circular iris to maximize the H_x magnetic component of the TE_{101} mode. The sample is introduced in the cavity through an insertion hole located just in the center of the cavity and on the top of it. The insertion hole has a diameter of 3cm to guarantee that the insertion hole is under cut-off and no microwave energy leakage through it with the TE_{10} mode, which is the fundamental mode in the cylindrical waveguide. In the center of the cavity the excited mode has a maximum of E_y component, and a behaviour of a $\sin(\cdot)^2$ in the axial component and no variation in the vertical component, which guarantees the homogeneity of the field in the sample, which is supposed to be about 1cm of diameter as maximum. To allocate the sample in the center, a quartz tube is used, which is transparent to the microwave energy and allows centering the sample. It also allows introducing different atmosphere (or even vacuum). To monitorize the temperature, a pyrometer is located on the top, as shown in figure 1. This allows including a control system to automatize the heating process, based on a hybrid PID system. A close scheme of the cavity is shown in the next figure 2, where the position of the sample is shown and also the sliding short-circuit that changes the resonant frequency of the cavity and is controlled by the PID described in the next section to guarantee the tuning of the cavity.

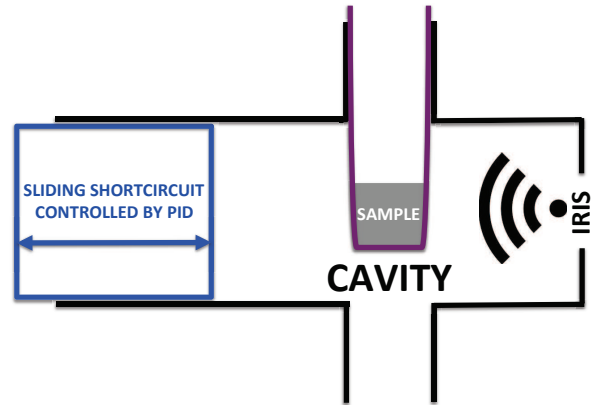


Fig. 2. Cavity scheme.

3. CONTROL SYSTEM SCHEME

Due the strong nonlinear behaviour of the heating process, a control and monitoring system has been included as a new component in the microwave cavity design. The main goal of this new subsystem is to manage and optimize the sintering process along the heating-cooling stages.

The main physical variables involved in sintering process, as temperature and microwave energy, exhibit both continuous and discrete dynamic behaviour. For that reason, a hybrid control structure has been selected (Goebel et al. (2009)). This type of controllers can manage both real-time feedback loops, based on PIDs algorithm, to manage continuous variables and a logical decision-making state-machine focused on discrete events.

Figure 3 shows the basic functionality diagram for the hybrid control system included in the new prototype. This scheme describes the relation between the six-states discrete machine and the PIDs continuous controllers.

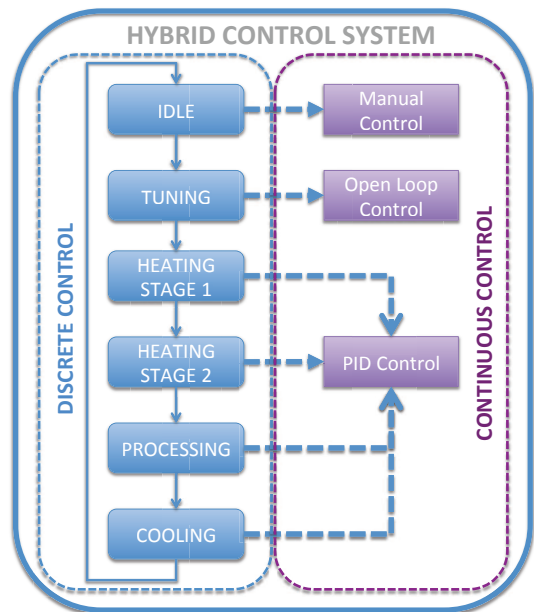


Fig. 3. Sintering hybrid control system.

The adjustment of control systems is a critical task and is strongly dependent on the continuous algorithms involved in the system. In that case, the main continuous structure

to be adjusted is the well-known PID ISA algorithm (Ang et al. (2005)).

$$U(s) = K_R(1 + sT_D + \frac{1}{sT_I}) \quad (1)$$

$$u(t) = K_R \left(e(t) + \frac{de(t)}{dt}T_D + \frac{1}{T_I} \int_{\tau=0}^t e(\tau)d\tau \right) \quad (2)$$

Where,

	Descripción
s	Laplace transform
$e(t)$	Error, computed as the difference between a desired setpoint and a measured variable
K_R	Proporcional term, weight of the <i>present</i> error in the control action
T_I	Integrative term, weight of the accumulation of <i>past</i> errors in the control action
T_D	Derivative term, weight of the <i>prediction</i> of future errors in the control action

K_R , T_I and T_D have been adjusted using optimization techniques based on simulation schemes, where the main goal is to guarantee an appropriate closed loop dynamic behaviour from the temperature point of view (see section 5).
hours

4. EXPERIMENTAL PROCEDURE

The materials used for the preparation of the nanocomposites were commercial alumina *AKP-53* provided by *Sumitomo Co., Japan* (purity 99.99%, mean particle diameter of 200nm), and nanometric zirconia (purity 99.99%, monoclinic, primary sized particles from 60-100nm) provide by *Nanostructured Materials, Inc.* The procedure of dispersing the zirconia powder in the Al_2O_3 powder involves the preparation 15 vol.% of ZrO_2 suspension through traditional milling. Cylindrical specimens with 10mm diameter and about 5mm height were prepared, by isostatic pressing (200MPa). After, green samples were sintered under air by MW at 1200°C, 1300°C and 1400°C using the heating rate of 100 $\frac{^\circ C}{min}$ with 1-minutes of holding time at the maximum temperature. The temperature of the sample is monitored by an infrared radiation thermometer (*Optris CT-Laser LT, 8-14 um*), which is focused on the test sample via the small circular aperture in the wall of the test cell. The emissivity and transmissivity of the material at different temperatures were calculated previously to sintering. For comparison, the same green compacts were conventionally sintered at 1300°C and 1400°C using the heating rate of 15 $\frac{^\circ C}{min}$ with for 2-hours of holding time at the maximum temperature. These temperatures were measured using a pyrometer positioned within the cavity of furnace. These samples also were sintered in a *Netzsch-DIL 402C* dilatometer at 15 $\frac{^\circ C}{min}$ constant heating rate in air atmosphere. Based on these results, steps for the sintering were defined. The bulk density of the sintered samples was measured by Archimedes principle. Vickers hardness (*ASTM E92-72*) and fracture toughness assessments were carried out using the indentation method with a conventional diamond pyramid indenter. In order to investigate sample microstructure, polished sections were thermally etched between 15 minutes in an electrical furnace under air 100 °C below their maximum sintering temperature to

reveal their microstructure. The microstructure sections have been observed using a field emission scanning electron microscope (*FEG-SEM, S4800 HITACHI, SCSIE*). The grain size of the sintered samples was determined by multiplying the average linear intercept by 1.56.

5. PID TUNING CONTROLLER

5.1 Model identification

Successive step reference changes have been made from temperature $T_e^{initial} = 500^\circ C$ to $T_e^{end} = 1200^\circ C$, in order to identify a set of transfer functions with the following structure:

$$P(s) = \frac{k}{T_s + 1} e^{-Ls} \quad (3)$$

where k is the process proportional gain, T the time constant and L the lag of the system. The resulting models are depicted in Figure 4. It is important to notice differences among models concerning k , T and L values, showing the high non-linearity of the process.

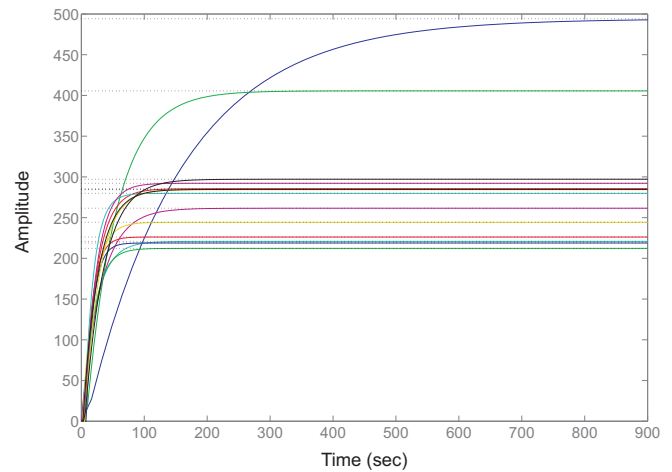


Fig. 4. Step response for identified models of the microwave sintering process at different temperatures T_e .

As nominal model is selected the identified transfer function at temperature $T_e = 850^\circ C$ (Equation 4).

$$P(s) = \frac{272}{17.25s + 1} e^{-3.85s} \quad (4)$$

5.2 Tuning procedure candidates

The following tuning procedures have been selected as candidates for implementation:

- Cohen Coon tuning method, with a 25% and 10% decay ratio respectively.
- Skogestad PI controller tuning, with $\tau_c = 0$.
- Lambda tuning method for PI and PID controllers, with $\lambda = 0$.

As we require a PID controller tuning to track a ramp signal reference (not step change references, due to the nature of the sintering process), we justify the aforementioned controllers and their tuning parameters.

5.3 Multi-criteria decision analysis

It is an accepted fact that there is not an overall good set of parameters for a PID controller. This is because most

Table 1. Controller performance on simulated models.

Controller	$J_1(k_p, T_i, T_d)$	$J_2(k_p, T_i, T_d)$	$J_3(k_p, T_i, T_d)$	$J_4(k_p, T_i, T_d)$
Cohen Coon 1	3.73	5.06	1.68	0.38
Cohen Coon 2	9.05	4.99	4.56	0.38
Skogestard	4.94	5.17	1.65	0.37
Lambda 1	5.06	5.10	1.44	0.37
Lambda 2	1.82	5.26	0.33	0.32

of the times, the control engineer is looking for a set of parameters with a desired (or affordable) trade-off among different objectives and specifications. Therefore, different parameters tuned for a PID controller could have different trade off performance and a multi-criteria decision making process needs to be carried out by the engineer (Reynoso-Meza et al. (Accepted)).

Even as we are using well known and reliable tuning techniques, it is needed to evaluate their overall performance before being implemented. Four objectives have been selected to evaluate their functionality:

$$J_1(k_p, T_i, T_d) = e(k)_{max}|_{P(s)} \quad (5)$$

$$J_2(k_p, T_i, T_d) = \sum_{k=T_0}^{T_f} |u(k+1) - u(k)| \Big|_{P(s)} \quad (6)$$

$$J_3(k_p, T_i, T_d) = \sigma \left(e(k)_{max}|_{P'(s)} \right) \quad (7)$$

$$J_4(k_p, T_i, T_d) = \sigma \left(\sum_{k=T_0}^{T_f} |u(k+1) - u(k)| \Big|_{P'(s)} \right) \quad (8)$$

where e_{max} is the maximum tracking error and $J_2(k_p, T_i, T_d)$ the total variation of the control signal. Objectives $J_1(k_p, T_i, T_d)$, $J_2(k_p, T_i, T_d)$ have been calculated through simulation using the nominal process selected (Equation 4). Objectives J_3 , J_4 have been calculated using a Monte-carlo approach with 1000 uniformly random $P'(s)$ selected models, with intervals $k = 272 \pm 20\%$, $T = 17.25 \pm 50\%$ and $L = 3.85 \pm 100\%$. Such intervals have been determined from the set of models previously identified (Figure 4). This is justifiable, since the internal and microscopic characteristics of materials using in the sintering process, although the same, could considerable change from sample to sample.

In table 1, the values of the four objectives are shown whilst in Figure 5 they are depicted using parallel coordinates (Inselberg (2009, 1985)). According to simulations, Lambda 2 controller shows a most preferable behaviour, with the lowest maximum error and variability.

6. RESULTS AND CONCLUSIONS

In figures 6, 7 and 8 the performance on the sintering process at three different stages are presented. In table 2 the maximum error is the same stages is shown. The observed behavior from table 2 is consistent with the one depicted at table 1. Therefore, this validates the control objectives selected for the multi-criteria analysis stage to analyze and select the most preferable tuning technique for the sintering process. As result of experiments, lambda-2 tuning has been selected for the physical microwave system for sintering.

In summary, the developed control system managed successfully the microwave-cavity to obtain dense Al_2O_3 -

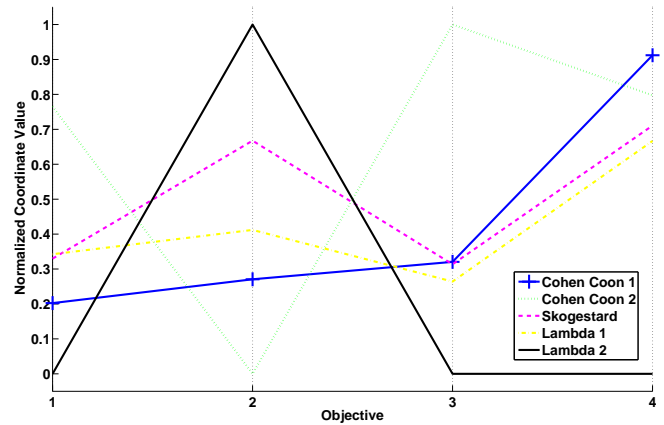


Fig. 5. Controller performance (on simulated models) visualization using parallel coordinates.

15 vol.%ZrO₂ nanocomposites within several minutes. Concerning the results of MW is that the density of a microwave-sintered material is larger than the density of a material conventionally sintered at the same measured temperature. More importantly, it shows that relative densities of 99% may be obtained without any crack. The sintering time required to achieve relative densities above > 98% is 350-minutes using CS, but the MW leads to the similar dense specimens in only 40-minutes. It is important to note that the final economic cost is very significant.

Concerning the adopted control approach, future work will focus on stating a holistic optimization approach, by means of multiobjective optimization. Also, this approach should be tested and evaluated with other standard techniques, as gain scheduling. Finally, more work on non-linear modelling should be carried out.

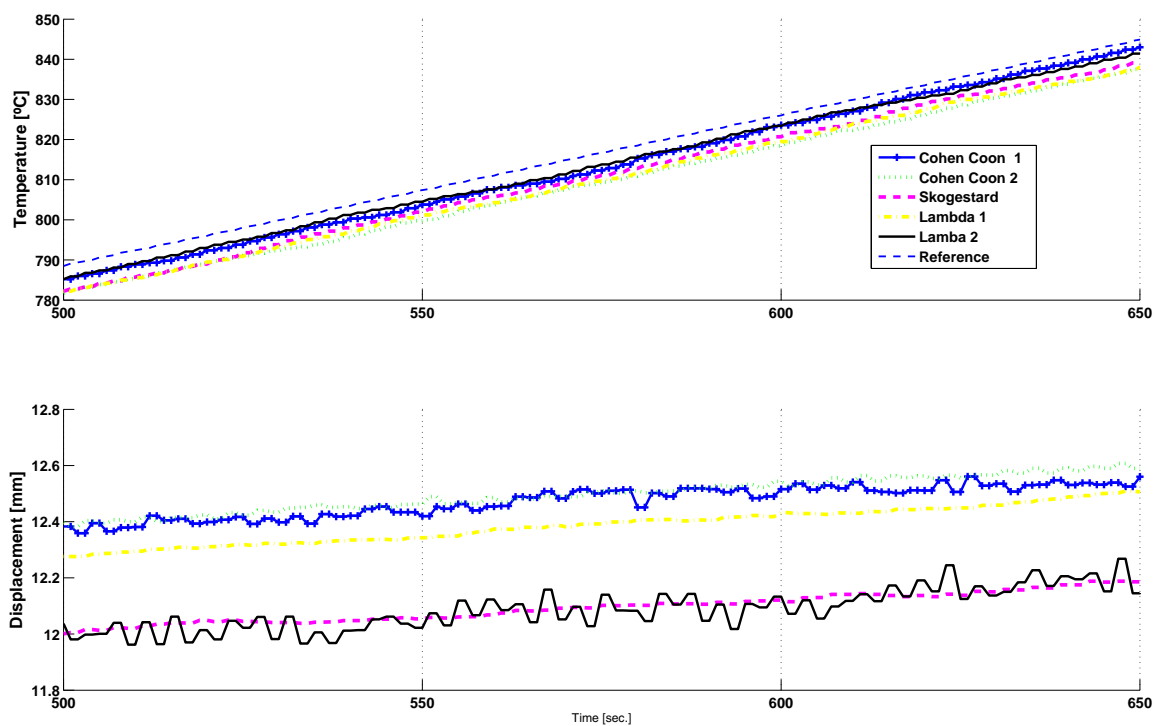


Fig. 6. Performance of selected controllers around $T_e = 800^\circ C$ on the physical microwave system for sintering.

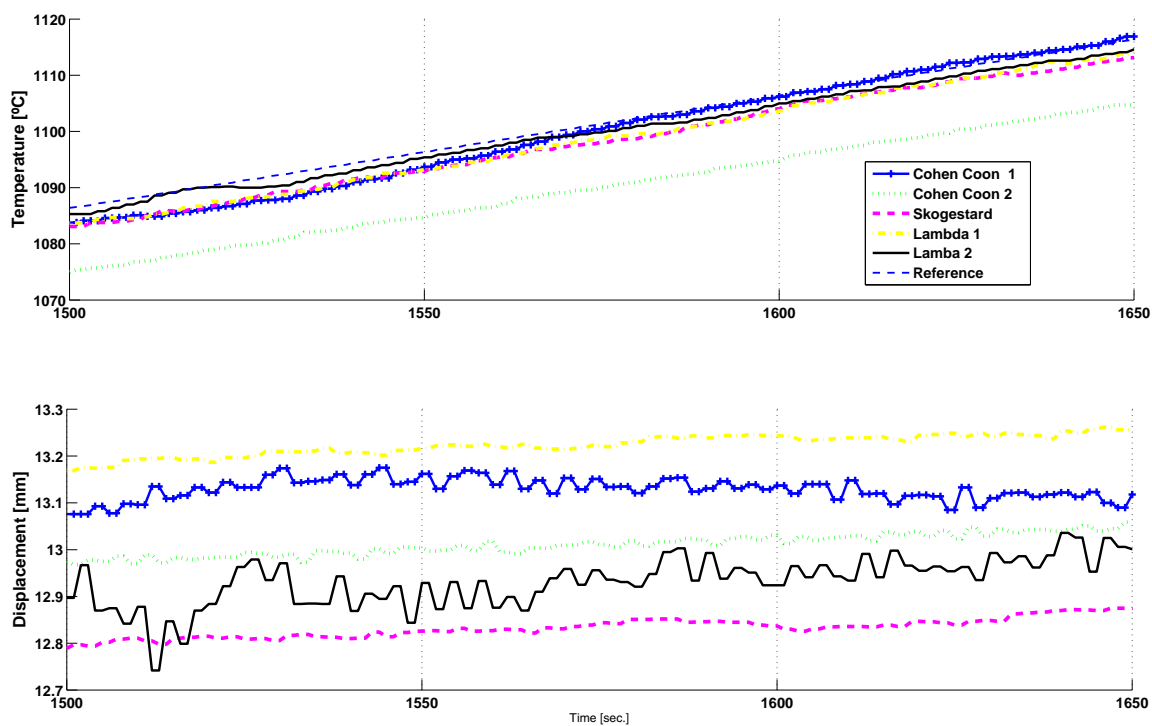


Fig. 7. Performance of selected controllers around $T_e = 1100^\circ C$ on the physical microwave system for sintering.

Table 2. Performance indicators on the physical microwave system for sintering. Maximum error [°C] is depicted for the specified intervals.

Controller	$T_e = [600^{\circ}C, 800^{\circ}C)$	$T_e = [800^{\circ}C, 1200^{\circ}C)$	$T_e = 1200^{\circ}C$.
Cohen Coon 1	5.5	5.58	5.4
Cohen Coon 2	9.4	7.46	7.5
Skogestard	7.3	5.96	5.69
Lambda 1	8.2	5.06	4.80
Lambda 2	4.9	3.38	1.69

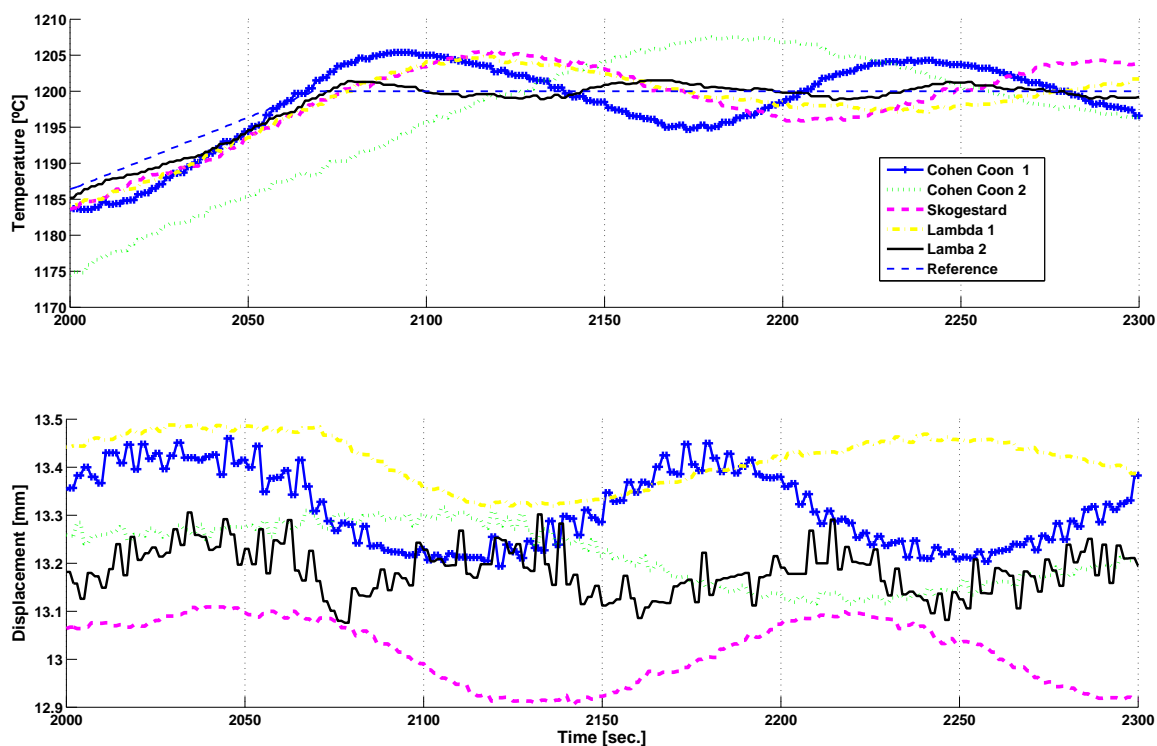


Fig. 8. Performance of selected controllers around $T_e = 1200^{\circ}C$ on the physical microwave system for sintering.

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