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Development of an Advanced Control System for a Chemical Vapor Deposition (CVD) Reactor for Polysilicon Production

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Traditionally high-grade polycrystalline silicon production is obtained by the Chemical Vapor Deposition (CVD) process in the so-called Siemens reactor. The problem of rod temperature control in such a reactor is broached in this work. An indirect estimation method of the polysilicon rod diameter and the temperature estimation by means of the rod electrical resistance are both proposed. The main issue to overcome is related to the intrinsically unsteady-state conditions of the Siemens reactor (and all the batch reactors as well). From this perspective, the problem of temperature measurement can be solved using the appropriate pyrometer, although the temperature at the center of the rod has still to be estimate numerically. The aim of this paper is to develop a novel adaptive control system for the Siemens reactor based on neuro-fuzzy approach for both the parameter tuning and the control loop itself. A comparative discussion between the proposed method and other advanced control techniques is proposed.

1. Introduction

The so-called "Siemens" technology, which is based on the Chemical Vapor Deposition (CVD) process, is the most widespread process for the production of high-purity polycrystalline silicon, the raw material most commonly used in photovoltaic cells and electronic devices. The industrial process is discontinuous since the reactor is a bell-like chamber containing a set of slim silicon rods, which are heated by intensive electrical energy supply. The rods progressively increase their volume due to the reduction of the hydrogen of the trichlorosilane (TCS) – $SiHCl_3$ (or decomposition of silanes – SiH_4), which is continuously fed to the reactor in gas phase. The rod diameter growth leads to an increase in the silicon deposition rate, and heat losses. Maintaining the necessary constant value of rod surface temperature requires an increase in the current, which is limited by mechanical stresses and the melting process of the silicon rod core. Controlling this process is troublesome due to uncontrolled radial temperature gradients inside the rods. This is influenced by two key factors: the distributed nature of the sources of internal heating by Joule effect and heat transfer with the environment. In response, the reactor requires an appropriate reduction of rods surface temperature by field/room operator during the campaign. An effective way to overcome this limitation is the use of high-frequency current sources (Kozin et al., 2014).

Polysilicon CVD production is very energy intensive and it is necessary to find a compromise between deposition rate and quality (purity and growing texture) of the final product. This goal can be achieved by rod temperature control and by suitably adjusting the required flow of reagents (with the proper molar composition) and silicon precursor per unit area of the rods. Since the deposition process takes place over several days, even small errors and any operating disturbance or set-point trajectory are cumulative effects. This greatly affects the quality and quantitative yield of final product along with the process costs.

In response minimizing the rods breakdown risk and ensuring an optimum silicon deposition conditions are a complex problem, especially in the absence of an efficient automatic control system (ACS). Therefore, the

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ACS design is critical and is primarily aimed at the control of some specific operating variables, such as the rod surface temperature and the flowrate of the reactants (silicon precursor and hydrogen). Electrical energy, whether properly employed, allows for a tight temperature control and a technique for the real-time model-based predictive control (MPC), applied to a laboratory-scale Siemens reactor, has been developed in (Viganò et al., 2010). Since the process is extremely energy intensive and models are always uncertain in some parameters, a robust optimal control problem has been formulated and solved based on the evaluation of Lyapunov equations, and the trade-off among productivity and energy cost has been studied via a multi-objective (MO) scalarization method too (Vallerio et al., 2014).
In this context, the aim of this work is the development of a new ACS for the Siemens reactor with the engineering feasibility from a practical perspective. However, its design is also significantly complicated by some factors as the increase in the diameter of the rods due to the silicon deposition, the decrease in the silicon electrical resistance with the increase in temperature and the possibility of measuring the rod temperatures only at some single points on their surface by a pyrometer. For this reason, it was decided to design an adaptive ACS for the rods temperature, including an approach for the indirect estimation of rods diameter and rods resistance. In addition, the influence of neuro-fuzzy approaches to the control performance is investigated when these methods are used in single elements of the adaptive ACS (the parameter adjustment block and the controller itself). The control method testing consists in a comparison made with three different adaptive ACSs.

2. Mathematical model of a CVD reactor as the controlled process

Polysilicon production is carried out in a kinetic regime defined by the surface reactions of the hydrogen contained in chlorosilanes. The chemistry of the TCS ($SiHCl_3$) hydrogen reduction is rather complicated. At typical temperatures for the Siemens-process, chemical reactions are not represented only by the silicon deposition, but also by the formation of new silicon-containing compounds, both in homogeneous and heterogeneous reactions, including etching processes. The most common compounds produced in the reduction of TCS are: HCl, H_2, $SiHCl_3$, $SiCl_4$, $SiCl_3$, SiH_2Cl_2, $SiCl_2$ (Woodruff et al., 1985). The most complete kinetics of the reduction of $SiHCl_3$ is presented by Valente (2001). Considering H_2, $SiHCl_3$, HCl, $SiCl_4$ as the main compounds, the reduction of the TCS hydrogen is modeled trough the following macro-reactions:

$$SiHCl_3 + H_2 \xrightarrow{k_1} Si + 3HCl \quad (1)$$

$$4SiHCl_3 \xrightarrow{k_2} Si + 3SiCl_4 + 2H_2 \quad (2)$$

The design and numerical research about suitable control systems for the Siemens-reactor led to mathematical model of the controlled plant, which describes the deposition of silicon, realizing a dynamic direct link between the basic technological variables, and takes into account the radial temperature profile in the silicon rods. The model assumes the perfect mixing in the gas phase. Adsorption, diffusion and neighboring rods mutual influence are neglected. Chemical reactions take place into boundary layer. The ideal gas law is used, given the composition and temperature of the system. The first-principles formulation of mass and energy balances that characterize the system are reported below:

$$\frac{dQ_g}{dt} = \left(Q_{SiHCl_3} + Q_{H_2} - Q_g\right)\frac{Q_{SiHCl_3} + Q_{H_2}}{V} \quad (3)$$

$$\frac{dC^r_{SiHCl_3}}{dt} = -k_1 C^r_{SiHCl_3} C^r_{H_2} - k_2 C^r_{SiHCl_3} + h_{m,SiHCl_3}\left(C^V_{SiHCl_3} - C^r_{SiHCl_3}\right)\frac{S_r}{V_r} \quad (4)$$

$$\frac{dC^V_{SiHCl_3}}{dt} = {}^1\!/_V\left(-h_{m,SiHCl_3} S_r\left(C^V_{SiHCl_3} - C^r_{SiHCl_3}\right) + \frac{Q_{SiHCl_3} P}{R_g T_g^{in}} - C^V_{SiHCl_3} Q_g\right) \quad (5)$$

$$\frac{dC^r_{H_2}}{dt} = -k_1 C^r_{SiHCl_3} C^r_{H_2} - \frac{1}{2} k_2 C^r_{SiHCl_3} + h_{m,H_2}\left(C^V_{H_2} - C^r_{H_2}\right)\frac{S_r}{V_r} \quad (6)$$

$$\frac{dC^V_{H_2}}{dt} = {}^1\!/_V\left(-h_{m,H_2} S_r\left(C^V_{H_2} - C^r_{H_2}\right) + \frac{Q_{H_2} P}{R_g T_g^{in}} - C^V_{H_2} Q_g\right) \quad (7)$$

$$\frac{dC^r_{SiCl_4}}{dt} = \frac{3}{4} k_2 C^r_{SiHCl_3} + h_{m,SiCl_4}\left(C^V_{SiCl_4} - C^r_{SiCl_4}\right)\frac{S_r}{V_r} \quad (8)$$

$$\frac{dC^V_{SiCl_4}}{dt} = {}^1\!/_V\left(-h_{m,SiCl_4} S_r\left(C^V_{SiCl_4} - C^r_{SiCl_4}\right) - C^V_{SiCl_4} Q_g\right) \quad (9)$$

$$\frac{dC_{HCl}^{r}}{dt}=3k_1C_{SiHCl_3}^{r}C_{H_2}^{r}+h_{m,HCl}\left(C_{HCl}^{V}-C_{HCl}^{r}\right)\frac{S_r}{V_r} \tag{10}$$

$$\frac{dC_{HCl}^{V}}{dt}=\frac{1}{V}\left(-h_{m,HCl}S_r\left(C_{HCl}^{V}-C_{HCl}^{r}\right)-C_{HCl}^{V}Q_g\right) \tag{11}$$

$$\frac{dm_{Si}}{dt}=N_rM_{Si}V_r\left(k_1C_{SiHCl_3}^{r}C_{H_2}^{r}+\frac{1}{4}k_2C_{SiHCl_3}^{r}\right) \tag{12}$$

$$\rho_{\mathrm{Si}}c_{p,\mathrm{Si}}\frac{\partial T}{\partial t}=\frac{1}{r}\frac{\partial}{\partial r}\left(r\lambda\frac{\partial T}{\partial r}\right)+\frac{I^2}{\sigma(T,r)}\bigg|_{0\le r\le r_s} \tag{13}$$

$$-\lambda_{\mathrm{Si}}\frac{\partial T}{\partial r}=k_{conv}\left(T-T_g\right)+\varepsilon_r\sigma_B\left(T^4-T_w^4\right)\bigg|_{r=r_s} \tag{14}$$

$$\frac{\partial T}{\partial r}\bigg|_{r=0}=0 \tag{15}$$

$$\frac{dT_g}{dt}=\frac{1}{m_g\cdot c_{p,g}}\left[2\pi r_sL\cdot N_rk_{conv}\left(T-T_g\right)+\frac{PT_g^{in}}{R_g}\sum_{i=1}^{NC}c_{p,i}M_iQ_i-Q_gM_gc_{p,g}T_gV-S_wk_c\left(T_g-T_w\right)\right] \tag{16}$$

with Q_g – vapor-phase outlet flowrate, m^3/s; Q_i – inlet flowrate of *i*-reagent (H_2, $SiHCl_3$), m^3/s; V_r, V – boundary layer and vapor-phase volumes, m^3; S_r – boundary layer surface, m^2; S_w – wall surface, m^2; C^r_i – concentration on the rod surface, mol/m^3; C^V_i – vapor-phase concentration, mol/m^3;k_1 and k_2 – kinetic constants, m^3/(mol·s) and 1/s; $h_{m,l}$ – mass transfer coefficient, m/s; k_{conv} – convection coefficient, W/(K·m^2); P – vapor-phase pressure, Pa; R_g – universal gas constant, J/(mol·K); R – rod resistance, Om; L – rod length, m; r – rod radius, m; N_r – number of rods;. $c_{p,i}$ – specific heat, J/(kg·K); T_g^{in} – inlet flowrate temperature, K; T_g,T – vapor-phase and rod temperatures, K; T_w – wall temperature, K; M_{Si} – silicon molar mass, kg/mol; M_i – molar mass of *i*-reagent (H_2, $SiHCl_3$), kg/mol; m_{Si} – silicon mass, kg; m_g – vapor-phase mass, kg; ε_r – silicon emissivity; σ_B – Stefan-Boltzmann constant, *J*/(s·m^2·K^4).
A set of conditions used for the simulations is given in Table 1.

Table 1: Operating conditions

Parameter	Value	Parameter	Value
Rod surface temp (T_s)	1423 K	Kinetics constant (k_1)	0.36·exp(-10^5/(R_g·T)) m^3/(mol·s)
Wall temp (T_w)	373 K	Kinetics constant (k_2)	70·exp(-10^5/(R_g·T)) 1/s
Inlet flow temp (T_g^{in})	353 K	Convection coefficient (k_{con})	25 W/(K·m^2)
Number of rods (N_r)	12	Silicon thermal conductivity (λ)	39.5·$e^{-T/62.7}$ + 108·$e^{-T/332.7}$ W/(m·K)
Reactor volume (V_r)	3.34 m^3	Silicon emissivity (ε_{Si})	-2.79·10^{-4}·T + 0.93
Silicon density (ρ_{Si})	2,330 kg/m^3	Specific conductivity (σ)	1.85·106 $e^{-56300/(8.314\cdot T)}$ S/m

3. Design of the control system

Detailed simulations show that the reactor is in quasi-steady state and the process model for the temperature control loop can be described as a transfer function of the first order without delay time. It was found that, during the process integration, the time constant increases by 1 order of magnitude, and the gain decreases by more than 2 orders of magnitude. To design an ACS with that degree of non-stationarity an adaptive control method is used. To avoid insufficiency of data coming from the pyrometers, which are only placed in one single point of the rods, the rod resistance can be used as a controlled variable, since it is sensitive to even local rod temperature changes. The assumption that the controlled plant operates in quasi-steady state, while changing the manipulated variables within technical specifications, has been verified.

3.1 Adaptive control system based on a PI controller

A block diagram for the adaptive control system based on a proportional-integral (PI) controller is shown in Figure 1. An indirect estimation of the rods diameter (d_s) is based on the formula combining the temperature dependence of the conductivity of silicon and the calculation of the electrical resistance using Pouillet's law (see 'state estimator' block in Figure 1). Periodical measurements of the rods temperature T_p by means of a pyrometer with a definite sampling time are used to provide the ACS with stability guarantees. The value of d_p is estimated from the rods diameter d_s. A linear variation of d_p between the pyrometer sampling times is provided via a first-order extrapolator.

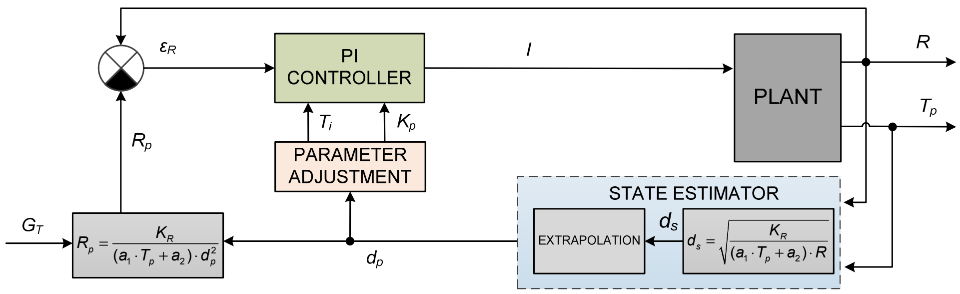

Figure 1. Control system based on a PI controller

The main control loop is based on a PI-controller where the error signal ε_R is the difference between the measured value of the rod resistance R and the corresponding target value R_p that allows to keep a specified surface temperature G_T. The controller receives the parameters (k_p – the proportional gain and T_i – the integral time) from a control parameter adjustment block that contains their analytical dependencies on the silicon rods diameter d_p (it has been established as a simulation result):

$$k_p\left(d_p\right) = 0.4\left|8.6 - d_p\right|^{2.4} \tag{17}$$

$$T_i\left(d_p\right) = 65 \cdot 10^{-2} \cdot d_p - 5.1 \tag{18}$$

In this case, an increase in the gain factor k_p of the controller to improve the dynamic response of the system leads to a decrease in the control performance. The neuro-fuzzy system can be used in this case, since it allows to face the problem without any competing operating condition.

3.2 Adaptive control system with neuro-fuzzy inference in the controller parameter adjustment block

The adaptive ACS of the rods temperature was modified, using a neuro-fuzzy network in the parameters adjustment block, with the aim of improving the control quality under those conditions in which plant parameters instability and effects of various disturbances and noises prove to be relevant. A block diagram for the adaptive control system with neuro-fuzzy parameter adjustment block is represented in Figure 2.

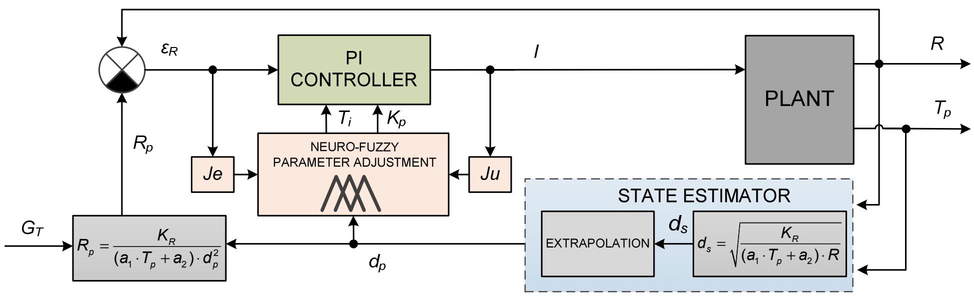

Figure 2. Control system with neuro-fuzzy inference in the controller parameter adjustment block

In this ACS the optimal values of the controller parameters is computed into the parameter adjustment block by minimizing the following functional:

$$\min\left(Je + Ju\right) = \min\left(\frac{100}{\overline{R}} \cdot \sqrt{\frac{\sum_{i=k}^{k+h-1}\left(\varepsilon_i^R\right)^2}{h}} + \frac{100}{\Delta I_{\max}} \cdot \sqrt{\frac{\sum_{i=k}^{k+h-1}\left(I_i - I_k\right)^2}{h}}\right) \tag{19}$$

with ε^R– rod resistance control error, I_i– manipulated variable (current), h –optimization interval, R – average rods resistance, ΔI_{max} – maximum permissible deviation of current, d_p – average rod diameter.

The parameter estimation block is based on an apparatus for neuro-fuzzy networks – Adaptive Neuro-Fuzzy Inference Systems (ANFIS) that uses linguistic rules and fuzzy reasoning (Jang, 1993). For the identification of the ANFIS system, the back-propagation method and combination of back-propagation and least square methods are used. Experimental data in the form of learning curves are used for the membership functions tuning. Using the expressions (17) and (18), the learning sample for the proportional gain $k_p(d)$ and the integral time $T_i(d)$ are evaluated. In order to increase the control precision, the gain factor was raised by the formation of various types of grid functions ($k_p(Je)$, $k_p(Ju)$, $T_i(Je)$ and $T_i(Ju)$) (linear, parabolic, exponential). It is found that the least values (minimum values/OPTIMAL) of *Je* and *Ju* are provided in correspondence with $k_p(Ju) = 1/\ k_p(d)$, $T_i(Ju) = T_i(d)$, $k_p(Je) = k_p(2d)$ and $T_i(Je) = 1/T_i(2d)$. The data obtained for training ANFIS are illustrated in Figures 3, 4.

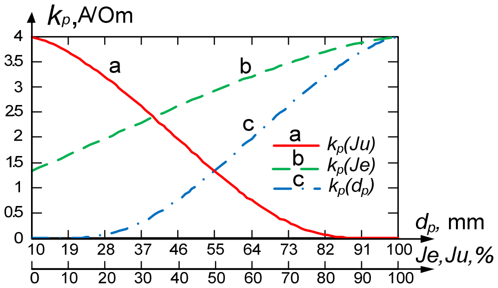

Figure 3. Obtained data for training ANFIS with respect to the gain k_p

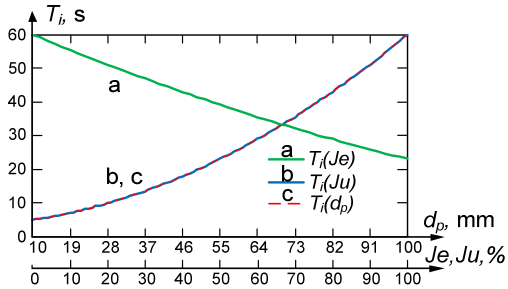

Figure 4. Obtained data for training ANFIS with respect to the integral time T_i

The fitting error of the basic data set is less than 0.1 % for 3 triangular-shaped membership functions used in this work. Increasing either the membership functions amount or samples amount in the learning sample by more than 100, and modifying the membership functions shape do not lead to any significant effect in the further reduction of the error value.

3.3 Adaptive control system with neuro-fuzzy controller

The ACS, which controls the temperature of the solid rods by means of a neuro-fuzzy regulator, is represented in Figure 5. The basic data for ANFIS are obtained by averaging experimental dependencies $I(d_p)$. In addition, the formation of the grid functions $I(d_p)$, $I(\varepsilon+\int d_p dt)$, $I(\int d_p dt)$, which differ from linear (parabolic, exponential) functions, do not guarantee improvements in the control performance and robustness. The proprotional gain k_p and the integral time T_i coming from the parameters adjustment block are derived from expressions (17) and (18).

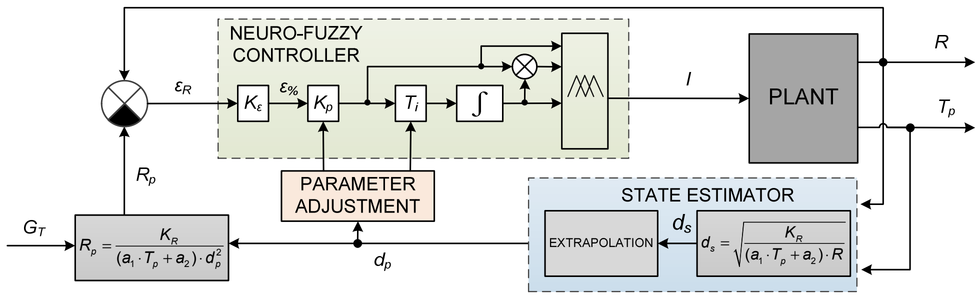

Figure 5. Control system with neuro-fuzzy controller

4. System responses analysis

The control quality of the designed ACS is enquired by comparing different control setups. All results are obtained with the same experimental trends for TCS and hydrogen flowrates, which are used as the input load variables for the simulation. The root mean-square deviation (RMSD) of the rods temperature during the campaign for all kinds of ACS is 1 grad. The most important fragment of the simulation results is shown in Figure 6, 7. In this case, for all ACS the maximum deviation from the set point was less the 15 grad. This maximum error is small since it is difficult to attain this result in the manual control mode.

Investigations on the robustness of the control schemes are performed via the analysis of the transient response when reactor parameters changes occur. The temporal trends for the main variables are computed when a simultaneous variation in the specific conductivity of the silicon rods by 0.7 times as well as the deposited polysilicon by 1.5 times occurs. In this case the control systems for the rod temperature provide almost the same control quality: the RMSD of the temperature are 1.3, 1 and 1.1 grad corresponding to 1st, 2nd and 3rd ACS.

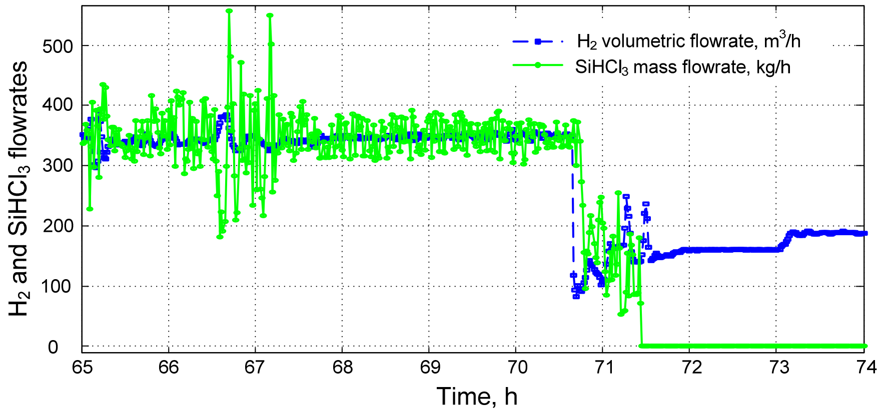

Figure 6. Inlet flowrates of reagents

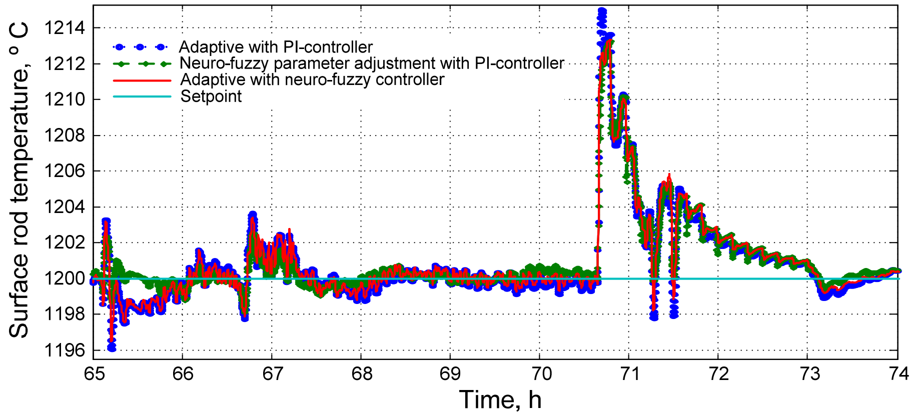

Figure 7. Surface rod temperature

5. Conclusions

A new adaptive ACS for polysilicon rod temperature in CVD reactors is designed with the engineering feasibility from a practical perspective. The novelty of this ACS consists of using an approach for the indirect estimation of the rods diameter and considering the rods resistance as controlled variable. Some advantages and improvements in the control quality is obtained by applying neuro-fuzzy approaches into the parameter adjustment block of the adaptive ACS. This may be explained by the computation of better optimal values for the PI controller parameters combined with an increased robustness. The basic concepts upon which the proposed control scheme is based are general and can be easily applied to other types of reactor.

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