

# Optimization of the Combined Modification Process of Thermo-Mechanical Densification and Heat Treatment on Chinese Fir Wood

Tao Li, Jia-bin Cai, and Ding-guo Zhou\*

The interactive effect of the thermo-mechanical densification and heat treatment on the set-recovery of modified wood were investigated for optimizing the combined modification process in this study. Process parameters such as thermo-mechanical densification temperature, duration of densification, and heat treatment temperature were selected as main factors. Three levels of each of these factors were chosen, and then nine experiments plus one verification experiment were conducted according to the principles of the Taguchi DoE method and the results of ANOVA analysis. It was observed that the dimensional stability of combined modified Chinese fir wood in the compression direction can be effectively improved by elevating the heat treatment temperature and densification temperature, the percentage contributions of which were 76.04% and 21.18%, respectively. Meanwhile, the set-recovery had no dependence on the duration of the densification process. The value of the set-recovery in the verification experiment agreed quite well with the predictions. From an economic view, the optimal condition for the combination modification of Chinese fir wood was that of a densification temperature of 170 °C, densification duration of 10 min, and heat treatment temperature of 200 °C.

*Keywords:* Chinese fir wood; Thermo-mechanical densification; Heat treatment; Set-recovery; Taguchi design of experiments; ANOVA

*Contact information:* College of Materials Science and Engineering, Nanjing Forestry University, Nanjing, 210037, P.R. China; \*Corresponding author: dgzhou@njfu.edu.cn

## INTRODUCTION

Wood has been used for building and construction material for thousands of years, mostly because of its low cost, renewability, strength, and low processing energy requirements (Hill 2006). To satisfy the increasing demand for raw forest product materials, Chinese fir (*Cunninghamia lanceolata*), one of the most widely planted softwood species in the south of China, has received increasing attention over the past decades. However, because of its relatively low density and poor mechanical properties, Chinese fir wood has been mainly utilized in the wood-based panel and packaging industry in China (Peng *et al.* 2006). With the growth of society's environmental awareness and the increasing availability and prices of tropical hardwood species that have good and constant properties, there is a pressing need to upgrade fast-grown and nondurable wood species into dimensionally stable and durable wood products, such as solid wood flooring, by means of wood modifications that have low environmental impact.

The thermo-mechanical densification process applies heat and compression to wood in the direction perpendicular to the grain and has been in use since around World

War II (Seborg *et al.* 1945). This process has been developed to improve the density and the strength properties of “soft” wood for high value-added solid wood products. However, as a result of regaining moisture from humid air or rainwater in specific use conditions, the final products of densified wood, without any fixation after-treatment process, will have an inevitable tendency to completely or partially recover from the compression set, a quality known as set-recovery (Rautkari *et al.* 2010).

Wood modification by heat treatment (with the use of steam, nitrogen gas, vegetable oil, or other heat transfer mediums) under high temperature conditions ranging from 140 to 240 °C, was first reported by Stamm and Hansen (1937). This treatment, following comprehensive study and successful commercial utilization in the wood industry since the end of the 20th century (Hill 2006), has been accepted as a qualified method for permanent fixation of the compression deformations of some forest products. Two main mechanisms of fixing the compressive deformation by heat treatment have been introduced: Due to the degradation of hemicellulose, the densified wood can be protected from being resoftened by reducing the hygroscopicity of the wood cell walls, making them inaccessible to water; and the inner stresses and elastic strains stored in the wood during compression can be released at an elevated temperature, which can cause a sufficient flow of the cementing lignin (Mendes *et al.* 2013; Kutnar and Kamke 2012; Pan *et al.* 2010; Del Menezzi *et al.* 2009; Welzbacher *et al.* 2008).

Although some of the above-mentioned articles report the effects of various factors on the properties of combined modified wood or wood-based panels, the interactive effect of densification and heat treatment on the set-recovery phenomenon, in which the process parameters are at different levels, has been rarely reported (Gong *et al.* 2010). With the advantages of keeping the cost of research activity at a minimum level, minimizing the difference between the target value and the predicted value, and its universal applicability to engineering fields, the Taguchi design of experiments (DoE) method with orthogonal arrays has been utilized in research concerning materials processing and manufacturing all over the world (Mohan and Reddy 2013; Abdullah *et al.* 2012; Torkaman *et al.* 2010). The main objective of this study was to optimize the combined process of thermo-mechanical densification and heat treatment (hereinafter refers to as “combined modification process”). The Taguchi DoE method was implemented to evaluate the effect of the process parameters on the set-recovery of modified Chinese fir wood based on the anisotropic characteristics and high variability of wood properties.

## EXPERIMENTAL

### Design of Taguchi Experiments

According to the principles of the Taguchi DoE method, thermo-mechanical densification temperature (densification temperature in short), duration of thermo-mechanical densification (densification duration), and heat treatment temperature were selected as the main factors of the combined modification process based on considerations and conclusions coming from previous experiments and pertinent literature (Cai *et al.* 2013; Kutnar and Kamke 2012; Gong *et al.* 2010). Meanwhile, a factorial design, specifically a three-level factorial design, was chosen. The experimental design for the above-mentioned process parameters and their three presented levels listed in Table 1, using an  $L_9(3^4)$  orthogonal array, is shown in Table 2. The error term in the

third column of Table 2 was used for the experimental error in the nine separate experiments in total.

**Table 1.** Factors and Levels of the Taguchi DoE

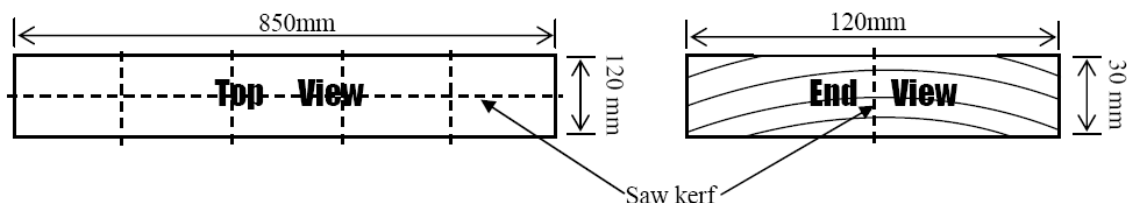
Factors	Labels and unit of factors	Levels		
		Low(L)	Middle(M)	High(H)
Densification temperature	A (°C)	140	155	170
Densification duration	B (min)	10	20	30
Heat treatment temperature	C (°C)	170	185	200

**Table 2.** Results of the Set-recovery of Combined Modified Chinese Fir Wood in the Taguchi DoE

Experiment Number	Factors				Set-recovery (%)
	A (°C)	B (min)	Error	C (°C)	
1	L (140)	L (10)	L	L (170)	24.01
2	L (140)	M (20)	M	M (185)	18.76
3	L (140)	H (30)	H	H (200)	11.89
4	M (155)	L (10)	M	H (200)	10.92
5	M (155)	M (20)	H	L (170)	20.44
6	M (155)	H (30)	L	M (185)	17.75
7	H (170)	L (10)	H	M (185)	13.55
8	H (170)	M (20)	L	H (200)	8.88
9	H (170)	H (30)	M	L (170)	16.72

## Material

Kiln dried flat-sawn lumber of Chinese fir wood, measuring 35 mm by 130 mm by 2000 mm (radial × tangential × longitudinal direction), was purchased from the market for use in this study. The density of Chinese fir wood ranged from 0.29 to 0.33 g/cm<sup>3</sup> at 12% moisture content. Upon arrival at the laboratory, the lumber was carefully selected and machined to obtain six clear wood boards with dimensions of 30×120×850 mm<sup>3</sup>. Afterward, each wood board was cut and sawn into ten end- and edge-matched smaller boards (30×55×160 mm<sup>3</sup>), as shown in Fig. 1. Ten groups (6 wood samples in each group, 60 in total) were collected for the following experiments: 90% (nine groups) of the samples were employed at random in the L<sub>9</sub>(3<sup>4</sup>) orthogonal array testing, and the remaining 10% (six in total) were used for the verification experiment. Accordingly, different process parameters of the combined modification process could be ideally investigated on the same sample without regard to the variance of wood properties in the longitudinal direction.



**Fig. 1.** Method of sawing wood boards for preparation of samples

## Combined Thermo-Mechanical Densification and Heat Treatment Processes

To eliminate the risk of cracks in the wood during the densification process, wood samples were conditioned prior to the experiment in an environmental chamber at 25 °C and 65% relative humidity until they attained about 12% MC.

A hot press (see Fig. 2) with two plane platens (400×400 mm), in which the upper platen was stationary and the lower platen was moveable, was utilized to compress the wood. In the densification process, the velocity of the upward movement of the lower platen could be set and monitored by the enclosed linear displacement transducer with a software program on the microcomputer.

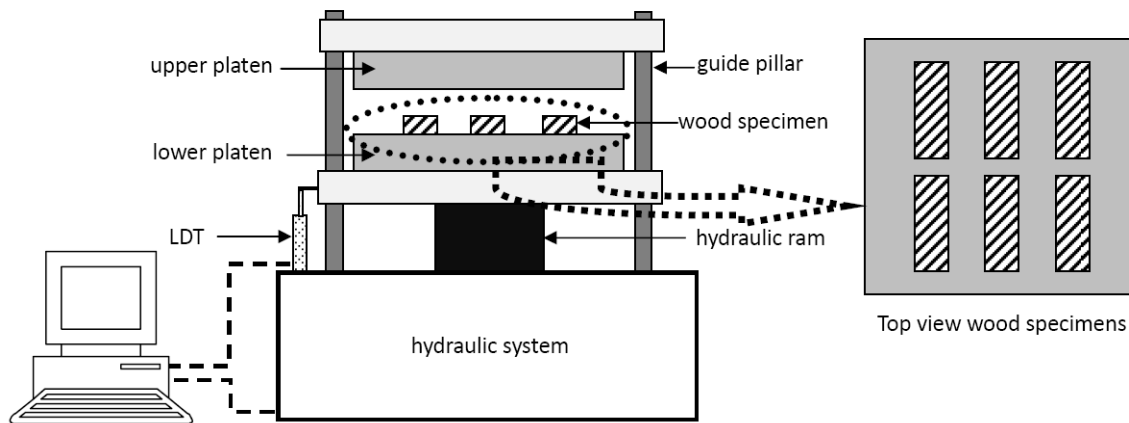


Fig. 2. Schematic diagram of the hot press used for thermo-mechanical densification

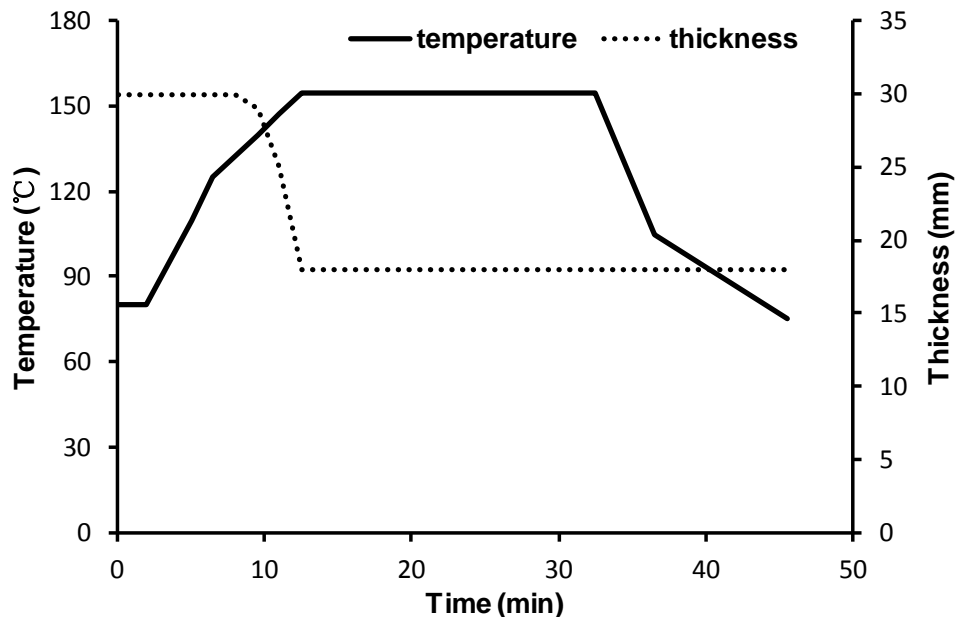


Fig. 3. Schedule for the densification process in experiment No. 5

When the two platens were warmed to 80 °C, six samples were taken to the closed press and preheated for 2 min under a negligible pressure. Then, the samples were

heated and compressed with a preset parameter program (the process parameters of heating rate and compression speed were fixed in the nine experiments) before the desired densification conditions were achieved. Upon the desired temperature (140 °C, 155 °C, or 170 °C) and 40% nominal compression set (fixed also, based on the initial thickness of the wood sample) being achieved, it was required that the densification condition be maintained for some time (10 min, 20 min, or 30 min) to equalize the temperature and internal stress in the wood. Finally, the samples were kept in the press until the temperature difference between the platens and ambient environment dropped to about 50 °C by the circulation of cool water in the inner pipes of the upper and lower platens. A pressing operation process in the No. 5 testing is illustrated in Fig. 3.

Subsequently, the densified wood was thermally modified using different temperature conditions (170 °C, 185 °C, or 200 °C) with superheated vapor in a small modified laboratory high-temperature drying kiln. The details of the heat treatment procedure are described in a previous study by the authors (Cai *et al.* 2013). The duration of each heat treatment experiment was kept constant at 1.5 h, which is the minimum time required to obtain the same color throughout the thickness of the wood samples, based on the results of preliminary heat treatment experiments.

### Determination of Set-recovery of Combined Modified Wood

After the heat treatment process, the modified Chinese fir wood was immediately trimmed and subdivided equally into three square specimens (20×50×50 mm<sup>3</sup>). Then, the specimens were successively kept in the oven at 105 °C and the environmental chamber at 25 °C, 95% RH (referring to the moist conditions in daily indoor environments) until they reached equilibrium with the surrounding environment, *i.e.*, the thickness of specimens remained virtually constant. A 1086 dial indicator attached to 820FG small comparator stands (Mahr Ltd., Germany) was used to measure the thickness of wood at the center of the specimen. The set-recovery (S) refers to the rate of change of the thickness of modified wood in this paper and can be calculated using Formula 1,

$$S = \frac{l_a - l_0}{l_0} \times 100\% \quad (1)$$

where  $l_0$  and  $l_a$  are the thicknesses in the radial direction of modified Chinese fir wood in oven-dried and environmental (25 °C, 95% RH) conditions, respectively.

## RESULTS AND DISCUSSION

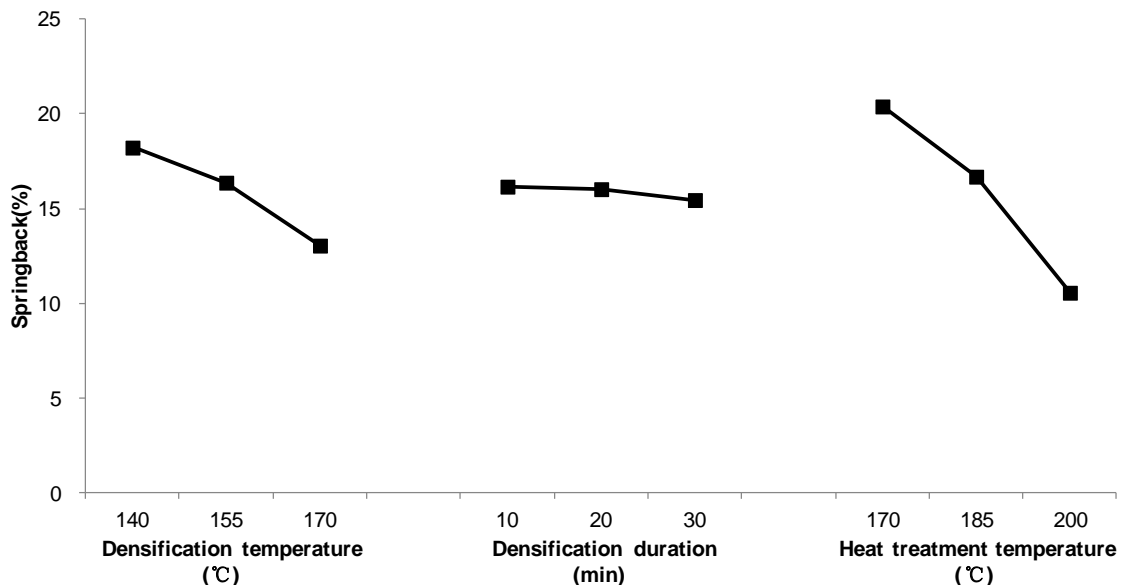
From Table 2, in which nine experimental conditions and the responses to be studied in the Taguchi DoE are summarized, the set-recovery of modified Chinese fir wood was in the range of 8.88% (the average value of 18 specimens) for No. 8 testing (densification temperature was 170 °C, densification duration was 20 min, and heat treatment temperature was 200 °C) to 24.01% for No. 1 testing (140 °C, 10 min, and 170 °C). Because the value of the maximum set-recovery was approximately 1.7 times higher than that of the minimum set-recovery, it is necessary to optimize the combined modification process to increase the dimensional stability of final products in the compression direction. However, the optimal process parameters of the combined modif-

ication process cannot be determined only based on the observation of these outcomes. It was therefore necessary to evaluate individually the importance of these three factors, densification temperature (A), densification duration (B), and heat treatment temperature (C), on the responses.

**Table 3.** Results of the Range Analysis

	Factors			
	A	B	Error	C
$k_L$ (%)	18.22	16.16	16.88	20.39
$k_M$ (%)	16.37	16.03	15.47	16.69
$k_H$ (%)	13.05	15.45	15.29	10.56
$R$ (%)	5.17	0.71	1.59	9.83

**Note:**  $k_i^j$  = Mean value of the set-recovery of modified Chinese fir wood for factor  $j$  (A or B or C) at the  $i$  level (low or middle or high);  $R^j = \text{Max}\{k_i^j\} - \text{Min}\{k_i^j\}$ .



**Fig. 4.** Effects of densification temperature, densification duration, and heat treatment temperature on the set-recovery of modified Chinese fir wood

First, the experimental data in Table 2 were examined using a range analysis method, and the results of which are illustrated in Table 3. Numbers 1, 2, and 3 experiments were the tests for which the densification temperature was 140 °C (the low level). The mean value of the responses of these experiments ( $k_L^A=18.22$ ), given in Table 3, is the mean of the low level in column A. Thus, the experiments for the second data point ( $k_M^A=16.37$ ) were the experimental conditions of column A at the middle level, and so on. The average effects of each factor at different levels on the responses are plotted below in Fig. 4. Using factor A as an example, the range ( $R$ ) of one factor is equal to the difference between the maximum and minimum values of  $k^A$  ( $R^A = k_{\max}^A - k_{\min}^A$ ). The larger the range of the factor, the more significant is its influence. As seen from Table 3 and Fig. 4, these factors can be ranked in order of their significance on the responses as follows:  $C > A > B$ . That is, the heat treatment temperature is the most important process

parameter ( $R^C = 9.82$ ) affecting the set-recovery of modified Chinese fir wood, followed by the densification temperature ( $R^A = 5.17$ ). However, because the range of factor B was only 0.71, the set-recovery appears to have had no dependence on the duration of the densification process, as indicated by the fact that the regression line of densification duration in Fig. 4 is nearly horizontal.

As can be seen from some previous studies on process optimization (Mohan and Reddy 2013; Abdullah *et al.* 2012; Torkaman *et al.* 2010), analysis of variance (ANOVA) combined with an  $F$ -test is a useful analysis tool to determine the percentage contributions of various factors to the response and to identify the optimal conditions in the Taguchi DoE. The influences of the three process parameters on the set-recovery of modified Chinese fir wood were therefore statistically analyzed with the aid of ANOVA by SAS statistical analysis software (SAS Institute, USA). The total degrees of freedom were evenly distributed in each factor (with three levels), with eight error terms for an orthogonal array with nine experiments. The sum of squares term, tabulated in the third column of Table 4, is an index of the relative importance of each factor in changing the response value. Accordingly, the mean square for a factor is calculated by dividing the sum of squares by the degrees of freedom. The symbol  $F$ , which refers to the ratio of the mean square of the factor divided by that of the error, is a crucial criterion for distinguishing the important factors from those with less significance. If the  $F$  value of a factor is larger than 9, with a level of significance of 0.1, due to the variability of wood properties, the factor then has a significant influence on the response. At the same time, the percentage contributions of the three process parameters to the set-recovery, with the addition of that of the error term, were computed individually by dividing the sum of squares of each term by the total sum of squares. The results of the ANOVA are shown in Table 4 and Fig. 5.

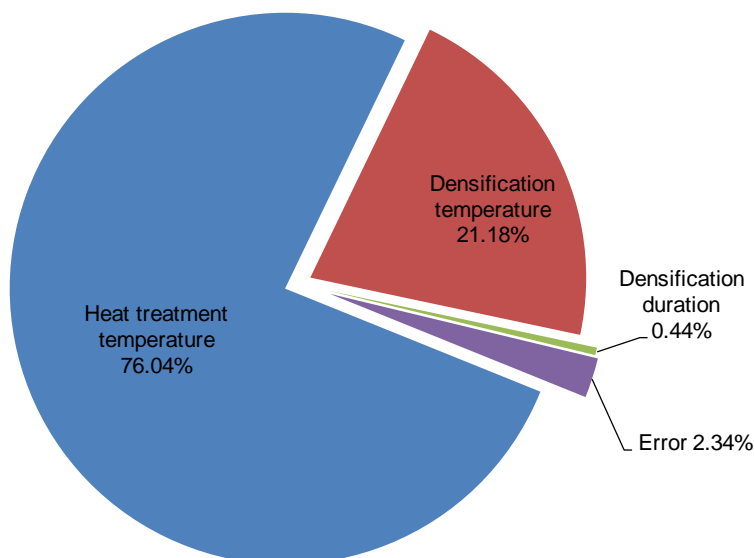
**Table 4.** Results of the ANOVA

Factors	Degrees of freedom	Sum of squares	Mean square	$F^*$
C	2	147.77	73.89	32.51
A	2	41.17	20.59	9.06
B	2	0.85	0.42	0.19
Error	2	4.54	2.27	
Total	8	194.33		

\*Significant parameter:  $F_{0.1}(2, 2) = 9$

It can be observed from the  $F$  values of various factors in Table 4 that heat treatment temperature, with a 76.04 percent contribution in Fig. 5, was the most dominant parameter for decreasing the set-recovery in the combined modification process. The next contributing factor was A, *i.e.*, densification temperature, the  $F$  value and percent contribution of which were 9.06 and 21.18%, respectively. Both of these factors have significant influences on the responses. At the same time, the  $F$  value of factor B was only 0.19, the result of which indicates that densification duration had a minor effect on the set-recovery of modified Chinese fir wood, in accordance with the range analysis results. The inefficiency of densification duration suggests that, with respect to decreasing the set-recovery of modified Chinese fir wood, there is no need to prolong the densification time in the combined modification process, under the consideration of the sufficient time and the elevated temperatures in the heat treatment procedure. Similar

results have also been reported by Welzbacher *et al.* (2008), in which vegetable oil was used as the heat transfer medium in the heat treatment.



**Fig. 5.** Percent contribution of various factors to the set-recovery of modified Chinese fir wood

In the last row of the ANOVA table, the error term contains information about three sources of the variability in the results: uncontrollable factors, factors that are not considered in the experiments, and experimental error. As a general rule of thumb, if the percent contribution of the error term is less than 50%, this is a good experiment (Roy 2001). In this study, the percentage contribution of error (2.34%) has been found to be very low; these results indicate that the proposed ANOVA analysis can be effectively used to determine the relative significance of the effect of each factor on the set-recovery.

In the Taguchi method, the experiment corresponding to the optimum working conditions might not have been conducted during the entire period of the experimental stage (Roy 2001). It is obvious from Fig. 4 that the set-recovery of modified Chinese fir wood should be minimized at the high level of densification temperature, densification duration, and heat treatment temperature. Accordingly, a verification experiment in which these three factors are all at optimum levels was performed, and the set-recovery of the new modified Chinese fir wood specimens was measured to be 8.45%, slightly lower than the 8.88% obtained in the No. 8 testing above.

## CONCLUSIONS

1. The set-recovery is significantly influenced by heat treatment temperature and densification temperature. An increase in these values can effectively cause a decrease in the set-recovery of modified Chinese fir wood. Meanwhile, the densification duration has a minor effect.
2. A verification experiment was conducted with optimum working conditions, and the result (8.45%) corresponded quite well with the predictions based on the above discussion about the densification duration with ANOVA analysis at 90% confidence level.



3. The cost-effective management of the combined modification process, under the consideration of the inefficiency of densification duration on the set-recovery, can be determined as follows: densification temperature of 170 °C, densification duration of 10 min, and heat treatment temperature of 200 °C.

## ACKNOWLEDGMENTS

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