Experimental research of the regularities of iron-bearing ore desulfurization for the temperature conditions of liquid phase sintering

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The results of experimental analysis of the regularities of chemical and metallurgical process of iron-bearing ore desulfurization for different conditions of liquid phase sintering are presented. This process is conducted with addition of catalytic impurities and variation of chemical composition of the heat-carrying gas, in order to reveal the optimal procedure of chemical and metallurgical process of ore smelting, which provide maximal possible removal of impurities from ore composition. It was found out for iron-bearing ore raw material that the rate and temperature conditions of impurities sintering depend on chemical composition of additives and physical state of initial material. The data about the chemical and metallurgical process of iron-bearing ore desulfurization, depending on ore coarseness and addition of solid fuel at low temperature during smelting process, are obtained. It was also established that lowering of the starting temperature for impurities decomposition and acceleration of desulfurization in the area of charge heating can be achieved via charge fluxing by fluorine-containing limestone and decrease of oxygen content in a gaseous phase. Essential deviations from solid phase procedures in the mechanism of sulphate decomposition at the temperatures of liquid phase sintering are revealed. Analysis of the experimental data allowed to establish necessity of introduction of the dual-stage route in the technological process of iron-bearing ore desulfurization; this route includes solid phase and liquid phase removal of sulfuric impurities, while liquid phase removal should be implemented with high oxidation degree of the slag phase.

Key words: iron-bearing ore, ore desulfurization, sintered charge, temperature, iron monoxide, heat exchange, thermogram, heat-carrying gas.

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Introduction

Up-to-date state of the industry requires fabrication of high-quality steel products by the metallurgical enterprises. It is mentioned at the same time that most part of iron ore deposits, which were started to operate during the Soviet period, are characterized by decrease of Fe content and increase of part of silica and sulfur; it is especially noted that these ores contain significant amount of barite sulfur, which can be hardly removed during sintering [1, 2]. E.g., Fe content in the iron ores of Atasuy group decreased by 1.55–6.82 %, while content of impurities increased. Rise of silica content in charge increased amount of limestone and dolomite for its fluxing, what led to additional lowering of desulfurization degree and increase of sulfur content in agglomerate (in addition to increase of sulfate sulfur content) [3]. It is necessary to provide essential improvement of beneficiation technology for iron ore raw material in order to remove maximal amount of impurities, which deteriorate physical and chemical properties of future steel. Decrease of amount of impurities of sulfur, calcium and barium sulfates allows to make steel more resistant to the effect of oxidation processes [4, 5]. The processes of impurities removal from iron-bearing ore were examined by such national scientists as F. M. Bazanov, I. I. Rovenskiy, N. N. Berezhnoy and foreign specialists O. Lellep, A. Vinius, F. Akema et al. [6, 7].

The developed techniques consider removal of impurities at the stages of solid phase ore beneficiation and suggest that their removal at the stage of liquid phase sintering is practically useless [8]. In the meantime, examination of kinetics of the liquid phase stage of sulphate dissociation allows to develop an up-to-date technique for smelting of iron-bearing ores, which provides correspondence between domestic steel and its international analogues (and even advantage of domestic steel). It can help domestic metal products to be more competitive in the global market. Consideration of the key parameters and features of the liquid phase procedures of sulfate desulfurization determines importance and novelty of the presented research. The conducted investigations allow to develop mathematical and technological model of dual-stage removal of impurities at the stages of solid phase and liquid phase iron-bearing ore sintering.

The aim of this work is analysis of influence of the properties of fractions and impurity in iron-bearing ore on chemical and metallurgical process of its desulfurization for the temperature conditions of liquid phase sintering with addition of heat-carrying gas having various chemical composition, as well as determination of possible directions of improvement of this technological process.

Methods of research: experimental investigations and analysis of regularities of desulfurization of different fractions

of iron-bearing ore, using catalytic impurities and heat-carrying gases for the temperature conditions of liquid phase sintering.

Experimental research of desulfurization of different fractions of iron-bearing ore with different fractions during liquid phase sintering

Two series of experiments were carried out in the process of experimental investigation. In the first series, fuel was not added to charge. The temperature above sintered layers was held as 1300 °C for all experiments. At the same time, the temperature inside the layer reached 1150 °C to the end of sintering process. Experiments were conducted both with non-fluxed charge and with charge fluxed by usual and fluorizated limestone in the amount providing obtaining of basicity $CaO/SiO_2 = 1.2$. During the sintering process, ore fractions (0–0,5 mm), (3–5 mm) and (8–10 mm) was used. In the experiments with small fraction, charge was briquetted under pressure $1.96 \cdot 10^7$ Pa with obtaining porosity of samples 29 %. Sintered charge was blown during sintering by gas, which contains 0–8 % (vol.) of oxygen. The obtained results are displayed in the **Table 1**.

In the second series, solid fuel (coke) was added to charge in the amount 5.0 and 7.5 % (mass.). The temperature above the layer was held as 1300 °C and 1340 °C. The temperature in the layer was varied mainly depending on degree of charge fusion penetration. Continuous recording of the temperature in the layer by potentiometer allowed to determine the maximal temperature and to calculate the average stationary one. Ore fractions (0–0.5 mm) and (3–5 mm) were subjected to sintering. In experiments with more fine fraction, charge was briquetted with fuel having coarseness (0–0.5 mm), under pressure $1.96 \cdot 10^7$ Pa with obtaining porosity of samples 32 %.

To provide fuel burning, charge was blown upwards either by air, or by gas, which contains 5 % (vol.) of oxygen. Analysis of gases, emitting from the layer, showed that in the case of air blowing, they contain 10–12 % (vol.) CO_2 and 6–9 % (vol.) O_2, while in the case of use of weakly oxidized blowing, carbon dioxide content increased up to 20–30 % (vol.) and oxygen content decreased down to 1.0–2.0 % (vol.).

Analysis of the data obtained in the first experimental series with three fractions allowed to reveal influence of coarseness of sintered ore on desulfurization at relatively low temperature (1150 °C). Corresponding relationships (curves 1–3) for non-fluxed and fluxed charges in the atmosphere containing 8 % (vol.) O_2 are presented in the **Fig. 1**.

When fluxing both by usual and fluorizated limestone, no essential difference in desulfurization process was revealed; however, desulfurization degree was higher in the case of adding fluorizated limestone. At the same time, homogenous lowering of desulfurization degree was found out with increase of ore coarseness. Significant desulfurization (about 95 % (mass.)) was achieved for the fine fraction, and sulfur content decreased down to 0.1 % (mass.). Increase of coarseness to 8–10 mm led to lowering of desulfurization degree by 15 % (mass.) and rise of sulfur content in agglomerate to 0.2 % (mass.). Temperature elevation in the layer to 1300 °C owing to fuel burning provided practically complete desulfurization of fluxed charges. There is no difference noted for fine and coarse fractions.

Substantial differences were revealed for non-fluxed ore. Desulfurization of briquettes from fine fraction at the temperature 1150 °C was stopped for this kind of raw material, and achieved desulfurization degree does not exceed 50 % (mass.). Increase of ore coarseness to 3–5 and 8–10 mm elevates harshly desulfurization rate, while the value of desulfurization degree hardly differs from this value for fluxed charges.

Dependence between desulfurization degree of iron-bearing ore and average stationary temperature in the layer is displayed in the **Fig. 2**.

At the same time, the data for the temperature 1150 °C were obtained during experiments without fuel, while other

Table 1. Chemical composition of ore sinter after roasting in the atmosphere (first series of experiment)

No.	Charge composition, %			Ore coarseness, mm	Oxygen content, %	Results of chemical analysis			Desulfurization degree, % (mass.)
	Ore	Limestone	Fluorizated limestone			S, % (mass.)	FeO, % (mass.)	Fe, % (mass.)	
1	100	–	–	0–0,5	8	0.98	33.5	48.7	53.3
2	100	–	–	0–0,5	0	1.2	23.4	48.8	42.9
3	80	20	–	0–0,5	8	0.12	20.3	39.0	94.3
4	80	20	–	0–0,5	0	0.31	25.5	37.9	85.2
5	75	–	25	0–0,5	8	0.09	20.1	35.1	95.7
6	75	–	25	0–0,5	0	0.25	23.2	29.1	88.1
7	100	–	–	3–5	8	0.14	26.7	53.3	90.0
8	100	–	–	3–5	0	0.25	27.3	52.4	82.1
9	80	20	–	3–5	8	0.27	22.6	46.6	80.7
10	80	20	–	3–5	0	0.23	26.2	46.0	83.6
11	75	–	25	3–5	8	0.23	24.0	50.5	83.6
12	75	–	25	3–5	0	0.23	22.9	51.7	83.6
13	100	–	–	8–10	8	0.24	16.3	50.3	70.0
14	100	–	–	8–10	0	0.52	29.4	53.9	35.0
15	80	20	–	8–10	8	0.21	20.6	51.7	73.8
16	80	20	–	8–10	0	0.51	21.6	57.5	36.3
17	75	–	25	8–10	8	0.19	23.2	50.3	76.3
18	75	–	25	8–10	0	0.12	23.3	56.6	85.0
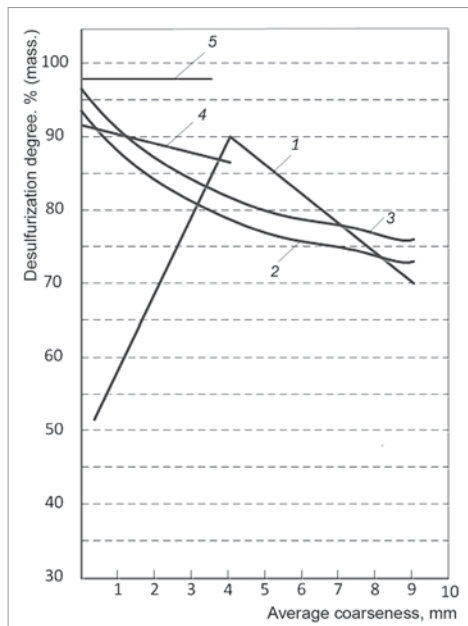
Fig. 1. Relationship between desulfurization degree of iron-bearing ore and its coarseness: curves *1, 4* — non-fluxed ore; curves *2, 5* — with adding usual limestone; curve *3* — with adding fluorizated limestone; temperature in the layer is 1150 °C for the curves *1–3* and 1300 °C for the curves *4* and *5*
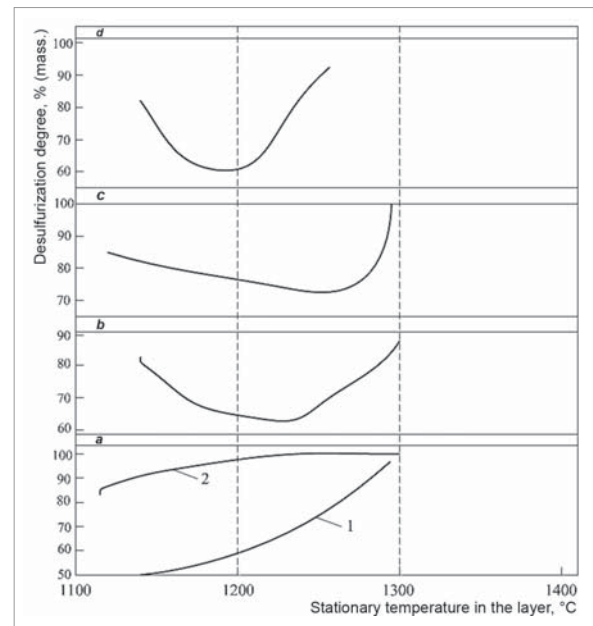

Fig. 2. Influence of sintering temperature on desulfurization degree of iron-bearing ore: *a* — fraction 1.5 mm: *1* — non-fluxed, *2* — fluxed; *b* — fraction 3—5 mm non-fluxed, *c* — fraction 3—5 mm fluxed by usual limestone, *d* — fraction 3—5 mm fluxed by fluorizited limestone

data — during experiments with fuel. As soon as essential difference in desulfurization when using oxidizing and weakly oxidizing atmospheres was not revealed, the average data were taken for building the graphs in the Fig. 2 for definite values of temperature, charge composition and coarseness. The experiments with FeO content exceeding the average level more than by 10 % (mass.), were not used for below-mentioned purposes.

It can be seen from the Fig. 2a that fine fluxed ore is characterized by relatively good desulfurization within the examined temperature range 1150—1350 °C. At the temperature from 1300 to 1350 °C, desulfurization degree is close to 100 % (mass.), while sulfur content in agglomerate lowers to 0.01—0.04 % (mass.). At 1150 °C desulphurization is worse — appr. 90—92 % (mass.), sulfur content is 0.1 % (mass.). The difference between usual and fluorizated limestone was not revealed.

Based on the experimental results, it is necessary to use high temperatures to provide successful desulfurization of fine non-fluxed ore. Even at the temperature 1340 °C desulfurization degree exceeds a little 90 % (mass.), while at the temperature 1150 °C this parameter makes only 50 % (mass.).

Relationships between desulfurization degree and temperature for coarse ore fraction are presented on the Fig. 2. These curves vary slightly after flux adding and are characterized by the minimum within the temperature range 1200—1250 °C. It should be taken into account that desulfurization degrees in unfavourable range vary very strongly from its values at the temperatures 1150 °C and 1250 °C. For example, residual sinter content in sinter, obtained with addition of usual limestone at 1250 °C, 1150 °C and 1280 °C are 0.68 % (mass.), 0.27 % (mass.) and 0.04 % (mass.) respectively.

Analysis of the experimental data allowed also to reveal absence of substantial influence of additive of fluorizated

limestone and lowering of oxygen content in gaseous phase on high-temperature desulfurization of agglomerated sinter. Several experiments displayed lowering of desulfurization degree when replacing oxidizing medium on non-oxidizing one.

When using fluorizated limestone at low temperatures without fuel addition, desulfurization degree is higher in comparison with usual limestone. In the case of higher temperatures, there are separate experiments with fluorizated limestone when desulfurization is practically stopped or is essentially slower than in experiments with usual limestone.

At low temperatures, weakly oxidizing medium and fluorizated limestone supported desulfurization process, as it can be seen from the works of other researchers [9, 10]. Thereby the obtained results at the stage of high-temperature sintering require special consideration. Comparative analysis of conducted experiments, when addition of fluorizated limestone and weakly-oxidizing medium led to deterioration of desulfurization in comparison with basic experiments with the same coarseness and temperature values, was carried out. It was established on the base of this analysis, that content of Fe monoxide is too high. This regularity is confirmed in all cases, when charge was completely molten. The relationship between sinter desulfurization degree and content of Fe monoxide is presented in the **Fig. 3**. The graphs in this figure were built based on the experiments, where fine fractions without fuel at the temperature 1150 °C, and practically all experiments with fuel (except 3—5 mm fraction at the temperature 1300 °C above the layer).

The last ones were not used, because melt amount in them was not sufficient for total dissolution of ore bulks.

The obtained regularity distinctly shows on connection between melt oxidizing ability and desulfurization. In this case, sinters with Fe monoxide content below 15 % (mass.) are desulfurized practically completely, and residual sulfur content
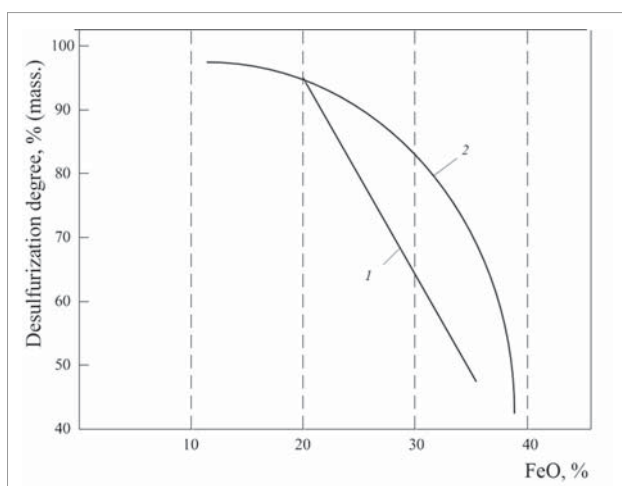
Fig. 3. Influence of FeO content in sinter on desulfurization degree: *1* – experiments without fuel addition at the temperature 1150 °C in the layer; *2* – experiments with fuel addition at the temperature 1200−1350 °C in the layer

in them exceeds 0.01−0.02 % (mass.). On the contrary, sinters with Fe monoxide content 35−40 % (mass.) preserve the most part of sulfur, and their desulfurization degree does not exceed 50 % (mass.). Taking this into account, it is clear that oxygen content in blow decreases degree of magnetite oxidizing and leads to rise of Fe monoxide content. Deterioration of desulfurization is probably connected with this appearance.

It also should be noted that use of blowing with decreased oxygen content (appr. 5−8 % (vol.)) in experiments with briquettes of fine ore with coke additives leads to better desulfurization in comparison with air blowing (both for usual and especially for fluorizated limestone). However, variation of Fe monoxide content is characterized by anomalous features for the above-mentioned cases. In weakly oxidizing medium, sinters are more oxidized and FeO amount in them is lower. It could occur only due to melting of briquette surface with low-melting additives, during intensive development of exothermic magnetite oxidation in air flow. Crust formation hinders further oxygen supply in a briquette and extraction of sulfuric gas. High monoxide properties and molten state of briquettes decrease desulfurization rate during air blowing [11, 12].

Additives of fluorizated limestone don't hinder oxidation of course. Thereby Fe monoxide content in the experiments without fuel is not higher in fluorizated limestone in comparison with usual limestone. Increase of FeO amount occurs only in presence of solid coke, and the temperature in the layer decreases in this case by 50−70 °C in comparison with non-fluxed charge or usual limestone. These facts testify about deterioration of fuel burning in presence of fluorine-containing melts. Fe oxide is reduced, FeO content increases and desulfurization deteriorates. More slow fuel burning and connected with it decrease of sintering machine productivity, when using calcium fluoride, are noted previously in researches of other specialists [13].

Probably fluorine melt moistures well coke pieces and prevents oxygen access to them. However, in the case when fuel ignition took place, fluorine-containing charge is characterized by practically complete desulfurization. Respectively, proper burning management in high-melting charge provides positive desulfurization effect via replacement of usual limestone by fluorine-containing limestone.

Thus, analysis of high-temperature desulfurization process of iron-bearing ores displayed that decrease of ore coarseness during sintering of fluxed agglomerate supports desulfurization. Fine non-fluxed charges, which are sintered at the temperature 1200 °C, are subjected to desulfurization worse that large charges. Desulfurization rate of large ore fractions within the temperature range 1200−1250 °C is significantly lower than within the temperature ranges 1150−1200 °C and 1280−1320 °C. There is direct correlation between desulfurization degree and Fe monoxide content in sinter during active melt forming. Maximal desulfurization is observed for FeO < 15 % (mass.). Lowering of oxygen content in gaseous phase does not improve high-temperature desulfurization. Addition of fluorine-containing compounds is useful for providing good fuel burning.

To understand relationship between sulfur content in agglomerates, which were previously in liquid phase, and Fe monoxide content, let us consider chemical mechanism of the processes of sulfur absorption and removal from iron-calcium-silicate slags contacting with gas. In particular, the research [14], which was devoted to the experiments with the melts of $CaO-SiO_2$, $CaO-Al_2O_3$, $MgO-SiO_2$, $FeO-SiO_2$, $MgO-Si_2O_3$, $CaO-SiO_2-Al_2O_3$ systems, established the areas of oxygen partial pressure, that are characterized by sulfur dissolution in sulfate or sulfide forms.

Sulfur ion coordinates four oxygen ions around itself and forms complex anion SO_4. Respectively, high activity of free oxygen anion is required for sustainable identification of sulfate ion in a melt. In binary systems with equimolar relationship between components this activity decreases in the row $CaO-Al_2O_3$, $CaO-SiO_2$, $MgO-SiO_2$. The same sequence is observed for sulfide sulfur on the base of examination of the system $FeO-SiO_2$. Sulfide capacity in this system is larger that in melts $CaO-Al_2O_3$ and $CaO-SiO_2$. That's why it is possible to suggest that sulfate capacity in FeO-containing melts will exceed sulfate capacity in calcium-silicate melts, because Fe monoxide is a well dissociating compound increasing concentration of free oxygen ions. It is evident that FeO replacement by Fe_2O_3 (or SiO_2) will lead to melt polymerization and, respectively, to lowering of oxygen concentration. Advantageous forming of sulfate sulfur can be described by the following reaction:

$$SO_2 + O^{2-} + \tfrac{1}{2}O_2 = SO_4^{2-} \qquad (1)$$
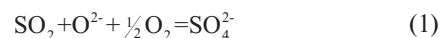
In this case, equilibrium of the reaction (1) will move to the left and sulfate ion will decompose with sulfur removal in gas. It also explains bas desulfurization in agglomerate melts, which contain large amounts of Fe monoxide (see Fig. 3). FeO replacement by Fe_2O_3 led to intensive desulfurization.

The examined decomposition mechanism for sulfates in a melt differs principally from the solid phase case, where oxygen partial pressure in gaseous phase and presence of Si, Fe, Al oxides are determining thermodynamic factors. As for sintering melts, which are located in weakly oxidizing medium, oxygen partial pressure will always exceed 10 Pa, thereby stability of sulfates will be mainly depended on slag

composition and presence of strong polymer forming cations S^{4+}, Fe^{3+}, P^{5+} and other in this slag.

For constant silica content in charge, correlation Fe^{3+} / Fe^{2+} is the main factor for adjusting residual sulfur content.

It can be noticed that desulfurization (even for favourable thermodynamic conditions for decomposition of a sulfate ion) can be hindered by diffusion of large SO_4 ions in viscous slags to a boundary "slag-gas", where reaction (1) occurs. Quick decrease of slags viscosity occurs during introduction of flux additives to acidic gangue of iron-bearing ore. However, the melting temperatures for the system $FeO–SiO_2$ were obtained at rather low level (1100–1170 °C) with small CaO additives. Introduction of large flux amount increases the melting temperature to 1205–1206 °C.

Conclusion

It was established during this experimental research that sulfur in ore can be removed in the sintering process both before ore smelting and after melt forming. Indeed, ore fractions with size 3–5 mm and 8–10 mm were not molten completely during the experiments without fuel, when the temperature did not exceed 1150 °C; however, despite this fact, the values of desulfurization degree in these cases reached 70–80 % (mass.). Melt forming in the experiments with fuel was characterized by desulfurization 90–97 % (mass.). But high-silica melt, which is forming at low temperatures (1150 °C) in non-fluxed briquettes has high viscosity for a fine fraction. Thus this melt, from one side, blocks solid ore particles and does not allow to remove gaseous reaction products. Moreover, sulfur, which transited to the melt, also can't be removed due to low rate of ion exchange in viscous fluid. It was found out that these factors hinder desulfurization from non-fluxed briquettes within the temperature range 1150–1200 °C. When sintering large fractions with size 3–5 mm and 8–10 mm, the melt is not forming practically and solid phase sulfur removal, which provides desulfurization degree 75–90 % (mass.) at the temperature 1150 °C becomes possible. Introduction of calcium oxide and calcium fluoride decreases viscosity of melts by several times and desulfurization degree from fine fraction is 85–90 % (mass.) at the temperature 1150 °C.

As soon as temperature in the layer is rising, viscosity decreases and non-fluxed briquettes from fine fraction show better desulfurization. Viscosity of calcium slags lowers less intensively with temperature rise (in comparison with silicon slags); it finalizes in slower elevation of desulfurization degree with temperature rise.

Experimental data displayed that temperature relationship of desulfurization degree in large ore fraction is principally different. At the temperature 1150 °C, when the melt practically does not exist, solid phase sulfur removal takes place. It provides desulfurization degree 80–85 % (mass.) during sintering, not depending on charge composition. Within the temperature range 1200–1250 °C, forming of viscous slags starts mainly on the base of ore gangue. These slags block ore pieces and hinder gas emission. Desulfurization from the melt becomes impossible due to high viscosity. Temperature rise up to 1300 °C leads to forming of free-running slag. Most part of ore in this case is dissolved and active desulfurization from the melt goes up to degree 90–95 % (mass.).

Therefore, the suggested desulfurization route during sintering of iron-bearing ore should include two stages: solid phase sulfur removal from large fractions (with size 3–10 mm) within the temperature range 1100–1200 °C and liquid phase sulfur removal – at 1250 °C.

It is required to provide high oxidizing degree of a slag phase for liquid phase desulfurization, and Fe monoxide content in agglomerate should not exceed 12–15 % (mass.). Additionally, the conducted experiments allowed to establish necessity of avoiding sintering within the temperature range 1200–1250 °C, because viscous slags, which are hindering both solid phase and liquid phase desulfurization, are forming within this temperature range. ⊡

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