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Synthesis of MgO powders from molten salts

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The reactions of anhydrous $MgSO_4$ and $MgCl_2$ in different molten nitrates and nitrites which produce fine MgO powders were studied. The stoichiometries of the reactions were established and the effects of various reaction conditions on the properties of the MgO powders produced were investigated. The addition of Lux–Flood bases to nitrate melts can lower the reaction temperatures and further lower the crystallite size of the MgO powders produced. The results showed that very fine MgO powders with high purity can be precipitated from molten nitrates and nitrites at temperatures below 600 °C, and these powders can be obtained with soft agglomeration if the powders precipitated are properly processed.

1. Introduction

It has been reported that molten salts can be used as an alternative low temperature route to prepare fine ceramic powders [1–6]. These studies were mainly concentrated on the preparation of zirconia powders by reactions of zirconium compounds in molten nitrate and nitrite melts, including the reactions of $Zr(SO_4)_2$ in the molten $LiNO_3$–KNO_3 eutectic [1], reactions of $ZrOCl_2$ in the molten $NaNO_3$–KNO_3 eutectic [2, 3], reactions of $Zr(SO_4)_2$ in different molten nitrates and nitrites [4, 5] and the acidic/basic effects on the reaction of $Zr(SO_4)_2$ in the $NaNO_3$–KNO_3 eutectic system [6]. The results have shown that the preparation of fine ceramic powders from molten nitrates and nitrites is a potential low temperature economical route.

However, studies on the preparation of other ceramic powders are few. For example, there has only been one research conducted by Shakir [7] on the reactions of magnesium compounds including sulfates and nitrides in the low melting $LiNO_3$–KNO_3 eutectic system. Although the results revealed that the reactions can lead to the precipitation of magnesia powders, only the stoichiometries of the reactions were discussed and the properties of the magnesia powders precipitated were not stated.

The present research is concerned with the reactions of $MgSO_4$ and $MgCl_2$ in different molten nitrates and nitrites leading to the preparation of magnesia powders, which can be used as a high-grade refractory material [8] and an excellent dopant to improve the properties of other ceramic materials, such as zirconia [9].

2. Experimental procedure

2.1. Materials

Anhydrous $MgCl_2$, $NaNO_3$, KNO_3, $NaNO_2$ and KNO_2 were kept in an oven between 150–180 °C for over 24 h to ensure that they were fully dried before experiments. Anhydrous $MgSO_4$ was prepared by heating $MgSO_4 \cdot H_2O$ gradually in an electromantle to 350 °C for 2–4 h until it was thoroughly dried. These chemicals were then kept in a desiccator.

The eutectic melts of $NaNO_3$–KNO_3 and $NaNO_2$–KNO_2 were prepared by melting the eutectic mixtures of $NaNO_3$–KNO_3 (50–50 mol %, m.p. 220 °C) and $NaNO_2$–KNO_2 (65–35 mol %, m.p. 220 °C) at 270 °C for one hour with occasional stirring. The prepared eutectics were also kept in a desiccator.

Other chemicals employed, such as Na_2O_2 and Na_2CO_3, were used directly from the bottles.

2.2. Procedure

Firstly, a STA-780 Series (thermal analyser, Stanton Redcroft) simultaneous thermogravimetric–differential thermal analyser TG–DTA with balance sensitivity of 0.01 mg was employed for the study of reactions. The experiments were performed as described previously [5]. However, because of the violence of the reaction (foaming and bubbling) and the relatively small space between the crucibles and the balance arm, the molten salts could sometimes creep over the top of the sample crucible and cause it to adhere to the balance even though only small quantities of samples ($<$ 15 mg) were used. Therefore, after completing the measurements of reactions of $MgSO_4$ with different nitrite and nitrate melts, the thermal analyses of the reactions were carried out using an old-fashioned Stanton thermobalance (Setaram G70) which uses a much bigger and deeper crucible with a much larger amount of sample in it. The sample crucible can be removed at higher temperatures when the salts are still molten. Thus, in this way, the above problem can be avoided. This balance can give the differential mass loss per 5 min increments of time versus temperature. In the range 25–600 °C, blank runs for both instruments and experiments in which nitrates and nitrites were heated alone showed negligible mass losses.

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The preparation of magnesia powders was performed in a Pyrex test tube in a vertical furnace. After the experiments, the extraction of the precipitated powders was conducted in a similar manner to that reported by Al-Raihani and co-workers [4].

The powders prepared were identified by a Philip PW-1050 X-ray diffractometer (XRD) and the crystallite size was estimated according to Scherrer's equation [10]. The morphology of the powders was observed by a high resolution Philips EM 301 (100 kV) transmission electron microscope (TEM). The TEM samples were prepared in the same way as described by Al-Raihani *et al.* [4]. Chemical analysis was performed with a Perkin Elmer 2380 atomic absorption spectrometer (AA).

3. Results and discussion

3.1. Reactions of magnesium compounds with different melts

In normal furnace runs, it was observed that anhydrous $MgSO_4$ reacted with the $NaNO_2$–KNO_2 eutectic melt starting at about 240°C which is just above its melting point (220 °C) with the evolution of brown nitrogen dioxide and the formation of a white powder. At 350 °C the solvent started to bubble slowly but this became more violent with increasing temperatures when the bubbles came nearly to the top of the Pyrex tube. The reaction was continued until above 450 °C.

The TGA graph (Fig. 1, curvel) indicated that there were two overlapping stages of mass loss ranging from 240 to 450 °C. The first took place from approximately 240 to 340 °C with nearly 28% mass loss of the total $MgSO_4$ added, and the second from 340 to 450 °C with about 34% of mass loss of the total $MgSO_4$ added. The overall mass loss per $MgSO_4$ was approximately 62.4 ± 0.3%, which is close to that predicted on the basis of the following reaction, 63.1%

$$MgSO_4 + 2NO_2^- \rightarrow MgO + SO_4^{2-} + NO_2 + NO \tag{1}$$

Anhydrous $MgSO_4$ did not react with molten nitrate melts until higher temperatures than that for the reaction in the $NaNO_2$–KNO_2 solvent. For example, $MgSO_4$ began to react with $NaNO_3$ at about 480 °C, approximately 170 °C higher than its melting point (307 °C), with evolution of brown nitrogen dioxide and formation of a white powder. Above 500 °C the melt started to bubble and at temperatures around 550 °C the bubbles came up to the top of the Pyrex tube. This process continued until 600 °C which was the end point of the normal furnace run chosen, because nitrates decompose at temperatures higher than 600 °C [11, 12]. After a certain time holding the temperature at 600 °C, no further evolution of brown nitrogen dioxide and bubbles could be seen, showing that the reaction was complete.

It can be seen from the change of curvature of the TGA graph (Fig. 1, curve 2) of the reaction of $MgSO_4$ with $NaNO_3$ that there were two overlapping stages of mass loss taking place before 600 °C. This demonstrated that the reaction was complicated and occurred in steps. When the temperature was above 600 °C, the continuing mass loss indicated that the reaction was not complete and decomposition of the melt [11, 12] took place at the same time. The overall mass loss per $MgSO_4$ to 600 °C was approximately 78.7 ± 0.5%, which was lower than the prediction of the following equation, 89.7%

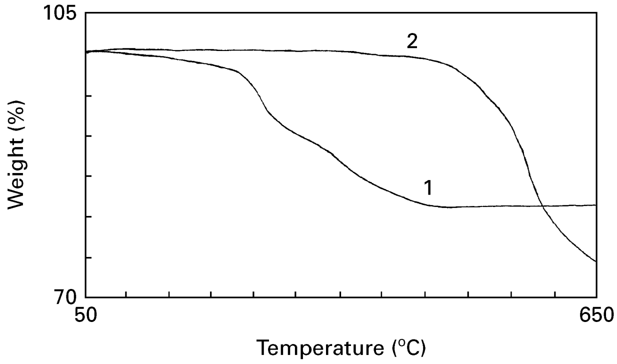

Figure 1 TGA graph of $MgSO_4$ with $NaNO_2$–KNO_2 (curve 1) and $NaNO_3$ (curve 2) (5 °C min^{-1}).

$$MgSO_4 + 2NO_3^- \rightarrow MgO + SO_4^{2-} + 2NO_2 + \frac{1}{2}O_2 \tag{2}$$

Considering that the reaction had not been completed at 600 °C, the experimental mass loss might be expected to be lower than the prediction. However, the relatively close experimental and predicted values seemed to demonstrate that most of the $MgSO_4$ had been reacted and that Equation 2 might reasonably represent the stoichiometry of the overall reaction.

The TGA plot of the reaction of anhydrous $MgSO_4$ with the $NaNO_3$–KNO_3 system indicated a similar trend. The differences were that the reaction started at about 450 °C, approximately 230 °C above the melting point of the $NaNO_3$–KNO_3 eutectic (220 °C) and 30 °C lower than the temperature of the reaction of $MgSO_4$ with the $NaNO_3$ single melt. Shakir and Kerridge's TGA results [7] showed that the reaction of $MgSO_4$ with the $LiNO_3$–KNO_3 melt began at about 440 °C, about 300 °C above its melting point (132 °C) and slightly lower than the temperature of the reaction in the $NaNO_3$–KNO_3 melt.

When using anhydrous $MgCl_2$ as starting material, it began to react with the $NaNO_3$–KNO_3 eutectic melt at about 400 °C with evolution of brown nitrogen dioxide and formation of a white powder. After 400 °C the solvent began to bubble slowly and the bubbles came nearly to the top of the test tube on further heating at about 500 °C. The TGA result (Fig. 2, curve 1) showed that the reaction mainly took place in the temperature range 400–600 °C and the maximum reaction rate was at about 500 °C. The reaction continued to about 600 °C. The overall mass loss per $MgCl_2$ at 600 °C, 109.7 ± 0.4%, was close to that predicted on the basis of the following reaction, 113.4%

$$MgCl_2 + 2NO_3^- \rightarrow MgO + 2Cl^- + 2NO_2 + \frac{1}{2}O_2 \tag{3}$$

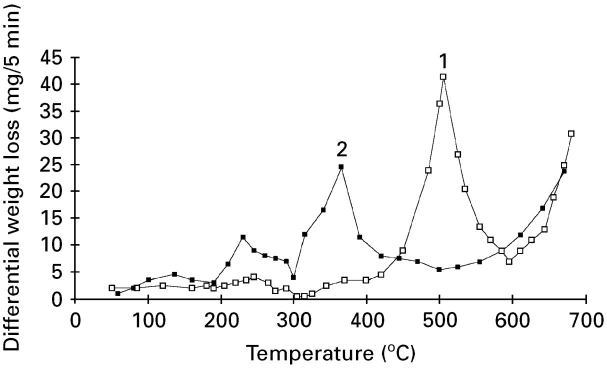

Figure 2 TGA graph of $MgCl_2$ with $NaNO_3$–KNO_3 (curve 1) and $NaNO_2$–KNO_2 (curve 2) (5 °C min^{-1}).

The reason that the experimental mass loss is lower than the prediction is probably that the reaction (Equation 3) was not absolutely complete at 600 °C. This can be seen from the TGA graph (Fig. 2, curve 1). With further heating, the melt began to decompose [11, 12].

However, the reaction of $MgCl_2$ in the $NaNO_2$–KNO_2 melt took place at a much lower temperature than in the $NaNO_3$–KNO_3 system, starting at about 200 °C, just below the melting point of the $NaNO_2$–KNO_2 eutectic (220 °C) and was complete at approximately 440 °C, i.e. the reaction was much faster. The TGA graph (Fig. 2, curve 2) indicated that there were two main overlapping stages of mass loss ranging from 200 to 440 °C. The first took place from about 200 to 300 °C, with a maximum reaction rate at approximately 230 °C and about 16% mass loss of the total $MgCl_2$, and the second from about 300 to 440 °C, with a maximum reaction rate at 350 °C and approximately 46% mass loss of the total $MgCl_2$ added. The overall mass loss per $MgCl_2$, 62.1 ± 0.3%, was very close to that predicted on the basis of the following reaction, 63.0%

$$MgCl_2 + 3NO_2^- \rightarrow MgO + 2Cl^- + NO_3^- + 2NO \tag{4}$$

The TGA graph of the reaction of $MgCl_2$ with the single $NaNO_3$ melt indicated a similar reaction to that in the $NaNO_3$–KNO_3 system, ranging from approximately 400 to 600 °C, with the maximum reaction rate at about 500 °C. The total mass loss, 108.5 ± 0.4%, was also close to that predicted on the basis of the reaction of Equation 3.

For comparison, the reaction results of $MgSO_4$ and $MgCl_2$ with different molten nitrate and nitrite systems are listed in Table I. It can be seen that when using the same starting material, either $MgSO_4$ or $MgCl_2$, the reactions tend to start at lower temperatures in the nitrate systems with lower melting points. The temperatures for complete reaction of $MgSO_4$ in nitrate melts were all over 600 °C, at which temperatures the melts would start to decompose [11, 12]. However, the temperatures for complete reactions were approximately 600 °C, if $MgCl_2$ was employed in nitrate melts. All the reactions in molten nitrates took place at much higher starting and finishing temperatures than in molten nitrites. On the other hand, comparing the reactions of $MgSO_4$ and $MgCl_2$ in the same melt, it was found that, when using $MgCl_2$ as the starting material, the reaction not only occurred at but was complete at lower temperatures as well.

From these results, it can be concluded that the employment of nitrites, which are more basic melts than nitrates, can lead to lower reaction temperatures. Therefore, it is of interest to study the effects of acidity/basicity on the reaction and preparation of magnesia powders.

3.2. The effects of acidity/basicity on the reactions

In order to allow satisfactory comparison, the $NaNO_3$–KNO_3 eutectic, a less basic melt than the $NaNO_2$–KNO_2 melt, was selected as solvent as well as reaction medium. The basicity of the $NaNO_3$–KNO_3 melt was increased by adding different kinds of Lux–Flood bases including Na_2O_2, Na_2CO_3 and $NaNO_2$–KNO_2. The acidic/basic influences on the reactions of $MgCl_2$ in the $NaNO_3$–KNO_3 eutectic melt containing different bases were measured by TGA and the results are shown in Fig. 3.

These showed that the reaction temperatures were greatly reduced by adding bases to the $NaNO_3$–KNO_3 melt. All the reactions in the melts containing bases started below the melting point (220 °C) of the $NaNO_3$–KNO_3 eutectic. The reaction temperatures were decreased by more than 200 °C by adding bases to the melt, since the reaction in the $NaNO_3$–KNO_3 eutectic with no base added started at about 400 °C.

TABLE I Reactions of $MgCl_2$ and $MgSO_4$ with molten nitrates and nitrites

Starting material	Melt	Melting point (°C)	Starting temperature (°C)	Peak temperature (°C)	Finishing temperature (°C)
$MgSO_4$	$LiNO_3$–KNO_3[a]	132	440	520	> 600
$MgSO_4$	$NaNO_3$–KNO_3	220	450	550	> 600
$MgSO_4$	$NaNO_3$	307	480	570	> 600
$MgSO_4$	$NaNO_2$–KNO_2	220	240	270 360	450
$MgCl_2$	$NaNO_3$–KNO_3	220	400	500	≈ 600
$MgCl_2$	$NaNO_3$	307	420	500	≈ 600
$MgCl_2$	$NaNO_2$–KNO_2	220	200	230 350	440

[a] From reference [7].

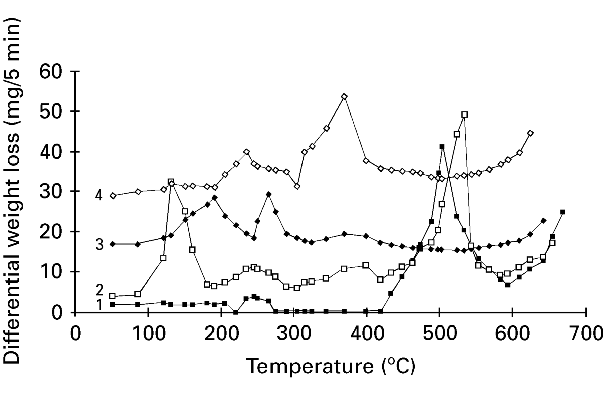

Figure 3 TGA graph of $MgCl_2$ in the $NaNO_3$–KNO_3 melt and the melt containing bases (5 °C min^{-1}). (■) $MgCl_2$ (10 mol %) + $NaNO_3$–KNO_3; (□) $MgCl_2$ (10 mol %) + $NaNO_3$–KNO_3 + Na_2CO_3 (30 mol %); (◆) $MgCl_2$ (10 mol %) + $NaNO_3$–KNO_3 + Na_2O_2 (30 mol %); (◇) $MgCl_2$ (10 mol %) + $NaNO_3$–KNO_3 + **$NaNO_2$–KNO_2** (30mol %).

The lower temperatures were probably due to the contribution of O^{2-} arising from the dissociation of Na_2O_2 and Na_2CO_3 in the melt [6], reacting with Mg^{2+} to precipitate MgO.

3.3. Synthesis of magnesia powders

On the basis of the TGA results on the reactions in molten nitrates and nitrites, the synthesis of magnesia powders was performed under various reaction conditions. The results, in which the powders were characterized by XRD, are listed in Table II.

It can be seen from Table II and the XRD patterns (Fig. 4) that the products were either MgO or a mixture of MgO and $Mg(OH)_2$ depending on the starting materials, solvents and reaction temperatures. When using $MgCl_2$ as the starting material in the $NaNO_3$–KNO_3 system, there was complete reaction to MgO at 600 °C (30 min), but incomplete reaction at temperatures below 600 °C. On the other hand, when employing $MgSO_4$ as the starting material, higher temperatures or longer reaction times were necessary than when using $MgCl_2$ as the starting material. For example, pure MgO was produced at approximately 550 °C, (90 min) in the $NaNO_2$–KNO_2 system, whereas, pure MgO was yielded at about 500 °C, (90 min), when employing $MgCl_2$. In addition, when reactions took place in the $NaNO_2$–KNO_2 melt, the reaction was complete at lower temperatures and pure MgO powders could be obtained at 550 °C (90 min) whatever starting materials including $MgCl_2$ and $MgSO_4$ were employed.

The results were in agreement with the TGA results, showing that the reactions occurred and were complete in the nitrites at lower temperatures than in the nitrates, and that they took place and were complete at lower temperatures when using $MgCl_2$ as the starting material than when employing $MgSO_4$ in a same melt under the same reaction conditions.

The production of $Mg(OH)_2$ was thought to be caused by incomplete reaction. Unreacted $MgCl_2$ or $MgSO_4$ left in the melt could react with water during the washing process forming $Mg(OH)_2$ in the presence of newly formed MgO powders.

Table II also lists the crystallite size of MgO prepared under different conditions. These were deduced from the XRD peak broadening using Scherrer's equation [10]. The crystallite sizes of MgO powders prepared were found to be in the range 20–40 nm. With increasing temperature in the range 450–600 °C the crystallite size increased. The trend is shown schematically in Fig. 5.

By comparing the ratios of the relative intensities of the XRD peaks of $Mg(OH)_2$ and MgO produced at different temperatures (holding time 90 min), it was found that the proportion of MgO produced in the mixture of MgO and $Mg(OH)_2$ increased with the increase in temperature, i.e. more $MgCl_2$ reacted with increasing temperature. After holding for 90 min at 450 °C, the ratio of MgO to total MgO and $Mg(OH)_2$ was about 50%. However, when the temperature reached 600 °C, 100% MgO was present. The results of the ratios calculated according to the following equation are shown schematically in Fig. 6

$$\text{MgO ratio} = \frac{I_{\mathrm{MgO}(2\theta = 43^\circ)}}{I_{\mathrm{MgO}(2\theta = 43^\circ)} + I_{\mathrm{Mg(OH)_2}(2\theta = 38^\circ)}} \quad (5)$$

Under the same conditions, such as 450 °C/90 min, the reaction of $MgCl_2$ in the $NaNO_2$–KNO_2 melt produced more MgO than it did in the $NaNO_3$–KNO_3 melt. This was also shown by the relative intensities of the XRD peaks. The reaction produced 100% MgO at relatively lower temperatures in nitrites than in nitrates. Similar results were also obtained when utilizing $MgSO_4$ as the starting material. This indicated that the reaction occurring in the $NaNO_2$–KNO_2

TABLE II Synthesis of MgO powders from nitrate and nitrite melts

Starting materials	Solvent	Temperature and time (°C, min)	Products (XRD)	Crystallite size (nm)
$MgCl_2$	$NaNO_3$–KNO_3	450, 90	MgO, $Mg(OH)_2$	26.6
$MgCl_2$	$NaNO_3$–KNO_3	500, 90	MgO, $Mg(OH)_2$	28.2
$MgCl_2$	$NaNO_3$–KNO_3	550, 90	MgO, $Mg(OH)_2$	30.9
$MgCl_2$	$NaNO_3$–KNO_3	600, 90	MgO only	34.9
$MgCl_2$	$NaNO_3$–KNO_3	600, 30	MgO only	32.3
$MgCl_2$	$NaNO_2$–KNO_2	450, 90	MgO, $Mg(OH)_2$	22.8
$MgCl_2$	$NaNO_2$–KNO_2	500, 90	MgO only	25.0
$MgSO_4$	$NaNO_3$–KNO_3	500, 90	MgO, $Mg(OH)_2$	28.6
$MgSO_4$	$NaNO_3$–KNO_3	600, 90	MgO only	39.5
$MgSO_4$	$NaNO_2$–KNO_2	550, 90	MgO only	20.5

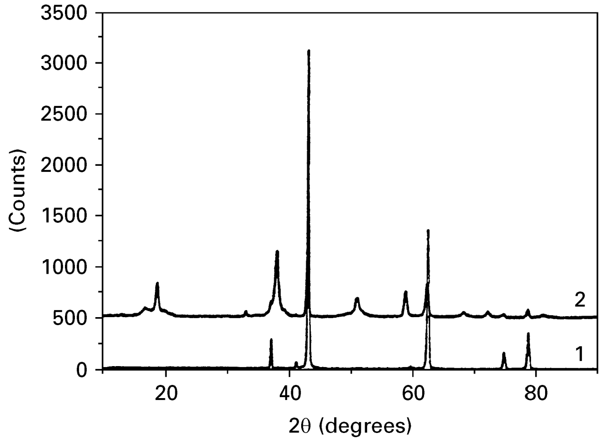

Figure 4 XRD patterns of the powders produced by reaction of $MgCl_2$ in $NaNO_3$–KNO_3 (1, MgO; 2, MgO + $Mg(OH)_2$).

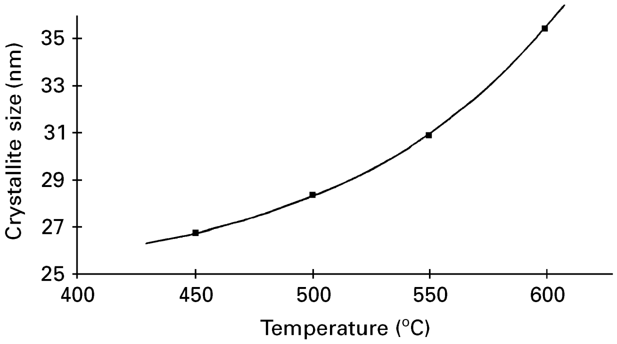

Figure 5 The relationship between crystallite size of MgO produced by $MgCl_2$ in $NaNO_3$–KNO_3 and reaction temperatures (holding time 90 min).

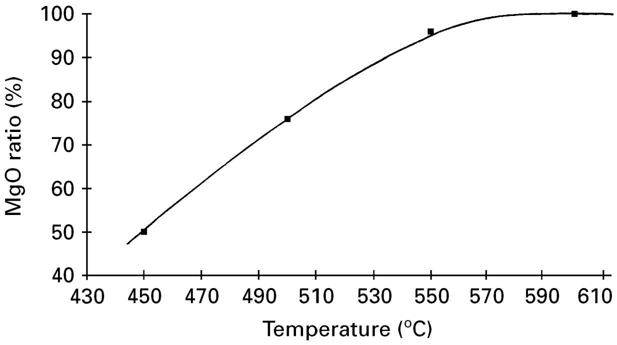

Figure 6 The relationship between the ratio of relative peak intensities of MgO/(MgO + $Mg(OH)_2$) and temperature (holding time 90 min).

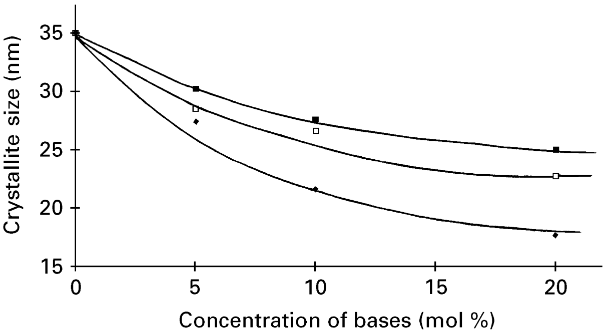

Figure 7 XRD patterns of MgO powders produced by adding Na_2O_2 to the $NaNO_3$–KNO_3 melt. (■) $MgCl_2$ + $NaNO_3$–KNO_3; (□) $MgCl_2$ + $NaNO_3$–KNO_3 + Na_2O_2 (5 mol %); (◆) $MgCl_2$ + $NaNO_3$–KNO_3 + Na_2O_2 (10 mol %).

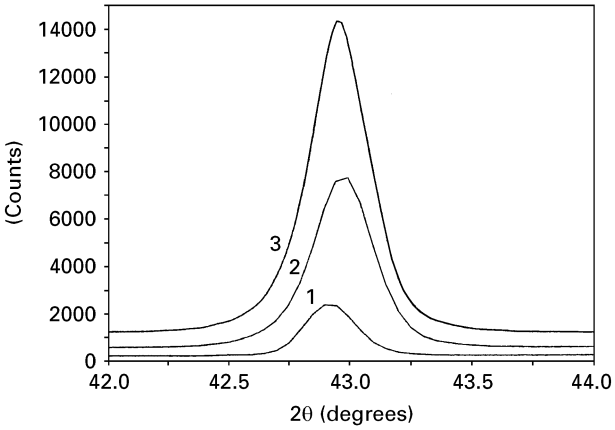

Figure 8 The basic effects on the crystallite size of MgO produced.

melt was faster and was complete at lower temperatures than in the $NaNO_3$–KNO_3 melt, which was in agreement with the TGA measurements.

The MgO powders precipitated in the $NaNO_2$–KNO_2 melt had smaller crystallite sizes than those produced in the $NaNO_3$–KNO_3 melt. The lower reaction temperatures and faster reaction in the nitrite melt were probably responsible for these smaller crystallite sizes [6].

It is thought that the acidic/basic difference in nitrites and nitrates resulted in the differences in the reactions and crystallite sizes of MgO powders produced. This led to the further study of the acidic/basic effect on the properties of the MgO powders produced.

Lux–Flood bases such as Na_2O_2, Na_2CO_3 and $NaNO_2$–KNO_2 were added to the less basic $NaNO_3$–KNO_3 melt to study their influences on the properties of the powders prepared. The reaction conditions were selected with an end temperature of 600 °C for 90 min in order to make sure that reactions occurred completely and 100% MgO was produced. XRD results (Fig. 7) indicated that with increasing concentration of base in the melt, i.e. the increase of the basicity of the melt, the XRD peaks became more and more broad, i.e. the crystallite size was decreased. The trend is shown in Fig. 8. With different bases added at the same concentration, the smallest crystallite size was produced by adding Na_2O_2 to the melt, then Na_2CO_3 and $NaNO_2$–KNO_2. This can be expressed in the following sequence in terms of their basicity

$$Na_2O_2 > Na_2CO_3 > NaNO_2\text{–}KNO_2$$

The results were similar to those obtained in the preparation of ZrO_2 powders [6]. Based on these results, the following relationship could be deduced

$$\boxed{\text{Higher basicity}} \longrightarrow \boxed{\text{Quicker reaction}} \longrightarrow \boxed{\text{Smaller crystallite size}}$$

TABLE III Chemical analysis of MgO prepared under different conditions

Starting materials	Systems	Temperature and time (°C, min)	Metal element (wt %) Na	K
$MgSO_4$	$NaNO_2$–KNO_2	500, 90	0.80 ± 0.03	0.21 ± 0.01
$MgSO_4$	$NaNO_3$–KNO_3	600, 30	0.21 ± 0.01	0.13 ± 0.01
$MgCl_2$	$NaNO_2$–KNO_2	500, 90	0.47 ± 0.02	0.12 ± 0.01
$MgCl_2$	$NaNO_3$–KNO_3	600, 90	0.15 ± 0.01	0.10 ± 0.01
$MgCl_2$	$NaNO_3$–KNO_3 + Na_2CO_3	600, 90	0.05 ± 0.01	0.03 ± 0.01
$MgCl_2$	$NaNO_3$–KNO_3 + Na_2O_2	600, 90	2.11 ± 0.01	0.02 ± 0.01

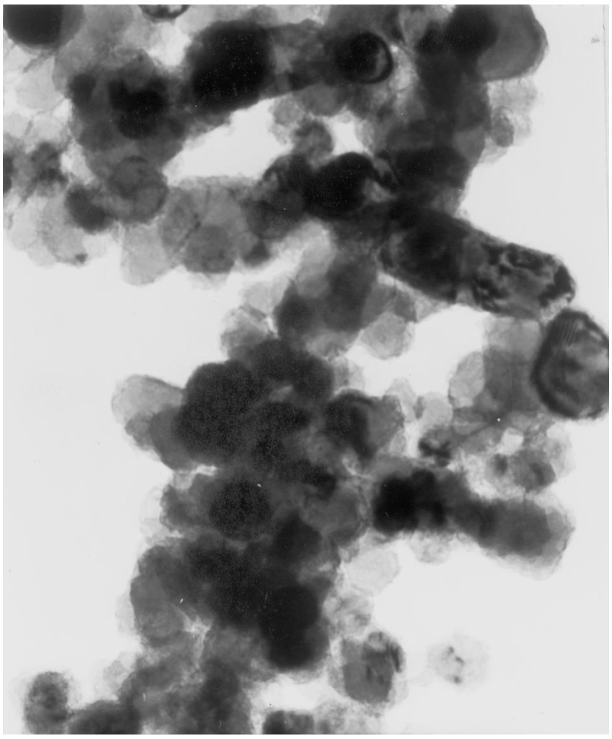

Figure 9 TEM micrograph of MgO obtained from the reaction of $MgSO_4$ with $NaNO_3$–KNO_3 at 600 °C, 90 min.

3.4. The purity of the MgO powders prepared

Table III lists the chemical analysis results of the MgO powders prepared under different conditions measured by AA. It can be seen that in general MgO powders with high purity were prepared. The purity of powders produced from the $NaNO_3$–KNO_3 melt was higher than that of powders prepared from the $NaNO_2$–KNO_2 solvent whatever starting materials were employed. When using $MgCl_2$ as the starting material, the powder purity could be further improved by adding the Lux–Flood base Na_2CO_3 to the $NaNO_3$–KNO_3 melt. However, the impurity levels increased if Na_2O_2 was added to the melt.

3.5. The extraction and TEM characterization of the MgO powders prepared

The precipitated MgO powders were extracted by the same method as that used for ZrO_2 [4]. After water washing, the powders were given a final wash by isopropyl alcohol accompanied by vacuum filtration. Then the powders were dried naturally.

It was observed from TEM that the MgO powders produced were spherical. If the powder was washed finally with alcohol accompanied by ultrasonic agitation, very uniform powders without severe agglomeration were obtained. A TEM micrograph of the powders prepared from $MgSO_4$ with $NaNO_3$–KNO_3 at 600 °C/90 min is shown in Fig. 9. It can be seen that the particles are well dispersed and that the size can be as small as about 50 nm. Comparing this with the crystallite sizes of the powders measured by XRD, which was about 40 nm, it appears that the elementary grain was monocrystalline. However the larger particles, which could be about 100 nm, showed some agglomeration of the powders.

4. Conclusion

Finely divided MgO powders ($<$ 100 nm) with low impurity levels of Na and K have been prepared by reactions using either $MgCl_2$ or $MgSO_4$ as starting materials in molten nitrates, nitrites or nitrates containing Lux–Flood bases.

The reactions of $MgCl_2$ and $MgSO_4$ in the molten $NaNO_2$–KNO_2 system occur and finish at lower temperatures and produce smaller crystallite size powders than in molten nitrate systems. In addition, in the same melt but using $MgCl_2$ rather than $MgSO_4$, the reaction takes place at lower temperatures.

The addition of Lux–Flood bases such as Na_2O_2 and Na_2CO_3 to the less basic $NaNO_3$–KNO_3 melt can decrease the reaction temperature as well as the crystallite sizes of the MgO powders precipitated.

The MgO powders produced were spherical, and the application of ultrasonic agitation during the washing process was effective in dispersing the powders produced and to give weakly agglomerated powders.

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