

轻烧温度及球磨工艺对盐湖  $\text{Mg}(\text{OH})_2$  制备镁砂烧结行为影响徐义彪<sup>1</sup>, 李亚伟<sup>1</sup>, 鄢文<sup>1</sup>, 周芬<sup>2</sup>

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**摘 要:** 以盐湖氢氧化镁为原料, 经 650、750、850 °C 和 950 °C 保温 1 h 轻烧, 随后将轻烧  $\text{MgO}$  团聚体球磨(0.5、1.0、3.0、5.0、12.0、24.0 h)破碎, 压制成型后于 1600 °C 保温 3 h 制备了烧结镁砂, 探讨了轻烧温度及球磨时间对镁砂显微结构及烧结性能的影响。结果表明: 随轻烧温度由 650 °C 升高到 850 °C, 轻烧  $\text{MgO}$  团聚体内微晶平均尺寸由 138 nm 增加至 180 nm, 尽管微晶活性降低, 但残余氢氧化镁减少, 镁砂致密性呈增大趋势。轻烧温度升高至 950 °C,  $\text{MgO}$  微晶平均尺寸迅速增加至 320 nm, 活性大幅降低, 镁砂晶粒尺寸及致密性显著降低。球磨工艺可以有效破坏轻烧  $\text{MgO}$  团聚体, 其平均尺寸球磨 0.5 h 后由 55.90  $\mu\text{m}$  迅速降至 2.69  $\mu\text{m}$ , 并随研磨时间增加至 12 h 逐渐降至 1.72  $\mu\text{m}$ , 但进一步增加球磨时间团聚体尺寸基本不变。 $\text{MgO}$  团聚体的破坏减少了生坯中大气孔, 提高了晶粒接触程度及传质速率, 促进了晶粒长大及样品致密化。850 °C 制备的轻烧  $\text{MgO}$  球磨 12 h 压制的生坯, 经 1600 °C 保温 3 h 烧结, 可制备平均晶粒 30.4  $\mu\text{m}$ , 体积密度达 3.42  $\text{g}/\text{cm}^3$  的致密镁砂。

**关键词:** 盐湖氢氧化镁; 烧结镁砂; 轻烧温度; 晶粒尺寸; 致密化

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镁砂具有熔点高(约 2 800 °C)及抗碱性渣性能优良的特性, 是炼钢转炉、电炉、钢包和其他冶金炉炉衬广泛使用的一种耐火原料<sup>[1-4]</sup>, 高的纯度及致密性是保证镁砂具备优良使用性能的关键。近年来, 随着钢铁工业的快速发展, 冶炼技术的进步对镁砂的品质提出了更高的要求, 高纯、高致密化也成为未来镁砂的主要发展方向<sup>[5]</sup>。

目前, 中国主要以菱镁矿为原料通过高温煅烧的方法生成烧结镁砂, 但中国菱镁矿一般是粗晶质菱镁矿, 高温下难以致密化, 使得生产的烧结镁砂体积密度在 3.30  $\text{g}/\text{cm}^3$  左右<sup>[6]</sup>。为提高镁砂致密性: 在工艺方面, 研究者们通过轻烧  $\text{MgO}$  水化处理及提高煅烧温度等措施促进了镁砂致密化, 但因设备条件及成本限制未能实现工业化生产<sup>[7]</sup>; 另一方面, 通过引入添加剂如  $\text{TiO}_2$ 、 $\text{Al}_2\text{O}_3$ 、 $\text{ZrO}_2$ 、 $\text{Y}_2\text{O}_3$  及  $\text{MgCl}_2$  等也可以有效促进镁砂烧结, 但生成的低熔相会显著降低镁砂的高温使用性能<sup>[8-11]</sup>。

与中国不同, 美国、西欧和日本等国一直致力

于采用海水镁盐合成高纯镁砂的研究, 开发出了高纯( $\text{MgO}$  含量 99%以上)高致密烧结镁砂(3.40  $\text{g}/\text{cm}^3$  以上)并占到世界高品质镁砂生产总量的 80%<sup>[9]</sup>。事实上, 中国除菱镁矿固态镁资源, 还有着丰富的盐湖镁资源, 仅柴达木盆地盐湖氯化镁储量就达 31.43 亿 t<sup>[12]</sup>。但长期以来, 中国盐湖仅主要用于制备钾肥, 镁资源未得到有效利用, 且作为钾肥副产品的水氯镁石长期堆积也破坏了生态环境<sup>[13]</sup>。此外, 相比菱镁矿, 盐湖镁资源制备的镁砂纯度高, 且制过程没有  $\text{CO}_2$  排放。为此, 利用中国盐湖镁资源生产高致密性的高档镁砂, 不仅可以有效促进中国镁质材料的快速发展, 还能为减排环保做出贡献。

基于此, 本工作以盐湖氯化镁生产的氢氧化镁为原料, 采用二步法制备了高纯的致密镁砂, 研究了轻烧温度及研磨时间对轻烧  $\text{MgO}$  形貌及烧结活性影响, 并探究了高温煅烧后镁砂的显微结构及致密化行为。

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## 1 实验

### 1.1 样品制备

采用氢氧化镁粉体[ $\text{Mg}(\text{OH})_2$ ] ( $D_{50} = 56.3 \mu\text{m}$ ,  $w(\text{MgO}) \geq 66.07\%$ ; 0.01% CaO, 0.08%  $\text{SiO}_2$ , 0.01%  $\text{Fe}_2\text{O}_3$ , 0.02%  $\text{B}_2\text{O}_3$ ,  $w$  为质量分数)为原料。图 1 中 TG-DTG 热分析(空气气氛)曲线表明, 氢氧化镁粉体原料的分解速率在 400 °C 左右达到最大值, 并在约 850 °C 时完全分解。基于此, 本工作首先将氢氧化镁粉体在 650、750、850 及 950 °C 热处理制备轻烧 MgO, 保温时间为 1 h, 加热速率为 5 °C/min。然后采用机械球磨法将制备的粉体进行磨细, 球磨时间为分别为 0.5、1.0、3.0、5.0、12.0 h 及 24.0 h, 球磨介质为氧化锆球和无水乙醇。然后, 将细磨干燥后的粉体在 100 MPa 压力下成型为尺寸  $\phi 20 \text{ mm} \times 20 \text{ mm}$  的生坯。最后, 将生坯于 1600 °C 保温 3 h 制得高纯镁砂样品。

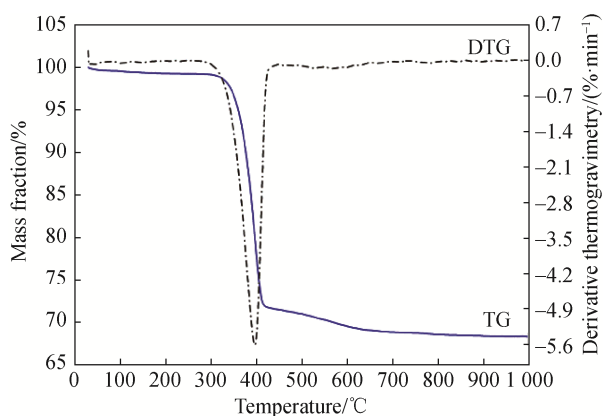
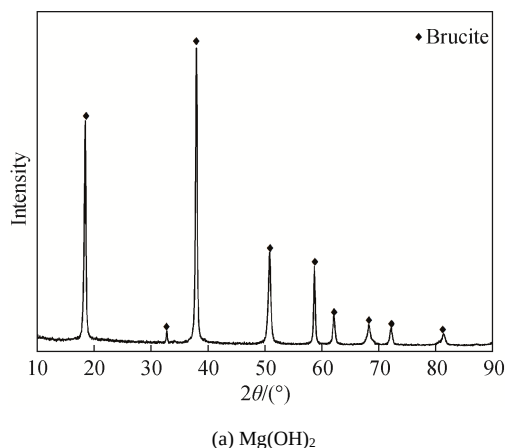
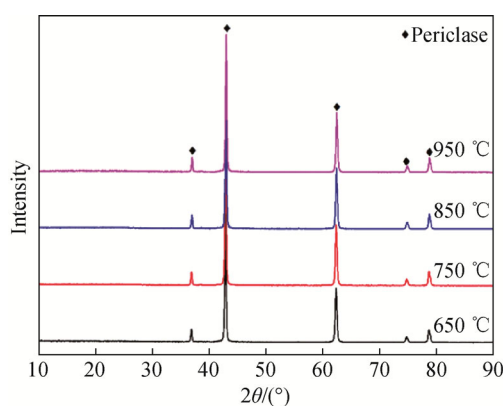


图 1 氢氧化镁原料的 TG-DTG 热分析曲线

Fig. 1 Thermal analysis results of TG and DTG curves for  $\text{Mg}(\text{OH})_2$



(a)  $\text{Mg}(\text{OH})_2$



(b)  $\text{Mg}(\text{OH})_2$  after treated at different temperatures

图 2 氢氧化镁原料及其不同轻烧温度处理后制得轻烧 MgO 的 XRD 谱

Fig. 2 XRD patterns of  $\text{Mg}(\text{OH})_2$  and  $\text{Mg}(\text{OH})_2$  after treated at different temperatures

### 1.2 样品表征

用 Nova Nano SEM400 型扫描电子显微镜观察样品的显微结构并结合 IE 350 Pental FET X-3 型能谱仪对样品微区成分进行分析。用 D8-Advance 型 X 射线粉末衍射仪分析产物物相组成, 测试条件: Cu 靶, 管电压 40 kV, 管电流 30 mA, 测试温度为 16 °C。用 HighScore Plus 软件对 XRD 谱进行拟合, 计算轻烧 MgO 晶胞参数。用 Mastersizer 2000 型激光粒度分析仪测试轻烧 MgO 粒度分布, 采用无水乙醇作为分散介质, 利用 BET 法计算其比表面积。采用 Archimedes 排水法检测镁砂样品的显气孔率和体积密度。采式(1)计算不同温度轻烧后氢氧化镁的分解率  $\alpha$ <sup>[5]</sup>。

$$\alpha = \frac{M_0 - M_1}{M_0 - M_t} \times 100\% \quad (1)$$

式中:  $\alpha$  为样品的分解率;  $M_0$  为样品的初始质量;  $M_1$  为样品轻烧后的质量;  $M_t$  为样品完全分解后的质量。

## 2 结果与讨论

### 2.1 轻烧温度对轻烧 MgO 物相组成及显微结构影响

图 2 为氢氧化镁原料及其在不同轻烧温度处理后所得轻烧 MgO 的 XRD 谱。由图 2a 可知, 氢氧化镁原料的 XRD 谱仅含有单一水镁石相(JCPDS 96-900-6794), 说明原料纯度很高。轻烧温度为 650 °C 时, 轻烧 MgO 的 XRD 谱上已观察不到  $\text{Mg}(\text{OH})_2$  相的衍射峰, 仅检测到方镁石相(JCPDS 01-089-4248)的衍射峰(图 2b)。随轻烧温度升高, 样品中方镁石的衍射峰强度不断增高, 且衍射峰更加尖锐, 表明提高轻烧温度促进了方镁石晶粒的发育长大, 使其结晶程度更好<sup>[14]</sup>。

氢氧化镁原料的显微结构及粒度分布如图 3 所示。由图 3 可知, 氢氧化镁粉体是片状  $\text{Mg}(\text{OH})_2$  微晶紧密堆积构成的球形团聚体, 且团聚体粒径主要分布于 40~100  $\mu\text{m}$  间。不同温度热处理后所得轻烧  $\text{MgO}$  粉体依然是球形团聚体, 且其粒度分布与氢氧化镁原料的基本相同(以图 4 所示 850  $^{\circ}\text{C}$  为例), 说明氢氧化镁团聚体热分解形成轻烧  $\text{MgO}$  团聚体后体积和形状基本没有变化。然而, 图 5 的放大 SEM 照片显示, 轻烧  $\text{MgO}$  团聚体由大量  $\text{MgO}$  粒状微晶及亚微米级气孔组成, 说明片状  $\text{Mg}(\text{OH})_2$  在热处理过程中分解为  $\text{MgO}$  微晶, 同时收缩形成团聚体内纳米气孔。此外, 轻烧  $\text{MgO}$  团聚体内的  $\text{MgO}$  微晶随轻烧温度升高不断变大, 这与 XRD 分析结果一致。 $\text{MgO}$  微晶平均尺寸在轻烧温度为 650、750  $^{\circ}\text{C}$  及 850  $^{\circ}\text{C}$  时, 分别为 135、145 nm 及 180 nm, 并在轻烧温度升高到 950  $^{\circ}\text{C}$  时迅速增大到 320 nm(表 1)。对应的, 轻烧  $\text{MgO}$  的比表面积随轻烧温度提高不

断降低, 在轻烧温度为 650、750  $^{\circ}\text{C}$  及 850  $^{\circ}\text{C}$  时分别为 629、592  $\text{m}^2/\text{g}$  及 455  $\text{m}^2/\text{g}$ , 并在轻烧温度升高为 950  $^{\circ}\text{C}$  时大幅降低至 260  $\text{m}^2/\text{g}$ 。

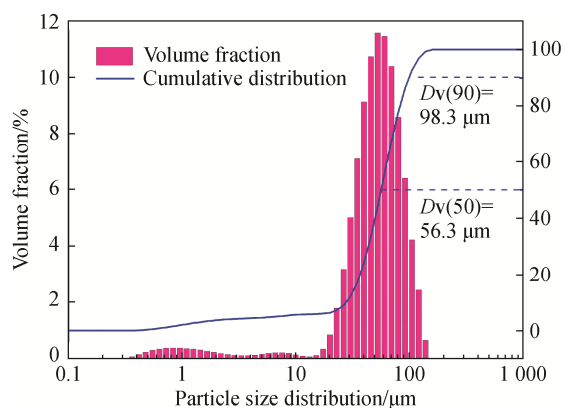
表 1 不同温度处理后轻烧氧化镁比表面积及  $\text{MgO}$  微晶平均尺寸

Table 1 Specific surface area and size of the  $\text{MgO}$  crystallite obtained at different temperatures

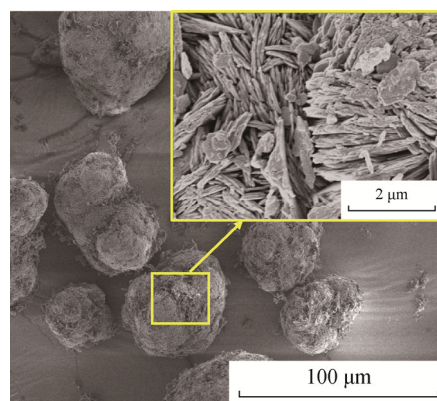
| Light calcination temperature/ $^{\circ}\text{C}$ | Average grain size of $\text{MgO}$ crystallite/nm | Specific surface area/ $(\text{m}^2 \cdot \text{g}^{-1})$ |
|---------------------------------------------------|---------------------------------------------------|-----------------------------------------------------------|
| 650                                               | 135                                               | 629                                                       |
| 750                                               | 145                                               | 592                                                       |
| 850                                               | 180                                               | 455                                                       |
| 950                                               | 320                                               | 260                                                       |

## 2.2 轻烧温度对镁砂样品烧结性能影响

表 2 为不同轻烧  $\text{MgO}$  粉体球磨 1 h 后压制的生坯在 1600  $^{\circ}\text{C}$  烧结制得镁砂样品的烧结性能。由表 2 可以看出, 650、750  $^{\circ}\text{C}$  及 850  $^{\circ}\text{C}$  轻烧后得到的  $\text{MgO}$



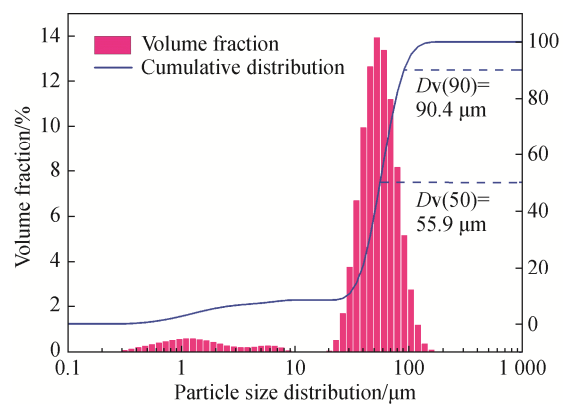
(a) Particle size distribution



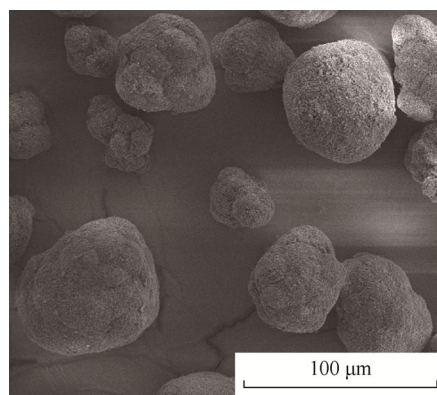
(b) SEM micrographs

图 3 氢氧化镁原料粒度分布及显微结构

Fig. 3 Particle size distribution and SEM micrographs of  $\text{Mg}(\text{OH})_2$  raw material



(a) Particle size distribution



(b) SEM micrographs

图 4 850  $^{\circ}\text{C}$  热处理后轻烧  $\text{MgO}$  粒度分布及显微结构

Fig. 4 Particle size distribution and SEM micrographs of light burned  $\text{MgO}$  after treated at 850  $^{\circ}\text{C}$

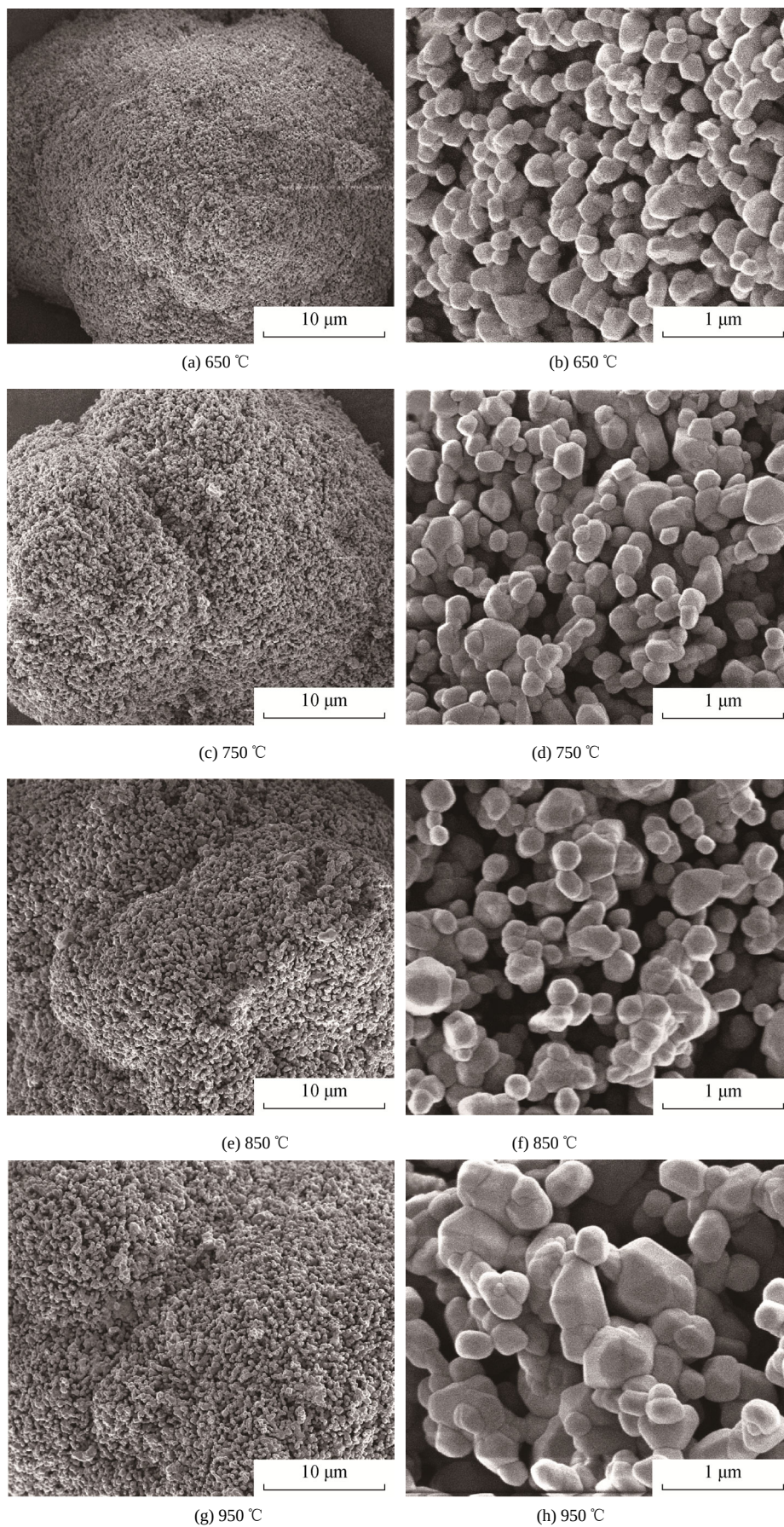


图5 不同温度轻烧后 MgO 的显微形貌照片

Fig. 5 SEM micrographs of light burned MgO treated at different temperatures

粉体高温煅烧制得样品致密性较高(相对致密度均在 90%以上), 体积密度分别为  $3.29$ 、 $3.31 \text{ g/cm}^3$  及  $3.32 \text{ g/cm}^3$ , 显气孔率分别为 3.7%、3.4%及 2.9%, 且样品致密性随轻烧温度升高略有增加。然而, 当轻烧温度升到  $950^\circ\text{C}$ , 对应轻烧  $\text{MgO}$  高温煅烧制得样品致密性显著降低, 相对致密度仅为 82.4%, 体积密度为  $2.95 \text{ g/cm}^3$ , 显气孔率高达 17.0%。此外,  $650^\circ\text{C}$  和  $750^\circ\text{C}$  时轻烧  $\text{MgO}$  中还有少量未分解的氢氧化镁, 而  $850^\circ\text{C}$  和  $950^\circ\text{C}$  条件下氢氧化镁已基本完全分解。

不同轻烧  $\text{MgO}$  粉体压制生坯  $1600^\circ\text{C}$  烧结样品表面形貌如图 6 所示。轻烧温度为  $650^\circ\text{C}$ 、 $750^\circ\text{C}$  及  $850^\circ\text{C}$  时, 对应  $\text{MgO}$  粉体高温煅烧制得样品晶粒尺寸较大, 主要分布于  $20\sim 50 \mu\text{m}$ ; 样品结构也相对较致密, 气孔主要以  $2\sim 8 \mu\text{m}$  的晶间孔形式存在, 还有少量  $1 \mu\text{m}$  以下的晶内气孔。这是由于这些轻烧  $\text{MgO}$  团聚体内  $\text{MgO}$  微晶小(表 1), 烧结活性高, 烧结过程物质传输及晶界迁移速度快, 促进了样品致密化

及晶粒长大。晶界的快速迁移及晶粒长大, 使得  $\text{MgO}$  团聚体中部分微孔来不及排除而被包裹于大晶粒内部<sup>[15]</sup>, 然而, 当轻烧温度升到  $950^\circ\text{C}$ , 煅烧样品结构疏松, 晶粒间存在大量气孔, 且晶粒细小 ( $5\sim 15 \mu\text{m}$ ), 这是由于轻烧  $\text{MgO}$  团聚体内  $\text{MgO}$  微晶较大, 烧结活性低, 驱动力小, 晶粒生长及气孔排除缓慢。另一方面,  $650^\circ\text{C}$  至  $850^\circ\text{C}$  煅烧得到的轻烧  $\text{MgO}$  晶格常数较大(表 2), 结构缺陷多, 促进了晶格活化及质点迁移, 加速了烧结过程及晶粒发育;  $950^\circ\text{C}$  煅烧得到的轻烧  $\text{MgO}$  晶格常数明显更小, 结晶性好, 烧结活性差, 阻碍了晶粒生长及样品的致密化。此外, 随轻烧温度从  $650^\circ\text{C}$  升高到  $850^\circ\text{C}$ , 煅烧样品气孔略有减少, 这是由于轻烧  $\text{MgO}$  分解率提高(表 2), 残余  $\text{Mg}(\text{OH})_2$  分解形成的  $\text{H}_2\text{O}(\text{g})$  及气孔减少。基于上述结果可知, 轻烧温度为  $850^\circ\text{C}$  时, 氢氧化镁分解完全且轻烧  $\text{MgO}$  活性高, 对应镁砂样品具有最佳的烧结性能, 但样品致密性依然低于  $3.40 \text{ g/cm}^3$ 。

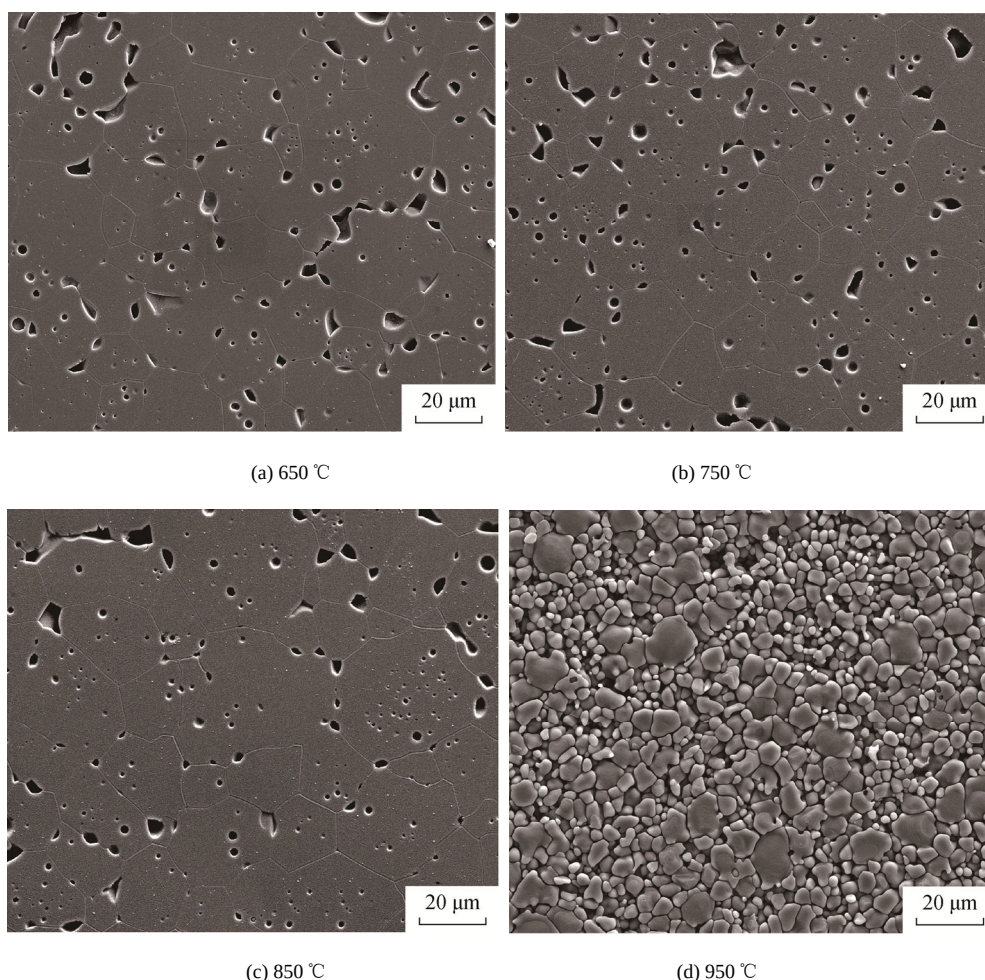


图 6 不同轻烧温度  $\text{MgO}$  粉体压制生坯烧结样品的表面形貌

Fig. 6 SEM micrographs of sintered specimens using light burned  $\text{MgO}$  treated at different temperature

表 2 不同轻烧 MgO 粉体球磨 1 h 后压制的生坯煅烧镁砂烧结性能

Table 2 Properties of sintered magnesia from different light burned MgO powders after ball milled 1 h

| Light calcination temperature/°C | Bulk density/(g·cm <sup>-3</sup> ) | Apparent porosity/% | Relative density/% | Decomposition ratio/% | MgO lattice parameters/nm |
|----------------------------------|------------------------------------|---------------------|--------------------|-----------------------|---------------------------|
| 650                              | 3.29                               | 3.7                 | 91.9               | 97.2                  | 0.421 27                  |
| 750                              | 3.31                               | 3.4                 | 92.5               | 98.5                  | 0.421 25                  |
| 850                              | 3.32                               | 2.9                 | 92.8               | 99.7                  | 0.421 22                  |
| 950                              | 2.95                               | 17.0                | 82.4               | 99.8                  | 0.421 13                  |

### 2.3 细磨工艺对镁砂样品烧结性能影响

由图 4 可知, 轻烧 MgO 是微晶构成的球形团聚体。一般认为, 团聚体的存在会降低生坯的可压缩性及 MgO 晶粒之间的接触, 阻碍镁砂的致密化过程<sup>[7]</sup>。为此, 在轻烧 850 °C 基础上, 通过球磨工艺破坏轻烧 MgO 团聚体, 进一步提高样品的致密性。轻烧 MgO 球磨不同时间后的粒度分布如图 7 所示。对比图 4a 可知, 球磨 0.5 h 后, 轻烧 MgO 的粒度呈双峰分布, 说明已有部分团聚体被有效打破。随着研磨时间增加, 大粒径的团聚体数量不断减少, 并在研磨时间达到 12.0 h 后基本消失, 均转化为 1~2 μm 的粉体。球磨 0.5 h 后, 轻烧 MgO 的平均粒径由 55.9 μm 迅速降低至 2.69 μm, 并随研磨时间增加至 12.0 h 逐步降低至 1.72 μm(图 8)。然而, 进一步增加研磨时间至 24.0 h, 轻烧 MgO 的平均粒径仅降低为 1.70 μm。图 9 轻烧 MgO 球磨前后的显微结构对比也表明, 球磨 12.0 h 轻烧 MgO 团聚体已均被有效打破。

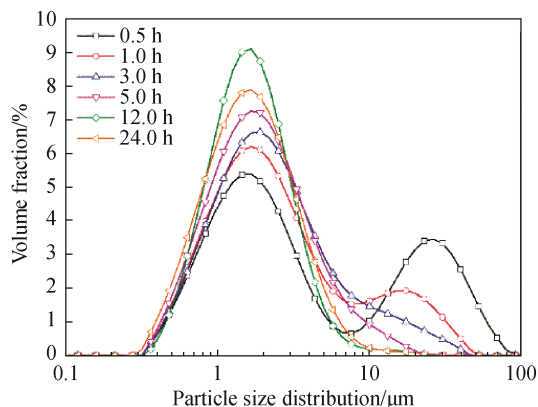


图 7 轻烧 MgO 经不同时间球磨后粒径分布曲线

Fig. 7 Particle size distribution of light burned MgO after ball grinding at different time

表 3 为 850 °C 轻烧 MgO 球磨不同时间压制生坯 1 600 °C 烧结制得镁砂烧结性能。由表 3 可以看出, 随球磨时间由 0.5 h 增加至 12.0 h, 样品的平均晶粒尺寸及致密性不断增加; 样品体积密度由 3.21 g/cm<sup>3</sup> 逐渐增加至 3.42 g/cm<sup>3</sup>, 显气孔率由 5.3% 降低至

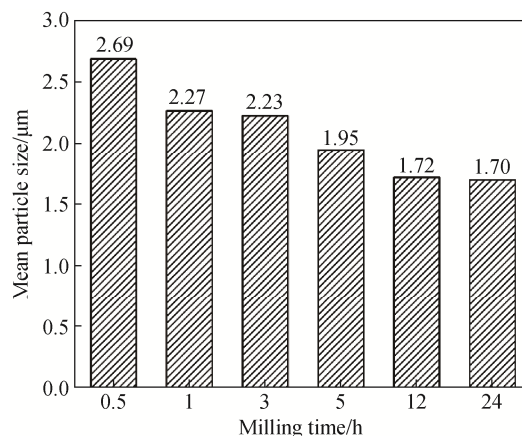
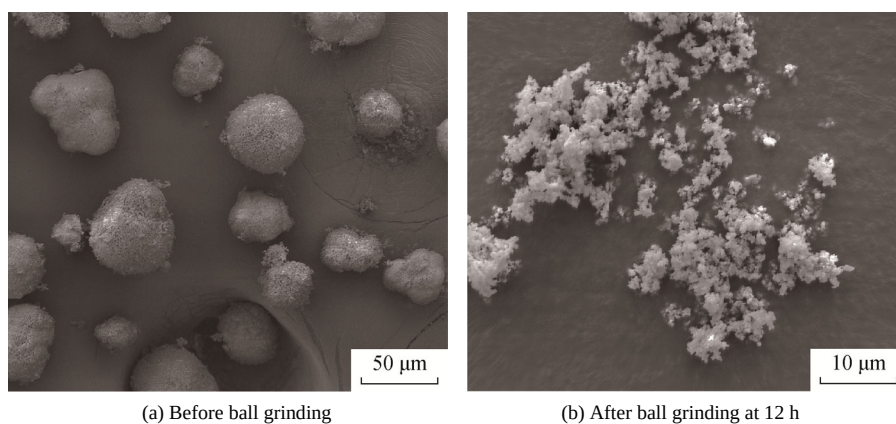


图 8 轻烧 MgO 经不同时间球磨后平均粒径

Fig. 8 Mean particle size of light burned MgO after ball grinding at different time

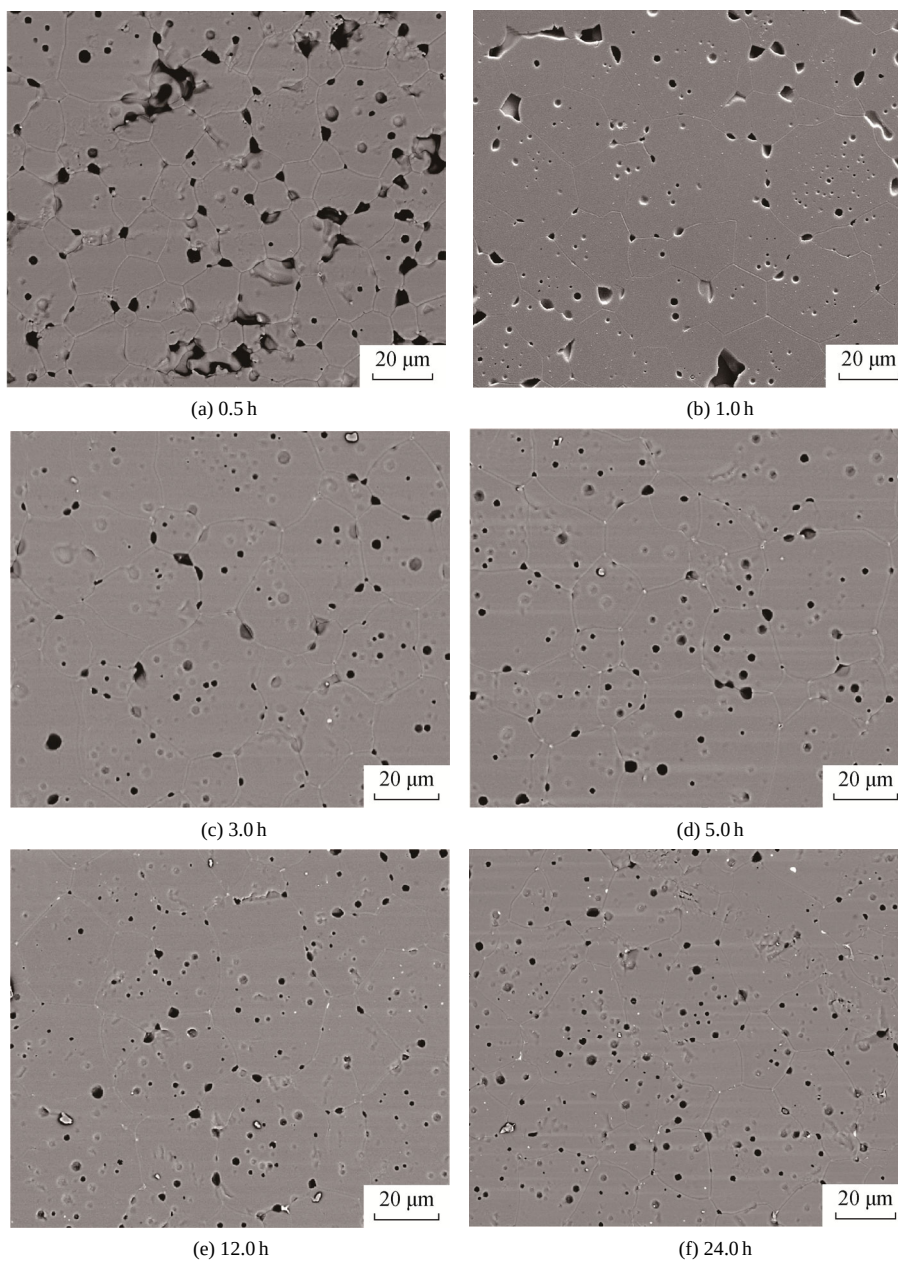
0.6%, 平均晶粒尺寸由 18.6 μm 增加至 30.9 μm。然而, 进一步延长研磨时间至 24.0 h, 样品的致密性基本没有变化, 体积密度仅由 3.42 g/cm<sup>3</sup> 增加至 3.43 g/cm<sup>3</sup>。

850 °C 轻烧 MgO 球磨不同时间压制生坯煅烧制得镁砂表面形貌如图 10 所示。由图 10 可以看出, 球磨时间为 0.5 h 时, 烧结镁砂样品晶粒尺寸较小, 主要分布于 10~30 μm; 样品结构也较疏松, 基体中存在较多不规则气孔, 且气孔尺寸较大, 最大孔径可达 10 μm。这是由于球磨 0.5 h 后, 轻烧 MgO 中还存在较多的团聚体(图 7), 压制生坯中团聚体间不能紧密接触存在较多大气孔, 同时团聚体间晶粒传质速度慢, 阻碍了晶粒长大及样品致密化<sup>[16]</sup>。随着球磨时间增加, 样品晶粒尺寸逐渐增大(表 3), 同时大气孔数量逐渐减少, 样品致密性逐渐提高, 这说明球磨工艺有效打破了团聚体, 提高了生坯的可压缩性及 MgO 晶粒之间的接触, 加速了传质, 促进了晶粒长大及样品致密化。球磨时间为 12.0 h 时, 样品中不规则大气孔消失, 仅有少量圆形小孔均匀分布于样品内, 进一步延长球磨时间至 24.0 h, 样品显微结构未发生明显变化。考虑到生产效率及节能, 轻烧氧化镁球磨时间 12.0 h 最佳。



(a) Before ball grinding

(b) After ball grinding at 12 h

图 9 轻烧  $\text{MgO}$  球磨前和球磨 12 h 后显微形貌照片Fig. 9 SEM micrographs of  $\text{MgO}$  before ball grinding and after ball grinding at 12 h

(a) 0.5 h

(b) 1.0 h

(c) 3.0 h

(d) 5.0 h

(e) 12.0 h

(f) 24.0 h

图 10 850 °C 轻烧  $\text{MgO}$  球磨不同时间压制生坯 1600 °C 烧结镁砂表面形貌Fig. 10 SEM micrographs of magnesia sintered at 1600 °C prepared by light-burned  $\text{MgO}$  treated at 850 °C with various milling time

表 3 850 °C 轻烧 MgO 球磨不同时间压制生坯 1600 °C 烧结制得镁砂烧结性能

Table 3 Sintering properties of magnesia sintered at 1600 °C prepared by light-burned MgO treated at 850 °C after various milling time

| Milling time/h | Bulk density/(g·cm <sup>-3</sup> ) | Apparent porosity/% | Relative density/% | Grain size/μm |
|----------------|------------------------------------|---------------------|--------------------|---------------|
| 0.5            | 3.21                               | 5.3                 | 90.0               | 18.6          |
| 1.0            | 3.32                               | 2.9                 | 92.8               | 24.3          |
| 3.0            | 3.38                               | 1.2                 | 94.4               | 26.8          |
| 5.0            | 3.39                               | 1.0                 | 94.7               | 28.2          |
| 12.0           | 3.42                               | 0.6                 | 95.5               | 30.4          |
| 24.0           | 3.43                               | 0.5                 | 95.8               | 30.9          |

### 3 结论

1) 随轻烧温度由 650 °C 升高到 850 °C, 轻烧 MgO 团聚体内微晶平均尺寸由 138 nm 增加至 180 nm, 尽管微晶活性略有降低, 但残余氢氧化镁减少, 烧结镁砂致密性呈增大趋势。轻烧温度进一步升高至 950 °C, 轻烧 MgO 团聚体内微晶平均尺寸迅速增加至 320 nm, 其烧结活性及驱动力大幅降低, 进而显著降低了样品晶粒尺寸及致密性。

2) 球磨工艺可以有效破坏轻烧 MgO 团聚体, 其平均尺寸球磨 0.5 h 后由 55.90 μm 迅速降低至 2.69 μm, 并随研磨时间增加至 12.0 h 逐渐降低至 1.72 μm, 但进一步增加球磨时间团聚体尺寸基本不变。MgO 团聚体的破坏降低了生坯中大气孔数量, 提高了晶粒接触程度及传质速率, 促进了晶粒长大及样品致密化。

3) 850 °C 轻烧得到的轻烧 MgO 球磨 12.0 h 压制生坯, 经 1600 °C 保温 3.0 h 烧结, 可制备平均晶粒尺寸 30.4 μm, 体积密度达 3.42 g/cm<sup>3</sup> 的致密烧结镁砂。

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## Effects of Calcination Temperature and Ball Milling on Sintering Behavior of Magnesia from Salt Lake Mg(OH)<sub>2</sub>

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### Extended Abstract

**Introduction** Magnesite, acted as a refractory material, is widely used in industrial metallurgical furnaces owing to its high refractoriness and excellent corrosion resistance to basic slag and metal melt. Generally, the fabrication of high-quality magnesia

aggregates with high purity and high bulk densities ( $>3.40 \text{ g/cm}^3$ ) plays a critical role in producing MgO-based refractories that show satisfying mechanical properties and corrosion resistance. In China, magnesite is mainly used as a raw material to produce sintered magnesia at high-temperature. while, as the generally coarse-grained of magnesite, it is difficult to produce high density magnesia even sintered at high temperatures, being due to the poor sintering property. As reported that the introduction of oxides additives such as  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ , etc. could effectively promote the sintering of magnesia. However, the additives also resulted in a low melting phase formation, which significantly reduced the high-temperature performance of magnesia. In fact, besides magnesite, China also has abundant magnesium resources in salt lakes. Compared to magnesite, magnesia derived from salt lake owns the advantage of higher purity and without  $\text{CO}_2$  emissions during the production process. In this work, magnesium hydroxide produced from salt lake was used as raw material to prepare the high-purity dense magnesia via a two-step method. Effects of calcination temperature and grinding time on the morphology and sintering activity of MgO were studied, microstructure and densification behavior of magnesia sintering at high-temperature were investigated also.

**Methods** Magnesium hydroxide powder [ $\text{Mg}(\text{OH})_2$ ] was used as raw material. Firstly, it was lightly-burned to produce MgO when calcined at 650, 750, 850  $^\circ\text{C}$  and 950  $^\circ\text{C}$  with a holding time of 1 h and a heating rate of 5  $^\circ\text{C}/\text{min}$ , repetitively. Secondly, the obtained powder was finely ground using mechanical ball milling for 0.5, 1.0, 3.0, 5.0, 12.0  $^\circ\text{C}$  and 24.0 h, respectively, here, zirconia balls acted as the media and anhydrous ethanol as solvent. Thirdly, the finely ground and dried powders were pressed into a green body with a size of  $\phi 20 \text{ mm} \times 20 \text{ mm}$  under a pressure of 100 MPa. Finally, the green body was sintered at 1600  $^\circ\text{C}$  for 3 h to prepare the magnesia samples.

**Results and discussion**  $\text{Mg}(\text{OH})_2$  would decompose into MgO agglomerates with crystallite and nano-pores when calcined at 650–850  $^\circ\text{C}$ . The crystal size of MgO continuously increased with the increase of calcination temperature, and rapidly increased to 320 nm when temperature reached to 950  $^\circ\text{C}$ . Correspondingly, the specific surface area of calcined MgO decreased with the increase of the temperature. In addition, a small amount of undecomposed  $\text{Mg}(\text{OH})_2$  still existed in lightly-burned MgO even treated at 750  $^\circ\text{C}$ . When calcined at 850  $^\circ\text{C}$ , the  $\text{Mg}(\text{OH})_2$  decomposed completely and the obtained MgO crystallite possessed high activity and best sintering properties. With the increase of grinding time, the number of MgO agglomerates decreased continuously and well disappeared when the grinding time reached to 12 h, and the average size of MgO agglomerates decreased to 1.72  $\mu\text{m}$ . However, by further increasing the grinding time to 24 h, the average size of MgO agglomerates only decreased to 1.70  $\mu\text{m}$ . The destruction of MgO aggregates could reduce the number of large pores and improve the contact between agglomerates in the green specimens, both could enhance the densification of the sintered specimens.

**Conclusions** When the calcination temperature increased from 650  $^\circ\text{C}$  to 850  $^\circ\text{C}$ , the sintering activity of crystallite in MgO agglomerations reduced slightly due to the increase of average size of MgO crystallite from 138 nm to 180 nm, while the density of sintered magnesia exhibited an increasing trend as content of residual  $\text{Mg}(\text{OH})_2$  in MgO agglomerations reduced. When calcined at 950  $^\circ\text{C}$ , the sintering activity of the crystallite reduced dramatically and average size sharply increased to 320 nm, which obviously decreased the sintering activity and reduced the grain size and density of the specimens. The MgO agglomerations can be effectively crushed via the ball milling process, whose average size sharply reduced from 55.90 to 2.69  $\mu\text{m}$  when ball milling for 0.5 h, and then this value decreased slowly to 1.72  $\mu\text{m}$  when ball milling increasing to 12 h. However, a further increase of ball milling time would not produce the average size. The crush of MgO agglomerations could reduce the large pores in green compacts and enhanced the bonding between MgO crystallites, therefore enhancing the grain growth and densification of the specimens. When calcination temperature and milling time were 850  $^\circ\text{C}$  and 12 h, respectively, a dense magnesia with an average grain size of 30.4  $\mu\text{m}$  and bulk density of  $3.42 \text{ g/cm}^3$  could be prepared when sintered at 1600  $^\circ\text{C}$  for 3 h.

**Keywords** salt lake magnesium hydroxide; sintered magnesia; calcination temperature; grain size; densification