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THE CORRELATION OF THE STRENGTH OF MAGNESIUM HYDROXIDE COMPACTS WITH HEAT TREATMENT

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ABSTRACT

The thermal decomposition of magnesium hydroxide is correlated with the effect of the temperature treatment on the strength of compacted discs of the material. The alteration in the strength of the magnesium hydroxide which had been subjected to various temperatures between 450° and 800°C was obtained as well as by altering degassing conditions between ambient and 300°C. Up to twenty samples were broken at each of these conditions and the results show a skewed distribution of data points. The experiments on powders heat treated up to 800°C showed an increase in strength beyond 400°C. The TG data showed decomposition in the range 350°C to 500°C although farther weight loss was shown to be due to the presence of about 2-3% carbonate. The most useful correlation between the heat treatment and the strength is seen in the linear relationship between the surface area and the modules of rupture.

INTRODUCTION

The variation in the strength of compacted powders with adsorption has been shown in previous publications [1,2,3,4]. These results are in accord with the Griffith Flaw Theory [5]. Adsorption generally causes a decrease in strength. The experimental strength under vacuum although considerably higher than data obtained in atmospheric conditions where adsorption is possible are still less than the theoretical strengths often by a factor of severalfold. This was explained by Griffith on the basis of a stress concentration behavior at the apex of flaws orientated in the direction of the applied stress.

Griffith gave the expression

$$R = \left(\frac{2Y\gamma}{\pi C}\right)^{1/2}$$

as relating the modules of rupture (R) of a compact with surface energy (τ) where Y is the Youngs modules for the solid and 2C is the length of the crack at which failure occurs. Adsorption causes a decrease in surface energy ($\Delta\gamma$) which can be calculated from the adsorption isotherm using the Gibbs adsorption equation [6,7]. This concept explains with some modification all the observed phenomena relating to the selective effect of adsorption upon the strength of compacts. Another factor which alters the strength of compacts is the heat treatment received by the material used in the preparation of the compact. Factors which

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must be considered are the effectiveness of degassing for different times and temperatures and alterations at higher temperatures in the chemical nature of the surface. The effect of heat treatment on compacted samples of coals has been extensively studied and it has been shown that alterations in the strength of coal compacts can be related to surface area, and pore structure, some of these being created by the conditions of preparing the compact [8,9,10,11]. In this study the effect of heat treatment and decomposition of magnesium hydroxide upon the strength of compacts of this material are reported.

EXPERIMENTAL

Apparatus

The modules of rupture of the compacted discs was determined on equipment which could be evacuated down to a vacuum or held isolated under given conditions of pressure. Previous equipment of this kind was constricted in glass, here the equipment was made largely of metal components and up to 20 discs could be tested at a time [1]. The discs to be tested were 31.75 mm in diameter and prepared under carefully controlled compacting pressures. The discs rest on two parallel supports and are broken by a force acting across their diameter. In previous papers a full description is available [1,3,4].

The surface area, and nitrogen adsorption isotherms were determined volumetrically [12]. A Stanton Redcroft TG unit was used for the heat treatment work. The heating rate for rising temperature experiments in air was $4° \mathrm{min}^{-1}$ and 0.5 g of sample was used in each experiment.

A Grubb Parsons infra red CO_2 analyzer was also used in this investigation.

Material

The magnesium hydroxide was prepared by leaving light magnesium oxide in contact with a large excess of water for a long time and then drying below 100°C. The manufacturers supplied the following information (arsenic < 2ppm, Lead < 10 ppm, Iron < 0.04%, Cl < 0.07%, SO_4 < 0.5%). Discs to the dimensions already specified were made by compaction using 5.00 ± 0.1 g of degassed powder. The compaction pressure was 10,000 lbs. and this was maintained for 3 minutes. The degassing conditions are reported for each series of experiments recorded in Tables 2, 3 and 4.

RESULTS AND DISCUSSION

The TG results in air revealed the decomposition of the magnesium hydroxide according to the reaction

$$Mg(OH)_2 \;\text{-->}\; MgO + H_2O$$

occurring in the region 350°-500°C. A further small loss in weight (about 3-5%

TABLE 1

TG Results on Decomposition of Magnesium Hydroxide

Atmosphere	Mass Loss %	Temperature Range in C°	Stages in Decomposition	Comment
Air	0-3%	25-350°	Adsorbed material	Water and CO_2 evolved
	3-30.5%	350-500°	Decomposition of of $Mg(OH)_2$ to MgO	CO_2 also evolved
	30.5-33%	500-750°	Sharp alteration in wt at 680°	CO_2 evolved
Vacuum	0-3%	25-250°	Adsorbed material	Water and CO_2 evolved
	3-28%	250-325°	Main decomposition to MgO	
	28-32%	325-550°		CO_2 evolved

TABLE 2

Variation of Breaking Load at Atmospheric Pressure with Degassing Condition of Powders

Degassing Conditions		Mean Breaking Load g	Standard Deviation g	Number of Observations
Temperature °C	Time h			
Room	-	4268	458	10
30	18	2880	951	53
56	18	2268	797	51
75	6	3371	1338	50
106	6	3266	1000	53
106	18	1979	755	51
150	18	2013	495	20
200	18	1656	299	19
250	18	1365	613	15

depending on individual samples) occurred in the region between 500°-750°C. The infra red gas analysis trace reveals that carbon dioxide was continually last first during the initial drying period, then during the main decomposition and finally in the temperature region 500-750°C already noted. It is estimated that the carbonate content of the magnesium hydroxide was between 2 and 3%.

The effect of degassing temperature on the strength showed not only a variation in the strength but also a wide distribution of the individual results. The data is recorded in Table 2. The most notable observation here is that degassing at 200°C gave the most reproducible results with a standard deviation of 299. The larger deviations recorded at temperatures below this were attributed to insufficient and irregular loss of moisture from the powders on degassing. The larger values recorded at higher degassing temperatures were thought to be due to stress caused by initial decomposition.

A series of heat treated powders were prepared under identical conditions as noted above except that the powder had been heat treated to temperatures up to 800°C before compacting into a disc. The results are shown in Table 3. The data indicates a decrease in strength initially followed by a sharp increase in strength over the temperature range 500-800°C. A more meaningful relationship is obtained if one considers the surface areas of the heat treated materials. This kind of information for a separate series of experiments is shown in Table 4. The magnesium hydroxide was heated in air at temperatures ranging from 200° to 800°C as the breaking load increase the measured surface area of the powder reduces. This means that the strength of the areas of contact between the particles in the disc is considerably enhanced if the powder is previously sintered. This can be explained as follows. As sintering proceeds the number of discrete particles reduces. When a given pressure is applied to a fixed weight of powder the number of points of contact reduces so the pressure at each point increases enabling the particles to be pressed closer together producing better adhesion.

The surface area of the powder, heat treated at 800°C is approximately the same as that for powder heated at 200° or 300°C for the same time. The strength of the compact however is approximately two and a half times as great. The difference must be that at 200 or 300°C we are still compacting hydroxyl groups together (as seen from the TG data). At 800°C these hydroxyls have almost all been removed and we have a pure magnesium oxide surface which produces a compacted disc of much higher strength.

It was also noted that the spread of results under any one set of conditions

TABLE 3

Variation of Breaking Load under Vacuum with Temperature of Treatment of the Powder

Temperature of Treatment °C	Peak Breaking Load g	Mean Breaking Load g	Standard Deviation g	Number of Observations
450	1530	1568	436	16
500	1150	1195	557	18
600	3150	3098	700	15
700	–	3504	1212	15
800	3700	5502	2784	14

TABLE 4

Variation of Strength with Surface Area of Powder

Temperature °C	Surface Area m^2g^{-1}	Breaking Load g
200	32	2000
300	33	1750
400	200	1600
500	127	1700
600	91	2650
700	61	3800
800	30	5100

were considerably skewed when the degassing was carried out under 106°C. This is associated with non-uniformity of the powders with respect to the extent of drying. These experiments also reveal that the more thoroughly the powder is dried the weaker is the resultant compact (see Table 2). Certainly the surface area available to nitrogen is reduced by the pressure of adsorbed water (Table 5).

It is apparent from the data presented here that both the extent and nature of the particle surfaces play a role in determining the effect of temperature on the strength of compacts of magnesium hydroxide.

TABLE 5

Variation in Specific Surface Area of Compacts and Powder with Degassing Temperature

Form of Material	Temperature oC	Surface Area m^2g^{-1}
Powder	Ambient	31.8
	100	31.5
	150	31.0
	300	32.3
Compact	25	22
	55	18.4
	100	27.8
	100	21.2
	150	22
	150	26.8
	200	26
	250	27.9
	300	26.2

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