The 1976 Howe Memorial Lecture
The Iron and Steel Society of AIME

Men and Magnesia

JOHN H. CHESTERS

The unusual title does not indicate an interest in pharmaceuticals—just an overwhelming conviction that success in research, development, and, particularly, application depends as much on men as on the materials or new techniques tested. The hypothesis will be illustrated by case histories with which the lecturer has been closely associated and where the human factors can be identified without any recourse to statistical methods. These range from the true density of pure magnesia to the origins of the sea water magnesia process.

THE introduction to a lecture (and even more to an article or book) is usually the most important, and also the most difficult, part of the exercise. It is important because if you don't capture your audience in the first few sentences you are likely to have lost them forever. It is difficult because you do not know yet what it is you are going to introduce. For this reason I find it best to write the introduction after I have finished the rest of the text. With memorial lectures there is an additional problem: the author sincerely wishes to honor the man concerned but probably never knew him. Henry Marion Howe died when I was sixteen. At that time I had at least one thing in common with this remarkable metallurgist—I was convinced that I wanted to get into industry—a conviction I might add that is shared by far too few of today's schoolboys and students.

Fortunately for me your past president, Jim Austin, whom I have known and admired for more than forty years, chose for his Memorial lecture a biography not only of H. M. Howe but of his equally eminent father—referred to by the biographer as Dr. Howe. A few days after reading the biography, in the hope of finding a personal link, I was wandering around London and found a new (to me) Greek restaurant, just two blocks west of Tottenham Court Road. I was tempted to enter—in spite of the fact that the windows, unlike those of the rather more elegant restaurants nearby, were not fitted with the latest refinement: steel mesh screens to keep out unrequested bombs—by the obvious exuberance of both staff and diners. The decibels were well above the Concorde level, but the Zorba type music quite intoxicating. So too, I found, was the wine. The association with Dr. Howe whose exploits in Greece, where, says Austin, he joined with Lord Byron in the war against the Turks, was irresistible. I could share his enthusiasm as I sat within two yards of the manager, who performed a perpetual triple miracle: checking the handbags of the ladies for bombs as they entered the restaurant, dancing to the music, and eating a shiskabab with obvious relish, even though I imagined this was his inevitable evening dish—they didn't serve very much else!

Thus stimulated I started to write this introduction.

The Howe Memorial Lecture was established in 1923 by the Iron and Steel Division, now the Iron and Steel Society, of AIME. The Lecturer is selected for his outstanding contributions to the science and practice of iron and steel metallurgy or metallography.

DR. JOHN H. CHESTERS has been interested in refractories since graduating from the University of Sheffield in 1928. He was awarded a Metropolitan Vickers scholarship to study the problem of refractory materials in the induction furnace and was awarded a Ph.D. for his work in 1931. In the same year he won a Robert Blair Fellowship to do research on refractories at the Kaiser Wilhelm Institut für Silikatforschung in Berlin. From 1932 to 1934, he held a Commonwealth Fund Fellowship at the University of Illinois.

In 1934 he took charge of the Refractories section of United Steel's newly formed Central Research Department. Ultimately he became director of the Corporate Laboratories of the British Steel Corporation, retiring in July 1971.

Among his awards, Dr. Chesters has received the Sir Robert Hadfield Medal of the Iron and Steel Institute, AIME's Robert Hunt Award and Benjamin Fairless Award. In 1969 he was made a Fellow of the Royal Society and in 1970 was awarded the Order of the British Empire.

If it sounds a drunken effort you will know why. At least I can claim "vino veritas". Not only, I remembered, had Dr. Howe gone to Greece and loved its people; he had gone later via Paris to Berlin where he got himself arrested although his mission was merely to distribute money collected for Polish refugees. In November 1939, shortly after we declared war on Germany, a United Steel colleague and I "volunteered" to join with one or more Frenchmen (it turned out to be one) in some economic warfare. We were told by H. M. Government that they badly needed low iron magnesite to make magnesium for aeroplanes and incendiary bombs, and that the Germans badly needed chrome ore for steelmaking. We were to rendezvous in Paris and proceed via Belgrade to Athens and probably to Turkey. Armed with half a dozen visas we flew at 1000 feet above sea level to Le Bourget. There we were conducted to a small tent where we were asked to show our papers. There had been no time in the 48 hours notice for protocol. Half the embassies and consulates were closed. Even the French visa was only obtained by pressure from the Foreign Office, though it was the French with whom we were to cooperate. Another was obtained by our courier literally sticking his foot in the door until we were allowed to enter. The consul here was very angry. He said it was a religious holiday and there was no one on duty. We replied that we were sorry to trouble him in this ruthless manner but "there was a war on". Like many top managers faced for the first time with doing the job themselves—in this case issuing a visa—he was flummexed. He tried half a dozen rubber stamps before he found one with the right message! In the rush my profession had been described in one of the papers as "Sorcier", a vain attempt to translate into French the word for "research worker"! When the little man in the tent at Le Bourget read this he showed obvious anxiety, if not shock. "Vous êtes vraiment sorcier"? he said. To which I merely replied "Oui, Oui". He stamped my passport immediately and handed it back—evidently preferring to admit a spy with magic powers, even though a possible spy, rather than risk being turned into a frog.

We soon realized that our first problem was to disguise the object of our mission. The stamp of the French Ambassador—an accident of timing—afforded us diplomatic status in France, but what after that? We hit on a solution, at least for the British representatives, that worked like a charm. We would pose as playboys with all that entails—or most of it! On arrival in Belgrade where, as everywhere, we had to display our passports, we took a splendid room in the best hotel and, having unpacked, proceeded to the bar where my British colleague drank whisky after whisky—he had spent most of his working life in India. Not being much of a drinker I asked the advice of the bar man, who suggested gin and grapefruit. It was not until about 3 A.M. that I realized my mistake, for each glass (and I had had about six) contained a complete Jerusalem grapefruit. The risk I was watching (getting tiddly and losing my passport) was replaced by a boiling stomach, which with an open hearth furnace would have suggested bottom trouble and the need to tap immediately. After playing poker dice with one another for about an hour we had dinner, starting, of course, with caviar. The next night, unclouded by the G and G's, I noticed an item on the bill that stuck out about two digits beyond the rest. "Whatever", I said, "is this". The waiter replied "That, sir, is for the caviar". But I said it is only one-tenth that price on the menu. "That, sir, he responded, is for a spoonful. You had a tin". Not to worry, what are a few extra dinars when you are commissioned to spend millions.

Most nights we strengthened our disguise by a visit to a night club—we must have gone more times in that month than would be considered reasonable even in Las Vegas. It was the Commercial Attachê at the Consulate who introduced us, and I was amazed to find that this was one of his main sources of information. My colleague insisted we were followed not only in Belgrade but on the train and in Athens, and by a suspicious looking German gentleman. I doubted it, though I had to agree that we kept seeing him! Maybe he was a playboy disguised as a German—after all they get the magazine too. Once in Belgrade we thought the game might be up when a pressman craved an interview, having heard that we were buying "manganese". We truthfully denied it, but accepted it as a warning shot. In Athens there were other surprises, *e.g.*, the hotel manager who begged me to give an audience to his friend. On the advice of my colleague, compared with whom I was, as we say in Yorkshire "No but a lad", I agreed to meet him in the center of the hotel lounge, where the possibilities of any funny stuff would be much reduced. After ordering tea, he said there was no point in beating about the bush. He understood that I was here to buy tobacco, and that, the Germans having pulled out, he was in control of the complete crop. I assured him that we were not at the moment buying anything but fun. We'd spent the morning on the Acropolis. He walked out not even waiting to finish the tea he had paid for. I discovered later that he had been watching the hotel register for a man with a name sounding like mine though differently spelled. On return to Belgrade we heard that the British Ministry were worried because my namesake was in fact a double agent supporting the British one moment and the Germans the next. On returning to the UK I read that he had been shot, though I never learned by whom.

I have never before told this story in public, so I hope you will forgive me for being carried away by it. I should add that we bought every bit of magnesite and chrome we could lay our hands on in Greece and were immensely impressed by the Anglophile Greek with whom, on the advice of the Ambassador we made our deal. We told him that the British Government would pay cash into his Athens bank for every ton loaded on a ship in Greece, regardless of whether it was sunk by enemy action or not. We both stuck to our bargain though the material got no further than North Africa until, much later in the war, the Mediterranean was reopened to shipping.

Many other coincidences floated into my mind. I too went to Berlin, but as a student, and was confronted, if not arrested, by the police. The cause? I had to complete in quadruplicate my entry permit. The opening columns I knew, or guessed: nahme, vornahme, gëburtstag: obviously name, christian name and birthday. The next "Ledig oder Veheiratet", fooled me completely. Actually it means "single or married". Thinking it must mean profession I wrote "student".

The police officer was furious: "Das können sie nicht schreiben" he said. But I had, and eventually he allowed me to leave.

Turning at last to HENRY MARION HOWE I find a similar Empathy. I've never used that word before so I hope I understand it. I can, for example, fully appreciate his excitement in being involved in the first 37 heats made in the USA on an openhearth furnace. I'm sure Howe was watching the hearth as closely as I would if only to know when to start running. I learned too that it was through his work on the Bessemer process that he met his wife. I didn't meet mine (also an American) that way but we did have a Bessemer honeymoon—surely a unique happening—during which I visited plants in such spots as Chicago and Pittsburgh, reporting each evening to my wife on my experiences and she on hers.

My final cross reference must be to the Bessemer Medal awarded to H.M.H. in 1895, seventy-one years before I got mine. Having since been a member of the selection committee I can say, without risk of rebuttal, that he must have stood head and shoulders above the crowd to have been elected at that distance. Four years later they gave it to Queen Victoria.

Having read several of the more recent Howe Memorial lectures, I hesitated before putting forward a subject as superficially frivolous as "Men and Magnesia". Before you even consider the pharmaceutical implications, I would state that I am not going to talk about toothpaste, though that was the route by which sea-water magnesia arrived, leave alone indigestion or constipation. This in spite of the fact that I once won (but was unable to accept) a Salter's fellowship financed by the pharmaceutical industry. I was prepared to argue then that the reactions of magnesia with water (see later) were as vital to the pharmacist as to the brickmaker.

No, I chose the title because, throughout my working life, I have been vitally interested in, and concerned with, magnesia, and been increasingly convinced that the way plants and processes function depends even more on men than materials. Silk purses (at least artificial ones) can, as Arthur D. Little demonstrated, be made out of sows ears, and equally what my German colleagues describe as a "richtige schweinerxei" can be produced even from top grade magnesia. With a melting point of about 2800°C (5072°F), and an almost unique resistance to attack by iron oxide, magnesia might fairly be described as God's gift to steelmaking. I hope now to show by a few selected case studies that men and magnesia can be friends or enemies. As one of my Energy friends keeps saying, it is a question of ATTITUDE, ATTITUDE AND ATTITUDE. But before starting I will make a final quote from Jim Austin:

"ALL HONOR TO HENRY MARION HOWE".

SINTERING IS A CHEMICAL PHENOMENON?

In 1928, only a few weeks after I started research on the lining of induction furnaces (Fig. 1) I was asked by my scholarship sponsors (Metropolitan Vickers) to proceed at once to a cable company in London to investigate the failure *on the first heat* of their newly installed MV furnace. I was greeted on arrival by what I took to be a cynical remark: "So you are the Basifrax expert". Basifrax was the name given by a Canadian company to their high lime dead-burned magnesite, which had already proved its value as an induction furnace lining. Later I noticed on the manager's desk a telegram that read "We are sending our Basifrax expert immediately". In the discussion that followed I was told that when power was applied to the furnace both the contents and the steel liner (see Fig. 2) heated up in the normal manner, but that when the liner softened and slid down into the bath of molten metal it was followed by the lining itself! It had not sintered to form in the traditional manner a smooth hard surface. I took a sample of the lining material and returned to the Refractories Department at the University of Sheffield—promising to report back as quickly as possible.

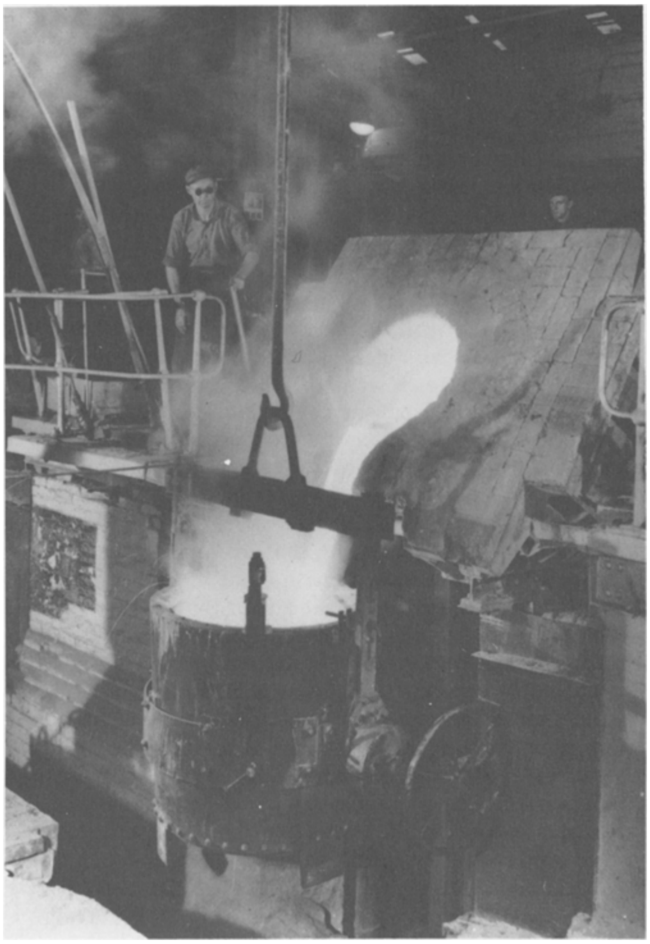
Fig. 1—Induction furnace tapping steel into ladle.

Fortunately I had at the laboratory a small sample of Basifrax that was alleged to have behaved satisfactorily elsewhere. I compared the grain size distribution of this with that of the material that had failed:

IMM Mesh	on 20	20-60	60-120	through 120
Basifrax—satisfactory	20	35	20	25
Basifrax—failed	35	50	10	5

Any refractory technologist looking at this data today would say that the trouble was undoubtedly due to insufficient "fines". In 1928 this was just a possibility. I decided therefore to fill three magnesite crucibles with:

Crucible 1	Basifrax as used in previous installations
Crucible 2	Basifrax from the London fiasco
Crucible 3	ditto but with a much increased through 120 fraction.

After heating the crucibles to 1500°C and cooling I examined the contents. Crucible 1 contained a hard cylinder but the magnesite in Crucible 2 could be poured out like dry sand. Crucible 3—the re-graded material—contained a cylinder as strong as that in Crucible 1.

Returning forthwith to London I was received by the top management, who were sitting round a large green baize covered table in what I took to be the boardroom. Asked what I had found out I produced some standard sieves from a ruc-sac and proceeded to demonstrate the low content of "through 120 mesh" material in the lining that had failed. I then showed them the dramatic effects observed in the crucible test. The chairman was not impressed, insisting that sintering was a chemical not a physical phenomenon. I agreed that chemistry played its part, but added that all I thought was needed at the moment was a grading similar to that used by previous clients. I asked him whether anyone in London could grind to specification. He said he didn't know, and implied he didn't want to know. Refusing to be put off I asked for a copy of the London telephone directory and found in the yellow pages a firm called Central Pulverising. I got them on the phone and asked if they could prepare the grading I wanted from the stock available. When they said they could I said I would get a cab and bring the material round so that they could grind it immediately. They replied that this would not be possible as their grinding procedures were secret. They would however collect the magnesite and return it regraded in a day or so. This they did and the grading was I found, to my surprise and joy, spot-on, or, since it was magnesite 'dead' right.

Then followed my first 'trial by fire'. The regraded material was rammed in position before my eyes and the power switched on. The next hour was traumatic. Would it bring a triumph of technology or another abysmal failure. I was comforted by the advice of my boss George McKerrow—Liaison Director of Research at MV—who had said: "If it succeeds just say 'Was that all you wanted?'. If it failed 'Now isn't that interesting'." Fortunately for me and for my confidence in the numerous trials that followed the lining was (to quote Frank Bagnall, a pioneer in induction furnace technology) as 'smooth as a baby's bottom'.

UNBURNED BRICKS—A POSSIBLE ALTERNATIVE TO RAMMED LININGS IN INDUCTION FURNACES?

The first experiments made with brick linings arose from our desire to compare different materials side by side under the same working conditions. The intention was to line the 3 cwt pilot furnace at MV with five rings of tongued and grooved bricks, having the same internal contour as the rammed lining. Each ring would consist of two bricks of, say, magnesite, two of chrome and two of 50-50 chrome-magnesite, at that time (1928) a novel refractory. The original intention was to use fired brick, but to save time a trial was made with gum bonded unfired bricks that proved

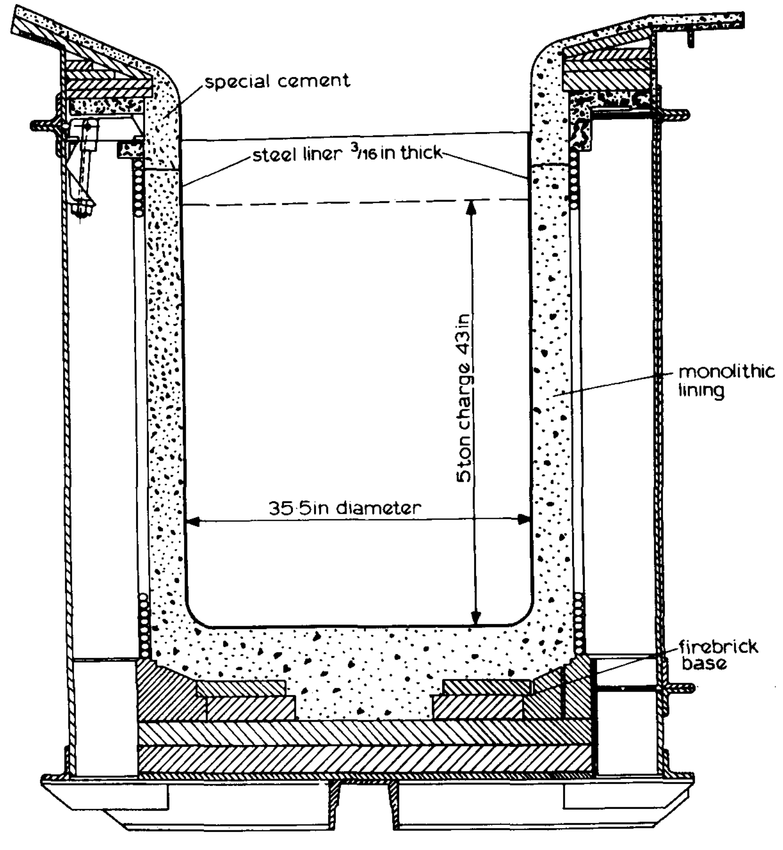

Fig. 2—Use of steel liner for forming monolithic lining in induction furnace.

quite satisfactory—no crumbling or spalling taking place. A scrap charge was melted and tapped. A bar iron charge was then melted and held for about an hour. This was then tapped and the lining examined and measured. The results with this particular trial were dramatic: the magnesite and chrome-magnesite bricks showed a loss of about one-quarter inch; the chrome bricks had virtually disappeared. Analysis of the steel showed a chromium pick-up approximately equal to the weight of chromium in the brick! Conclusion: unfired magnesite or chrome-magnesite can be used to line induction furnaces, but chrome brick disappear, mostly into the steel.

Encouraged by this result I asked Frank Bagnall of S. Fox & Co. Ltd. whether he would be game to try unfired gum bonded bricks in his 5 cwt furnace. He agreed to do so, and I arranged for the best available Greek magnesite at Pickford Holland & Co. Ltd. to be crushed, graded and rammed into bricks with only a gum bond to give dry strength. The only exception was with the two top courses which were fired to ensure adequate strength at a level where the working temperature is relatively low. I insisted on installing the lining (Fig. 3) myself, being painfully aware that any lack of concentricity with the steel coil could lead to a nasty accident; molten steel, water and 2000 volts being bad mixers! The bricks were set on a 2 inch layer of magnesite, and the 1/2 inch gap between bricks and coil carefully rammed with magnesite powder. The first melt was accompanied by a flame above the bath never seen before by anyone concerned. It was agreed that this might be due to gases evolved from gum in the bricks. The next heat was similar but the flame larger. It was bigger still on the third, and Frank Bagnall decided to run the fourth himself. This time the furnace really looked like a Bessemer and we were all pretty scared. FB showed his feelings by marching up and down the stage muttering "Bloody hell, bloody hell." After he had tapped the furnace he said: "It's no good Jack we'll have to take the furnace off." When it was sufficiently cool the lining was removed to reveal about 4 inches of water in the box below! I asked Frank whether the flame was due to my bricks. "No", he replied, "The bloody fitter didn't tighten the jubilee clip on the water connection to the coil". "Does that mean we do it again", I asked, to which he replied, "Yes". I doubt whether one manager in a thousand would, after five hours of tension—including major physical risk, have made that decision. It would have been so much easier to say: "It is unlikely to have been your bricks but we can't possibly take that risk again".

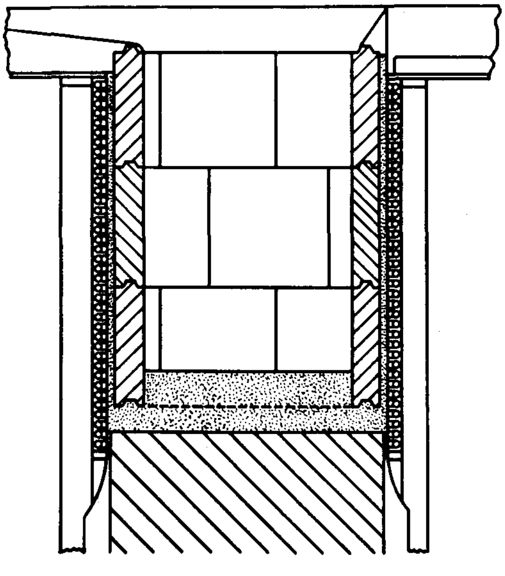
Fig. 3—Assembly of brick lining in 1/4 ton induction furnace.

At the time of the first MV trials on basic linings, trouble—generally a strike to the coil—was often experienced after as few as six heats. At the time of the unfired magnesite brick trial, sixty heats was considered excellent. Within a week or so of the "Bessemer run" a new lining of the same type was installed and did 115 melts, of which 86 were of the notoriously corrosive high manganese steel. This record was not exceeded at Fox's for at least a decade. The rammed linings were not however replaced by brick linings because we agreed that the slightest carelessness in construction could lead to a breakout of molten metal through the joints. Brick linings are however extensively used today, *e.g.*, in large foundry induction furnaces, where the lining thickness is much greater and an inner brick lining can be backed by a monolithic lining as thick as a new lining on a smaller furnace. We had confirmed one important assumption, *viz.*, that purity is a major factor in slag resistance.

DEAD-BURNED MANGESITE DOESN'T HYDRATE?

In the Spring of 1931 I was fortunate in being awarded a Robert Blair Fellowship to continue my studies on refractory materials at the Kaiser Wilhelm Institut für Silikatforschung in Berlin. I had given considerable thought to the research I should pursue and had naively decided to determine, in the year available, the effect of such impurities as silica, alumina, iron oxide and lime, on the hot strength of magnesite bricks. Looking back I can see that I had given myself at least ten man-years work, but that was not to be, for shortly before I left for Berlin the telephone in the Refractories Department rang and I answered it. An obviously disturbed voice asked for Mr. W. J. Rees, the head of our department. I explained that neither he, nor his righthand man, W. Hugill, was available, to which he replied, "Well, who are you?". I said I was just a student, and he asked: "Do you know anything about magnesite?". Having replied "Yes", he told me that he was in terrible trouble with his magnesite kilns, and begged me to come immediately. I went, and was horrified by what I saw (Fig. 4). Instead of the typical 10 pct rejection of fired brick he had 100 pct, and on five kilns! He was trying to meet the deadline for special shapes for a copper converter in South Africa and was so worried and exhausted by his failure that he was trying to sleep on a campbed beside the kilns so that he could *hear* what was happening.

After a brief discussion I decided literally to "have a bash"—still a basic part of my research philosophy. As I often say to students faced with a new problem "It doesn't matter so much what you do (first), but for God's sake do something". I asked whether he had any

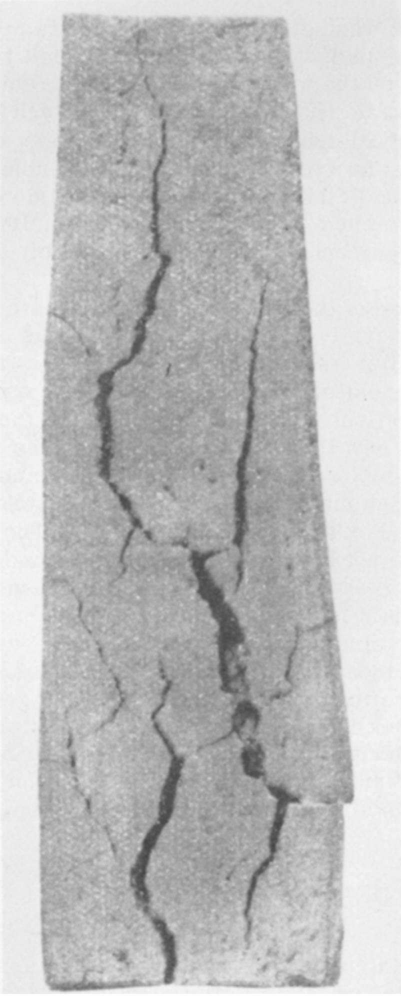

Fig. 4—Magnesite block for copper converter—cracked in firing.

blocks that had been dried but not fired and he pointed to a stack of them. With his permission I dropped one across a railway line to see whether the fracture showed any sign of lamination. I expected it to break like a carrot but instead it exploded—the six sides falling off to leave a sort of rugby (or American) football. I tried another and the same happened. He was as amazed as I was. "I can tell you one thing right away", I said, "those bricks were ruined before they went into the kiln". All the firing did was to release stresses, presumably built-up in the drier. Then, or very soon after, I wondered whether magnesia could slake like quicklime, only more slowly. If so could this result in growth and consequent stress. The probability that the catastropic failure was something to do with hydration was increased by the fact that he was using an unusually wet batch—water appearing on the surface as the final hand molding was completed by slicking.

Being both extremely sorry for the man, and intrigued by the discovery of a new area of ignorance—did magnesia hydrate, and if so what were the factors controlling it—I decided to make it the subject of my work in Berlin. To avoid delays on arrival I took with me a sample of induction furnace lining from S. Fox & Co. Ltd. It was in fact contaminated by slag, which led to my throwing doubt on the analysis (which showed substantial amounts of chromium and nickel) made by a woman analyst at the laboratory. When, after a repeat analysis—the truth emerged—she quite fairly took her revenge by turning on a fire safety shower that soaked me and, not having been used for years, refused to shut off.

On first reporting to Professor Eitel, the head of the Institute, I was told that I would be assisted in my studies by Dr. Woldemar Weyl. His main field both then, and later at Pennsylvania State University, being glass, indeed he soon became one, if not the, leading authority on colored glass. He was nevertheless interested in my magnesite story and recommended that I use an extremely sensitive piece of apparatus called a Tensi-eudiometer (Fig. 5). This was made of fused silica and glass, the relevant parts being a fused silica combustion tube that contained the sample and could be inserted into a furnace, and a mercury manometer. Briefly the idea was that if, say, a sample of magnesium hydroxide (brucite) was placed in the tube and the whole system evacuated, then any evolution of gas that occurred on heating up could be observed, and the corresponding temperature and amount of gas noted. After only a few weeks' work I knew that water was evolved from the hydroxide ($Mg(OH)_2$) at about 350°C, but not from the carbonate ($MgCO_3$) until over 400 to 500°C. A sample of basic carbonate produced by precipitation showed gas evolution over a wide range from 100 to 600°C. This was a good start, since it meant that I could take samples of dead-burned magnesite exposed to water for given times at various temperatures, and determine at least roughly the amounts of the compounds formed. For many years magnesite brickmakers had 'soured' the batch, *i.e.*,left it standing for days after mixing with water before pressing into bricks. This not only slaked any lime present—a common impurity in Austrian magnesite, which in the raw state is a mixture of magnesite and dolomite ($MgCO_3 \cdot CaCO_3$)—but would, I could now see, also hydrate any reactive magnesia that might be present due to localized under-calcination.

Come Christmas I decided to take a short break. I joined some Czechs I had known in England for a ski trip in the Riesengebirge. Incidentally the village we stayed in was, I swear, called Mexico, though I have never met anyone since who had ever heard of it. After three days of skiing on snow 2 feet thick it rained and the surface became a quarter inch of ice. Skiing being virtually impossible, and at the best highly dangerous, I decided I would go to the Tyrol, visiting on the way the wellknown Austrian magnesite works at Radenthein. Not having given notice of my arrival I was not unnaturally viewed with suspicion. It was soon obvious that I knew far too much about their product. They therefore offered to ring local ski resorts, and informed me quite soon that there was good snow at Mallnitz and that they would be glad to drive me to the next train. Before I left I had however gleaned three valuable bits of information:

1) They also had trouble with kiln cracking—losing about 10 pct,

2) The cracking was worse with big shapes which were dried slowly on the shed floor,

3) Kiln cracking was worse in summer than in winter.

I was already convinced that hydration was the bogey and this would clearly be worse for big shapes, but why the increased trouble in the summer? My guess was that kiln temperatures were, within the limits of pyrometry and control, identical, but the drying conditions would be very different between summer and winter if the shapes were merely dried on a floor. Could hydration increase markedly above some critical temperature? This was the question I had to answer quickly on my return to Berlin. I decided to build immediately a simple apparatus (Fig. 6) that would enable me to measure the expansion of a cylindrical testpiece when dried with various time-temperature schedules. I soon found (Fig. 7) that the expansion increased dramatically at temperatures over 60 to 70°C.

Now I was really getting somewhere, though, as I added in my 1932 paper to the British Ceramic Society, on the "Drying of Magnesite Bricks", I was surprised by the complexity of what at first appeared to be a simple problem, and suggested that this was why souring and drying had so long remained a mystery. The application of the work was however clear: magnesite should be really dead-burned, *i.e.* hard fired, the amount of fines in the batch (and thus the surface area exposed to reaction with water) should be minimized, and *most important* the water should be driven off *before* the temperature at which rapid reaction occurs is reached. That one manufacturer at least applied this research beneficially is proved by the fact that when I got married he sent me a beautiful radio with his thanks for the money saved as a result of applying my research on hydration. It was not the man who started it all but I gathered from my subsequent travels that the then typical wastage rate on firing of 10 pct was soon reduced to nearer 2 pct. Furthermore by 1943 expansion type measurements had been applied to other urgent problems (see Fig. 8). This shows the much greater expansion that can occur with "basic"—the name given at that time to the calcined dolomite used for ramming and fettling open hearth furnace and arc furnace hearths. Here expansions of over 5 pct linear were recorded, and over 2 pct for dead-burned sea-water magnesia to which 10 pct caustic magnesite had been added.

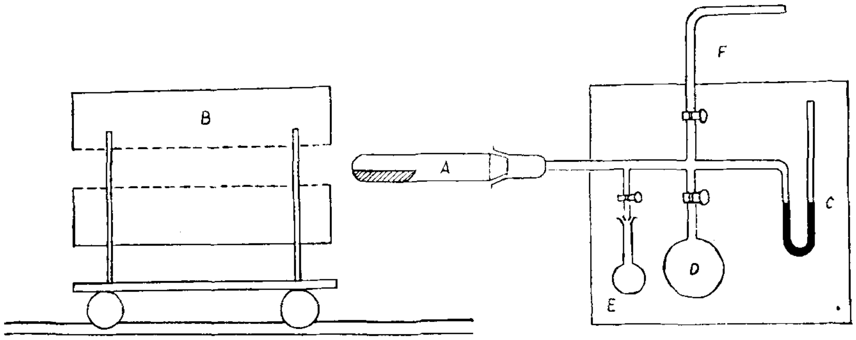

Fig. 5—Tensi-eudiometer used in hydration studies.

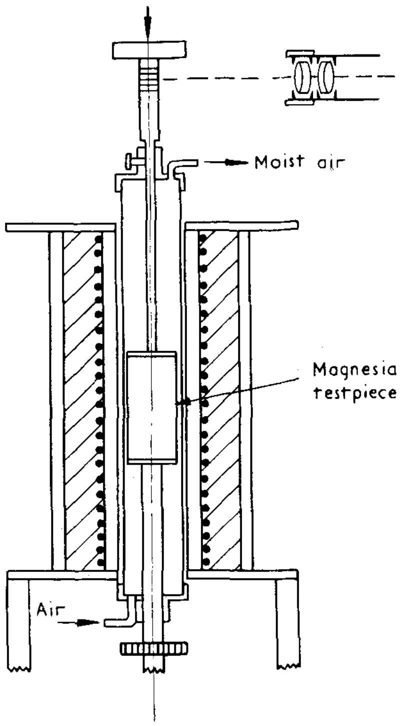

Fig. 6—Hydration-expansion apparatus (Mark I).

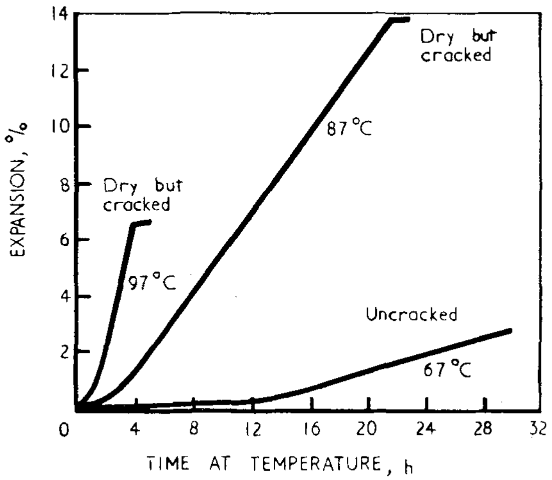

Fig. 7—Hydration-expansion as function of temperature. Effect of heat input to furnace. Specimen semi-enclosed.

THE MAXIMUM DENSITY OF PURE MAGNESIA IS 3.65?

Having confirmed in Berlin my feeling that refractories research was well worth pursuing, I applied to the Commonwealth Fund for one of their luxurious Fellowships. My professor told me that I hadn't a chance—no one from the University of Sheffield had ever got one and they certainly would not award one to a man studying such a humble subject as refractories. I reminded him that he had for the same reasons refused to support me for a Robert Blair Fellowship, which I had got, but he was not impressed, and I applied again with MV backing and little else. I knew (if he didn't) that a novel subject would be a help if only because I could not then be compared with the best physicists from Oxbridge or the like. Anyway I got it, and went, at the invitation of Professor C. W. Parmelee, to the Department of Ceramics at the University of Illinois. It was a happy choice, not only because it was a good place to work, but because it led indirectly to my acquiring a delightful American wife.

I decided to continue my specialization in magnesia, and to study next the burning of magnesite bricks. I wrote three papers on the subject for the British Ceramic Society, and remember with amusement that an American abstracter listed them (most improperly) as the "*Firing* of Magnesite Bricks": he was apparently aware that magnesia, unlike magnesium, would not "burn"!

A major consideration in the firing of magnesite had always been the substantial shrinkage that occurs even with dead-burned material. To understand this fully one must know how much shrinkage occurs within the grain and how much between the grains. The former demands a knowledge of the density of a perfect crystal of pure magnesia. My study of the literature showed a wide range of values—the one that worried me most being that given in the International Critical Tables, and attributed to a famous French scientist, H. Moissan. He had fired his sample in an electric crucible furnace, and gave a figure of 3.654. Other workers, *e.g.* Sjorgen, gave much higher figures, such as 3.90 for "natural periclase". The figure I repeatedly got for fused magnesia of reasonable purity was 3.58. Wondering whether there was something wrong with my pycnometry I wrote to five world authorities to ask for details of their procedure. They proved similar to my own. Lost as to what to do next I gave a lecture in the Eastern States (I can't remember where) after which a member of the audience came up and said "What do you think this is?" It was a transparent semi-cube about 1 in. by 1 in. by 1/2 in. (Fig. 9). I replied that it looked like glass. "It's what you have been talking about", he said, "MAGNESIA". I was astounded, as were several more phlegmatic people to whom I showed it. Now it is old hat, large pieces of transparent polycrystalline magnesia made by hot pressing being available for those with enough money to buy them. To me however this crystal was a God given op-

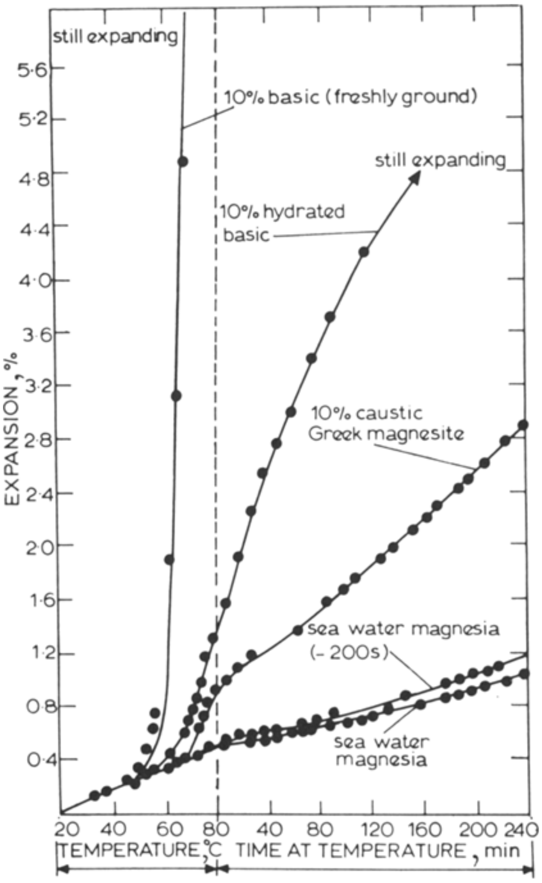

Fig. 8—Influence of additions on the hydration-expansion of magnesia at 90°C.

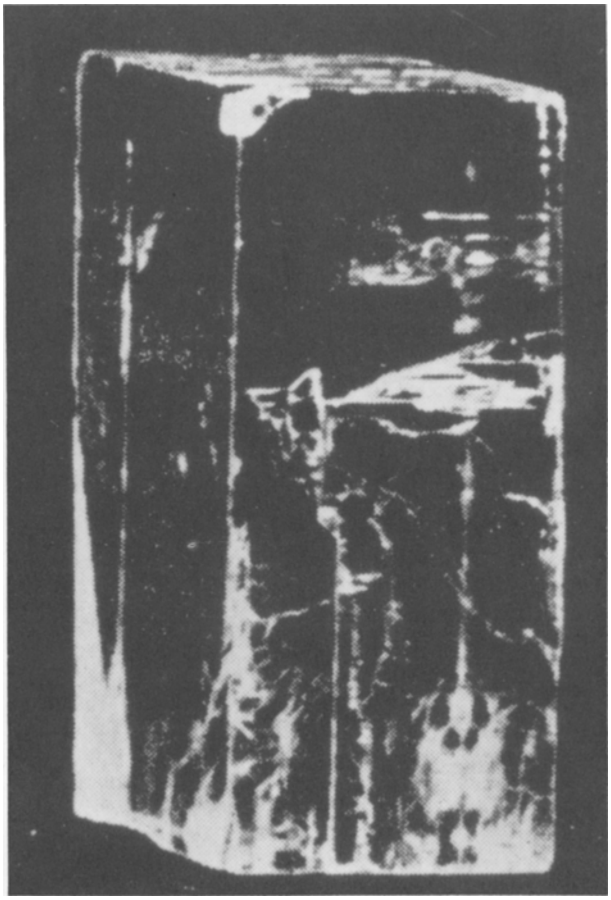

Fig. 9—Magnesia crystal formed in fusion furnace—approximately 90 pct of actual size.

portunity, for I could employ it to determine density using the glass stopper (weighed dry and then submerged in water) technique that has bored schoolboys for generations. I arranged to do such a test with all the finesse possible, even weighing the cotton thread! Before I could start, however, I had to acquire and, hopefully, retain the crystal. My usual luck (or guile) prevailed. The gentleman told me that he had been fusing magnesia for about ten years in a small furnace and that on wrecking it had found half a dozen such crystals in the cooler parts—produced, I imagine, by deposition from the vapor phase. I told him that if he would give me one I would do an X-ray Laue pattern for him and see whether it indicated a perfect cubic crystal of the simple sodium chloride type. Having taken all the precautions I could think of, I calculated what a crystal of this dry weight should weigh when immersed in pure water at the temperature used, and put the corresponding weights on one pan of the balance. I then raised the beaker of water till the crystal was fully immersed, and when all was steady turned the knob on the balance. Nothing happened, and I thought (and maybe said) ''Damn—it's sticking''. However when I added or removed a small weight from the pan the pointer moved and I found that the specific gravity (or density) of the crystal was 3.583—the sort of figure I had repeatedly obtained for the fused product. Higher figures, *e.g.* the 3.65 and 3.56 obtained for Austrian magnesites were apparently due to their containing 8 and 4 pct Fe_2O_3 respectively. Figures lower than 3.58 were due to the presence of micropores, which are very difficult to get rid of in pure (low iron) magnesites, such as the cryptocrystalline types found in Greece and Turkey.

It was only after doing this test that it occurred to me that an approximate figure could be got from the lattice spacing (Fig. 10) of magnesia as determined by X-rays.

The density ρ is given by the equation:

$$\rho = \frac{4M}{d^3N}$$

where M is the molecular weight of magnesia, N Avagadro's number (defined as the number of molecules in a gram molecule), and d the length of the unit cell edge. Using the value for d then quoted of 4.20Å, I came up with a calculated density of 3.59. Budworth in 1967 gave a unit cell value determined at Harwell of d = 4.211325Å and a corresponding density of 3.5847. This for the practical man is approximately 3.58, a value now used by most workers in the field, though the ubiquitous 3.65 still appears in the literature, which shows either that few people challenge men of Moissan's standing, or that textbooks tend to copy mistakes from one another.

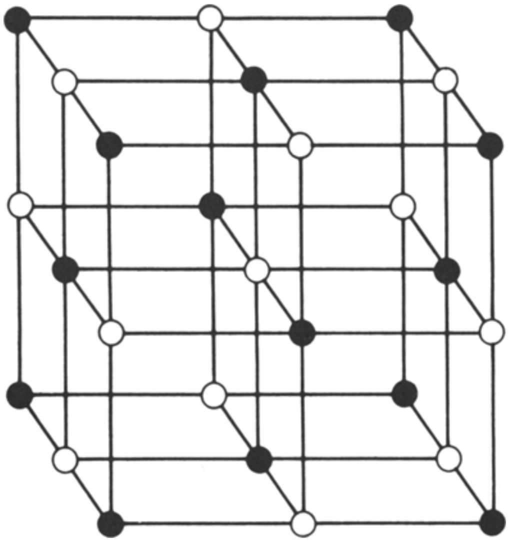

Fig. 10—Simple cubic lattice of magnesia showing location of magnesium and oxygen ions.

Incidentally quite eminent people have from time to time referred to the different crystal forms of MgO. That the difference is only one of size and perfection is well illustrated (Fig. 11) by an electron microscope of the MgO formed when magnesium metal is burned!

MAGNESIA CANNOT BE EXTRACTED ECONOMICALLY FROM DOLOMITE?

The Steetley Company being by far the largest producers of dolomite refractories in the United Kingdom, have, like so many others, long considered the possibility of separating the magnesia from this compound ($CaMg(CO_3)_2$). The obvious way is to calcine it lightly to give CaO and MgO or after hydration $Ca(OH)_2$ and $Mg(OH)_2$, and make use of the differences in physical or chemical properties of these compounds to effect a separation. This sounds easy but proved in fact to be very difficult to do on a large scale at an economic price. The first real breakthrough came in 1936. An American by the name of H. H. Chesny, who had been working with Marine Chemicals in California contacted

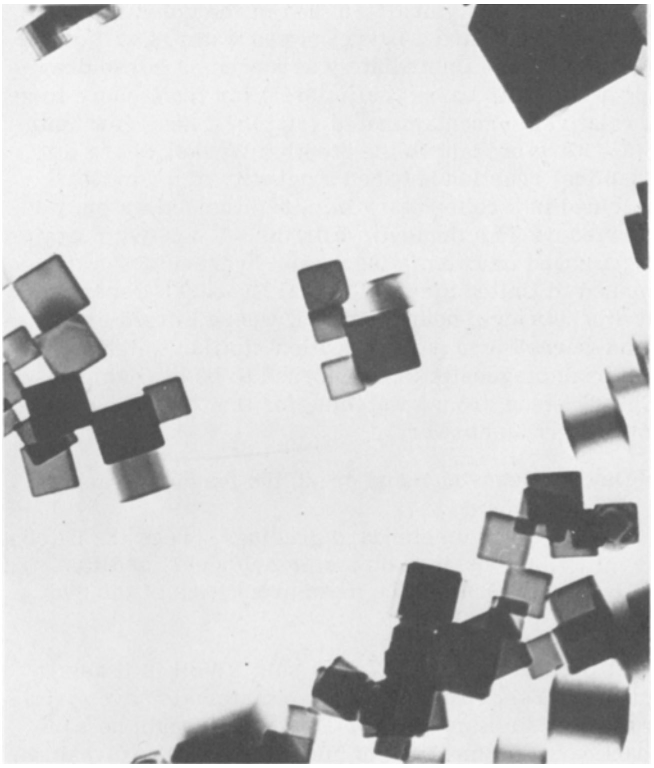

Fig. 11—Cubic magnesia crystals formed by burning of magnesium metal. Electron micrograph, magnification 15,000 times.

Mr. N. M. Peech of Steetley to say that he had invented a novel separation process that would produce magnesia at a price competitive with that from natural magnesite. Mr. Peech suggested that Alan Bradley (head of research at Steetley) and I go to London to meet Chesny and see whether he had really got something. I should add that Mr. Peech and I had already made an abortive trip to Germany in pursuit of a somewhat similar claim and had so little confidence in Chesny's story that I said to Bradley that it would be a trip to London—a relative treat at that time—but probably little else.

We proceeded to London and were greeted in the lounge of the Waldorf Hotel by a man who immediately inspired confidence. He explained that he proposed to react slaked calcined dolomite with sea water, obtaining as a precipitate one molecule of $Mg(OH)_2$ from the sea for every one he got from the dolomite, *i.e.* a double yield. He showed us his detailed analysis of the problem, and made further calculations on site with amazing facility. I suppose it was the first time I had seen a first-class chemical engineer in action, and was duly impressed. I could not however resist playing my trump card. It is well-known, I said, that many people have tried to make good silica bricks out of clean sand and failed, yet chemically the material is very similar to the quartzites normally used. How, I asked, do you know that good magnesite bricks can be made from your magnesium hydroxide? His reply out-trumped my trump: "That, he said, is your problem, all I am doing is to offer you magnesium hydroxide at an economic price."

Asked what he needed to demonstrate the new process he was ready with all the answers. Your Coxhoe dolomite plant he said is near the N. E. Coast. If you will get me typical samples of the dolomite you produce there, and a barrel of sea water, I will do the rest. This was immediately arranged—a horse drawing a cart with barrel sufficiently far from shore to get a relatively uncontaminated sample. These raw materials were brought to the Steetley Works, where the chemical reaction between a quantity of dolomite, calcined in a baby rotary kiln, and the sea water, was carried out in a domestic bath tub. A jam-jar containing a pound or two of magnesium hydroxide was then rushed to United Steel's Central Research Department at Stocksbridge, near Sheffield, where I dried and dead-burned it in parallel with a similar weight of raw Austrian magnesite that happened to be available. Chesny stood around watching for the three days we took to get an answer.

Day 1	Dead-burn and crush the magnesite and hydroxide
Day 2	Make up standard gradings, press the batches as one-inch diameter cylinders, and fire
Day 3	Determine the main properties of the two products.

At the end of day 3, Chesny said "Well is it any good?". Yes, I replied, the two materials have given strangely similar results. That was enough: he said thank you and (unknown to me) was on the next train to London to file his master patent on the simultaneous extraction of magnesia from dolomite and sea water.

Mr. Peech decided to act immediately. He arranged a meeting of all concerned to be held at Steetley to decide whether it was worth building a plant. Captain Hilton, managing director of United Steel at that time, agreed to take the chair. I was much impressed by his approach. I remember, for example, that when costs were quoted, say 4,105 Pounds for a "what-not", he just wrote down 4,000 Pounds. Having come from Metropolitan Vickers, he asked telling questions about the cost of the electrical equipment, which Chesny answered to his satisfaction. After a number of us had given opinions on the technology and the patent position, Captain Hilton said: "Well, I'm happy—I suggest you form a Company to exploit the process." The first plant was small, but was already operating at Hartlepool when the war started and our conventional sources of magnesite—notably Austrian and Greek—were cut off. It was soon taken over by the Government as a key plant for refractories (and therefore steel) but also as a potential source of magnesium for airplanes and incendiary bombs. It was however later decided to produce this lightly calcined (not dead-burned) magnesia at a separate plant at Harrington on the West Coast of Cumberland.

Today sea water magnesia is produced in many countries, notably the U. K. (see Figs. 12, 13, 14), the U.S.A. and Japan. There have been many improvements, particularly in the purity of the product. Thus the MgO may now be 98 to 99 pct and the CaO 0.6 pct compared with nearer 6.0 pct in the early days, with SiO_2 only 0.5 pct. These later figures are for Sardinian sea water magnesia produced by Steetley in cooperation with the Italians, where very pure limestone is employed instead of dolomite, yielding magnesia from the sea alone. The present Steetley capacity amounts to about 250,000 tons per annum. Of this approximately 40 pct is exported.

The latest developments include the production of specialized co-clinkers made by blending and burning together such mixtures as magnesia and chrome ore, magnesia and alumina and magnesia and lime. Furthermore a new production process has recently been perfected. This greatly reduces the water content of the precipitated magnesium hydroxide and, therefore, the fuel required to produce high density, large-grained, magnesia.

Fig. 12—Aerial view of sea water magnesia plant at Hartlepool. (Courtesy of Steetley Company).

STEELTIGHTNESS OF AN INDUCTION FURNACE LINING DEPENDS ON MINIMIZING SHRINKAGE AT STEELMAKING TEMPERATURES

My last case history is also my favorite. I used it in a Presidential Address to the Iron and Steel Institute in 1968 to summarize my philosophy of research and the helpfulness of Koestler's theory of *Bisociation* described in his 1964 book on the "Art of Creation" (Hutchinson, London). In this he suggests that "Discovery often means simply the uncovering of something which has always been there but was hidden from the eye by the blinkers of habit." He suggests that the bisociative act connects previously unconnected matrices of experience, and that the same mechanism operates in the worlds of humor, art and science. Most jokes he says are based on surprising bisociations. Thus (my example): the Titanic story:

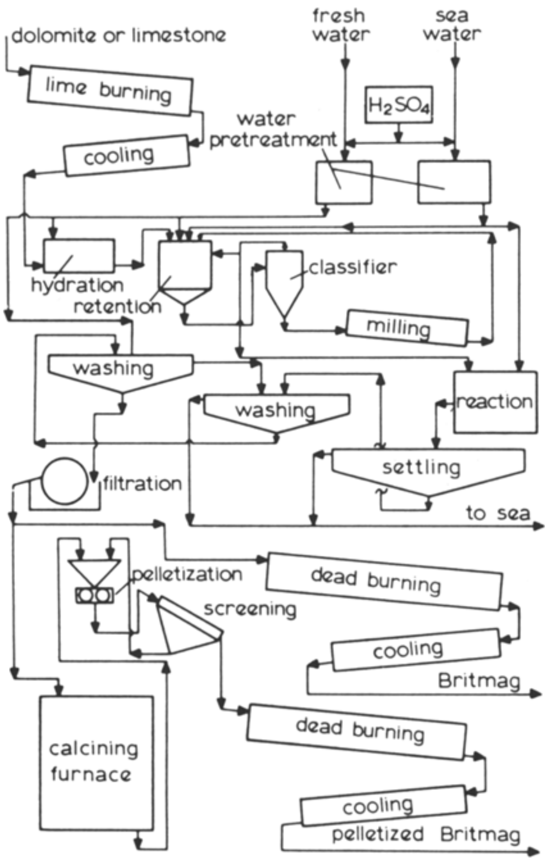

Fig. 13—Flow sheet for sea water magnesia process at Hartlepool. (Courtesy of Steetley Company).

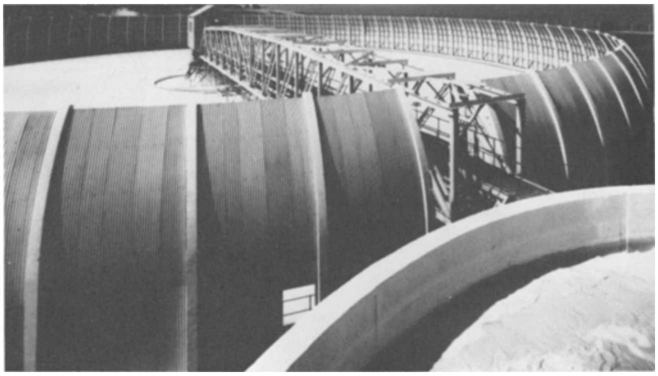

Fig. 14—Settling tank for separating magnesium hydroxide by precipitation. (Courtesy of Steetley Company).

Drunk sitting at the bar as the crash occurs: "I know I asked for ice, but this is ridiculous." Matrix 1: the collision of the ship with an iceberg; Matrix 2: the use of ice in cocktails. Even simpler is the interaction of physical and chemical matrices in the Romanian riddle: "How do the Oltenians make gunpowder?" Answer: "By grinding up guns, of course."

For bisociation to occur there must be some sort of "trigger" action. Koestler suggests this can be verbal (see below), visual or even chemical: "a tumblerful of gin or an amphetamine tablet".

Quite early in the war we were faced by repeated explosions in our 2 ton induction furnace at S. Fox & Co. Ltd., caused by the penetration of metal through the linings. The trouble appeared to be associated with a change in the magnesia used to make the proprietary brand of lining. The prewar lining, a 50-50 mixture of Greek and Austrian magnesite plus unspecified bonds, had run out and a switch had been made to sea water magnesia. We had foreseen the risk and arranged a trial of a sea water magnesia lining in a 5 cwt furnace well before the war broke out. This had been completely successful.

Conditions in 2 ton furnaces were apparently more severe and we lost eleven coils in six weeks and narrowly avoided killing a melter, molten metal having been blown over a man's shoulder with enough force to remove the glass dials from the instruments on the control panel! For six weeks the eight members of the Refractories Section devoted their entire efforts, using of course the refractory man's 'code', to solving the problem of preventing the molten metal reaching the coil. The special steels made in this furnace were desperately needed for aircraft, and emotion was running high.

Progress was, however, completely blocked until one day the melter casually remarked: "It's funny but I'm not happy unless they crack". This "trigger" statement from the melter's craft matrix formed a complete paradox with our own ideas and jerked me out of my refractory code. To use one of Koestler's favorite phrases, I had to "reculer pour mieux sauter". An hour or so later it occurred to me that there could only be one explanation of the paradox, namely that the small cracks we produced went through to the coil but the bigger ones existing previously only went part-way (Fig. 15). Shortly afterwards I felt intuitively that we should stop looking at the high-temperature properties of the linings and consider what was going on at temperatures intermediate between those of the hot face and the coil. Numerous tests had already shown that the shrinkage of our sea water magnesia linings, with their low bond content, was far less at 1600°C than that of the previously satisfactory material. A comparison of shrinkage over the whole range 0 through 1600°C showed a completely unexpected "Eureka" result (Fig. 16), namely a substantial positive expansion of the pre-war material in the temperature range 1100 through 1300°C.

We had discovered that steeltightness in basic in-

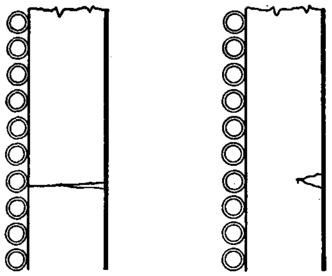

Fig. 15—Possible explanation of paradox that small cracks (new type lining for induction furnace) can be more dangerous than large cracks (old type).

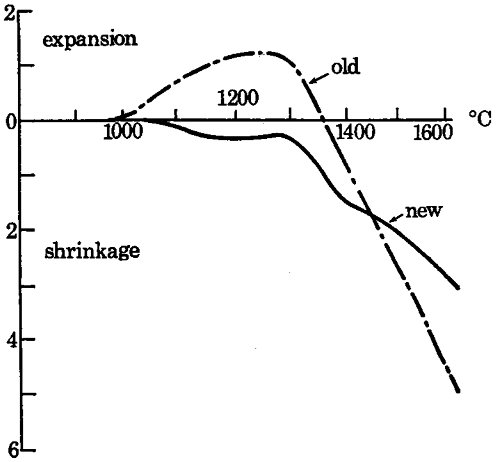

Fig. 16—Paradox explained: old type lining shows positive expansion in intermediate zone (1000-1300°C).

duction furnace linings depended not (as our code suggested) on the minimization of surface shrinkage but on the creation of a positive expansion in some intermediate temperature zone. A secondary but equally convincing realization was that as such linings wore back the expansion zone moved back *pro rata* towards the coil, protecting it against penetration even when the lining was extremely thin. We appealed to the suppliers to tell us what bonds they had used and were told: X pct of sand, Y pct of boric acid, and Z pct of a third component. The last we were asked to keep secret, though subsequent tests showed it had in fact no effect on steeltightness. The rest, as one eminent scientist used to say, was just engineering.

Using our well developed though still inadequate refractories code we examined the testpieces, fired at various temperatures, using X-rays and a microscope. This immediately revealed the mechanism: on heating, the sand was dissolved by the boric acid, the liquid glass reacted with the magnesia grains to form a refractory coating of forsterite ($2MgO \cdot SiO_2$) which forced the grains apart, yielding the observed positive expansion. At a slightly higher temperature this coating melted, leaving the magnesia surrounded by large quantities of liquid, causing the high shrinkage observed at top temperature. Being wise after the event I remembered that in the early days (1928–1931) we had deliberately developed basic linings, *e.g.*, magnesite-zircon, for small furnaces with a positive expansion at top temperature but had abandoned them because of their markedly lower slag resistance.

Those of you who have followed the story may be saying to yourself that our "discovery" was obvious. If so, I am delighted, for according to Koestler the newer the discovery the more obvious it seems *after* it has been made. The same is certainly true of a good patent. Having understood the problem, the solution was easy; we merely added controlled amounts of sand and boric acid to our sea water magnesia in order to obtain a minimum of 1 1/2 pct expansion at 1150°C, ignoring the original code of low shrinkage at 1600°C. Statistical quality control was then applied, and strikes to the coil eliminated for at least a decade.

CONCLUSIONS AND RECOMMENDATIONS

If my message has gone home these will I trust be obvious. I shall not therefore take time or insult your intelligence by repeating them. I shall end as I did my presidential address to the Iron and Steel Institute by hoping that those of you who have still a lifetime of research, development, or production ahead of you will enjoy it as much as I have done, and be as fortunate in the facilities available to them and, *more important*, the people with whom they are privileged to work.