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Metallurgy

Magnola: A novel commercial process for the primary production of magnesium

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ABSTRACT

The demand for magnesium, the lightest of all structural metals, is expected to undergo sustained growth over the next 15 years. Magnola Metallurgy is positioning itself to become a major producer by the year 2000. A key element in its strategy is the new process technology which, since 1986, has been developed at the Noranda Technology Centre. This new technology will ensure that Magnola will be the lowest cost producer in the world.

Serpentine tailings, containing an average of 24% by weight magnesium, are leached with HCl, neutralized and filtered to produce a silica-iron residue and $MgCl_2$ brine. The brine is then purified before being sent to the spray fluidized bed drier to be partially dehydrated to $MgCl_2.xH_2O$ prills. The prills are then fed to the patented Super-Chlorinator where the $MgCl_2.xH_2O$ is completely dehydrated and any residual MgO is reacted to produce an oxide-free $MgCl_2$. The anhydrous $MgCl_2$ is then fed into the Alcan Multi Polar Cell to produce magnesium metal. The chlorine from the cell is used to generate HCl which is recycled for the leaching of serpentine at the front end of the plant.

Currently, Magnola is in the pilot demonstration phase of the project. At the time of publication, the construction of the pilot plant will be completed and the testing underway. This paper provides an overview of the Magnola process along with an update on the current progress of the pilot plant.

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received his B.Eng. degree in 1993 from McGill University and his Ph.D. in 1997 from McGill University in metallurgical engineering. He has been employed by the Noranda Technology Centre where he has been implicated in various fields, including the development of the HCl chlorination process for the Magnola Project, CHC formation and deportment in magnesium plants, magnesium electrolysis, and elimination of NO_x from concentrated sulphuric acid. He is currently employed by Magnola Metallurgy and is involved in the basic and detailed engineering for a commercial magnesium plant.

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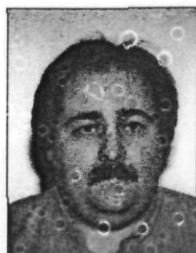
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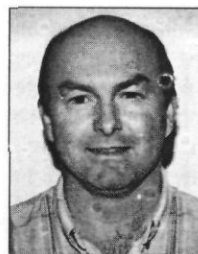
Thomas P. Walker
received a college diploma in electrotechnology from Vanier College in 1975. From 1975 to 1976, he worked as a site installer for a small design engineering company. From 1977 to 1980, he worked for Combustion Engineering Ltd. in quality engineering and as a project leader in research development. In 1980, he joined Noranda as a technology specialist in energy conservation. He later spent five years as a construction project manager before working on the Magnola process development project, and is credited with co-patenting the chlorination circuit for the current Magnola process.

Cesur Celik
received a B.Sc. in metallurgical engineering from Istanbul Technical University in 1974, and a Ph.D. in industrial metallurgy from the University of Birmingham, England. He worked at Cambridge University and the Seydisehir Aluminum Works in Turkey before emigrating to Canada in 1982. His first contact with Noranda was at Brunswick Mining and Smelting, with whom he carried out a variety of projects from a base at the University of New Brunswick. He joined Noranda Technology Centre in 1989 following four years as senior research scientist and group leader at Alcan's Arvida R&D Centre. He has since developed a substantial program in support of Noranda Aluminum and led key elements of the Magnola project, initially as the R&D program manager and later as operations manager of the Valleyfield pilot plant. Dr. Celik is currently employed as the manager for business project development at Noranda Advanced Materials leading the development of new electronic materials products.

Daniel Laroche
received a college diploma (DEC) in analytical chemistry, instrumentation specialty from CEGEP de l'Outaouais in Hull, Quebec in 1988. He has been working at the Noranda Technology Centre since June 1988 on various projects as a technologist. Most of his nine years with Noranda have been spent working on the Magnola project.

Michael M. Avedesian
received a B. Eng. degree from McGill University in 1969 and a Ph.D. in engineering from Cambridge in 1972. From 1972 to 1983, he worked for Noranda as project manager — engineering. He joined Domtar in 1983 as director, corporate research and manager of business development for its mineral sector. From 1990 to 1993, he was president and chief operating officer of the Institute of Magnesium Technology, and is currently chairman of the board. In 1993, Dr. Avedesian rejoined Noranda as director, magnesium, and is currently vice-president and general manager of Magnola Metallurgy Inc. Michael is a member of a number of professional associations, including the Ordre des Ingénieurs du Québec, the Professional Engineers of Ontario, and the American Institute of Chemical Engineers.

Magnola Metallurgy Inc.

Magnola Metallurgy Inc., is a recently formed Canadian company, currently co-owned by four strategic partners:

Noranda Metallurgy Inc. 52%
Aisin Group of Companies (Aisin Seiki Co. Ltd.; Aisin Takaoka Co. Ltd.; Aisin World of America Co. Ltd.).............................. 16%
Société Générale de Financement du Québec (SGF) 16%
SNC-Lavalin Inc. 16%

Magnola Metallurgy is truly a strategic partnership with each of the four partners making a significant contribution to the success of the project. Noranda is a diversified natural resources company that operates in three sectors — mining and metals, forestry, and oil and gas. The metals division of the company is a major world producer of many non-ferrous metals, such as Cu, Zn, Al, Ni, Pb, and precious metals. Noranda Metallurgy has extensive experience in building and operating non-ferrous metallurgical plants; their expertise is wide ranging from hydrometallurgical experience from their zinc, nickel and copper refining plants, to knowledge of molten salt electrolysis from their aluminum production facilities. The Japan-based Aisin group is an automotive parts supplier that represents an outlet for a large portion of Magnola's magnesium, and also provides knowledge and expertise in the casting and alloying of magnesium. SGF is the Quebec provincial government's investment arm, whose role it is to promote the development of new industries in the province of Quebec. They own a stake in a large industrial aluminum smelter (Allouette). SNC-Lavalin is a world class engineering construction firm and has a great deal of experience in the design and construction of smelters.

Magnola plans to begin construction of a 58 500 tonne/year commercial plant in 1998 and to start operation in the year 2000.

Pilot Plant

A 200 tpy fully integrated pilot plant has recently been constructed at the CEZinc site in Valleyfield, Quebec during the first half of 1996. At the time of publication, the plant will be in operation. Hatch & Associates carried out engineering, procurement, and construction management of the pilot plant. The primary objectives of the pilot plant project are to optimize the engineering design and operation to further reduce the costs of the commercial plant.

The Magnola process was initially conceived at the Noranda Technology Centre in 1986, and since then, a number of improvements have been discovered and implemented into the process. The development involved a number of activities that included bench scale laboratory testing, unit operations piloting at Noranda Technology Centre and various vendor sites, computer process simulation with the METSIM software, environmental studies on effluent streams and their characterization, site specific studies for the raw material and commercial plant location, technology licensing, and engineering (of the unit operations, the integrated pilot plant, and commercial plant preliminary engineering and cost estimates).

The pilot plant will be operated to provide valuable design information for the commercial plant. Subsequently, during the construction of the commercial plant, it will be used for purposes of operator training.

The Raw Material and the Metal

Magnesium is the eighth most abundant element in the earth's crust and third among metals. A large number of potential raw material sources exist for a commercial magnesium plant. These are presented in Table 1. Currently, the most commonly used raw material feeds for magnesium production are: magnesite, dolomite, seawater, lake brines and carnallite.

The raw material for the Magnola process is serpentine, derived from tailings produced by the numerous asbestos producers that once operated facilities in the eastern townships region of the province of Quebec. These mining residues contain up to 24 wt% Mg and have been accumulated in piles over the many decades of mining the serpentine ore for asbestos fibre (Fig. 1). There is sufficient quantity of feed material for the Magnola plant, far beyond the commercial life of this project, even considering several expansions in the future.

With respect to other major commercially produced metals, magnesium ranks seventh in terms of total world production (which does not include the commonwealth of independent states magnesium production dedicated to the to titanium production) (Table 2).

Table 1. Potential raw material sources for magnesium production

Type	Name	Composition	% MgO	% Mg
Oxides	Periclase	MgO	100%	60.3%
	Brucite	$Mg(OH)_2$ or $MgO.2H_2O$	69.1%	41.7%
Carbonates	Magnesite	$MgCO_3$	47.8%	28.8%
	Dolomite	$MgCO_3.CaCO_3$	21.8%	13.2%
Silicates	Olivine	$(Mg,Fe)_2SiO_4$	55%	27%
	Serpentine	$3MgO.2SiO_2.2H_2O$	43.6%	26.3%
	Enstatite	$MgSiO_3$	40%	24.0%
Sulphate	Keiserite	$MgSO_4.H_2O$	29.2%	17.6%
Chlorides	Bischoffite	$MgCl_2.6H_2O$	19.9%	12.0%
	Carnallite	$MgCl_2.KCl.6H_2O$	17.8%	8.8%
	Brines	$(Mg, Ca, K, Na)X_n$ where $X = Cl_2, SO_4, ...$		> 1%
	Seawater	MgX_n where $X = Cl_2, SO_4$,		0.14%

Table 2. 1993 annual metal production figures (USBM, 1994)

Metal	World Production (1000 mtpa)
Steel	728 000
Al	19 000
Cu	9 300
Zn	7 000
Pb	3 200
Ni	826
Mg	281
Sn	175

Fig. 1. Piles of serpentine tailings for production of magnesium.

Table 3. Energy consumption by processes

Process	Reaction	Energy consumption		
		Theoretical kWh/kg Mg	Cell kWh/kg Mg	Total kWh/kg Mg
Electrolysis	$MgCl_{2(l)} = Mg_{(l)} + Cl_{2(g)}$	6.8 $_{(T=655\ C)}$	11-19	23-31
Silicothermic reduction	$2CaO.MgO + (xFe)Si + nAl_2O_3 = xFe_{(l)} + SiO_2.2CaO.nAl_2O_{3(l)} + 2Mg_{(g)}$	5.3 $_{(T=1550\ C)}$	17-20	32-35

The economics of magnesium production dictate that production facilities either be located close to a feed source or to an inexpensive source of electricity. Therefore, very few countries have production capacities due to the lack of market demand, specific capital, technological and infrastructure requirements for production. The most significant primary producers are, therefore, the United States, the former USSR, Norway, and Canada.

Although a variety of commercial processes exist to produce this commonly available metal (Oye, 1990), the world magnesium production capacity has remained at relatively low levels (as compared to aluminum) and has shown only marginal growth since the end of the Second World War when the production of magnesium fell sharply due to the drop in demand for magnesium used in war planes. However, since 1982, the global primary magnesium consumption has averaged 4% annual growth rate and is expected to increase beyond 4.5% per annum over the next ten years. This will largely depend on the supply base increasing to meet the demand. New entries include the Dead Sea Works in Israel, Queensland in Australia, Magnola in Canada, and other existing producers planning to increase their production capacities in the near future.

Electrolytic magnesium production constitutes the most significant portion (70%) of the total world production capacity of 450 000 tpy (Graham, 1995) and it is the most cost efficient process (Table 3).

The Magnola process, described below, is also an electrolytic process that utilizes the most energy efficient commercial magnesium cell: The Alcan-Multi Polar Cell (MPC).

The Magnola Process

The patented (Peacey et al.,1991; 1994; Celik et al., 1992) Magnola process flow diagram is presented in Figure 2. The objective of this process is to produce high purity magnesium metal from serpentine tailings feed material at the lowest unit price per tonne in the world.

Feed Preparation

The front end loader is used to recover the serpentine mineral at the mine site. It is passed through a scalping screen to remove lumps of material as shown in Figure.2. Typical composition for the feed is 40% MgO, 38% SiO_2, 5%

Fe compounds (Fe_2O_3 and $Fe(OH)_2$), 13% H_2O and varying amounts of CaO and Al_2O_3 depending on the location in the tailings pile. The feed is consistent in mineralogy, chemistry and size distribution because the bulk of the raw material has already been processed in the asbestos mining operations. Therefore, with minimal preparation, the feed is ready for the extraction process. The Magnola Process is designed to accept the serpentine residues without additional feed preparation steps.

The high iron content of the feed necessitates passing the screened feed through a magnetic separator. Prior to the magnetic separation, however, the feed is slurried in recycle water and fresh make-up water. Magnetic separation provides additional benefits that include: improved Mg/Fe separation, lower MgO requirement for neutralization and better filterability of the leach cake. Demagnetized fraction of the feed slurry is then fed into the leach circuit.

Fig. 2. Magnola process flow-sheet.
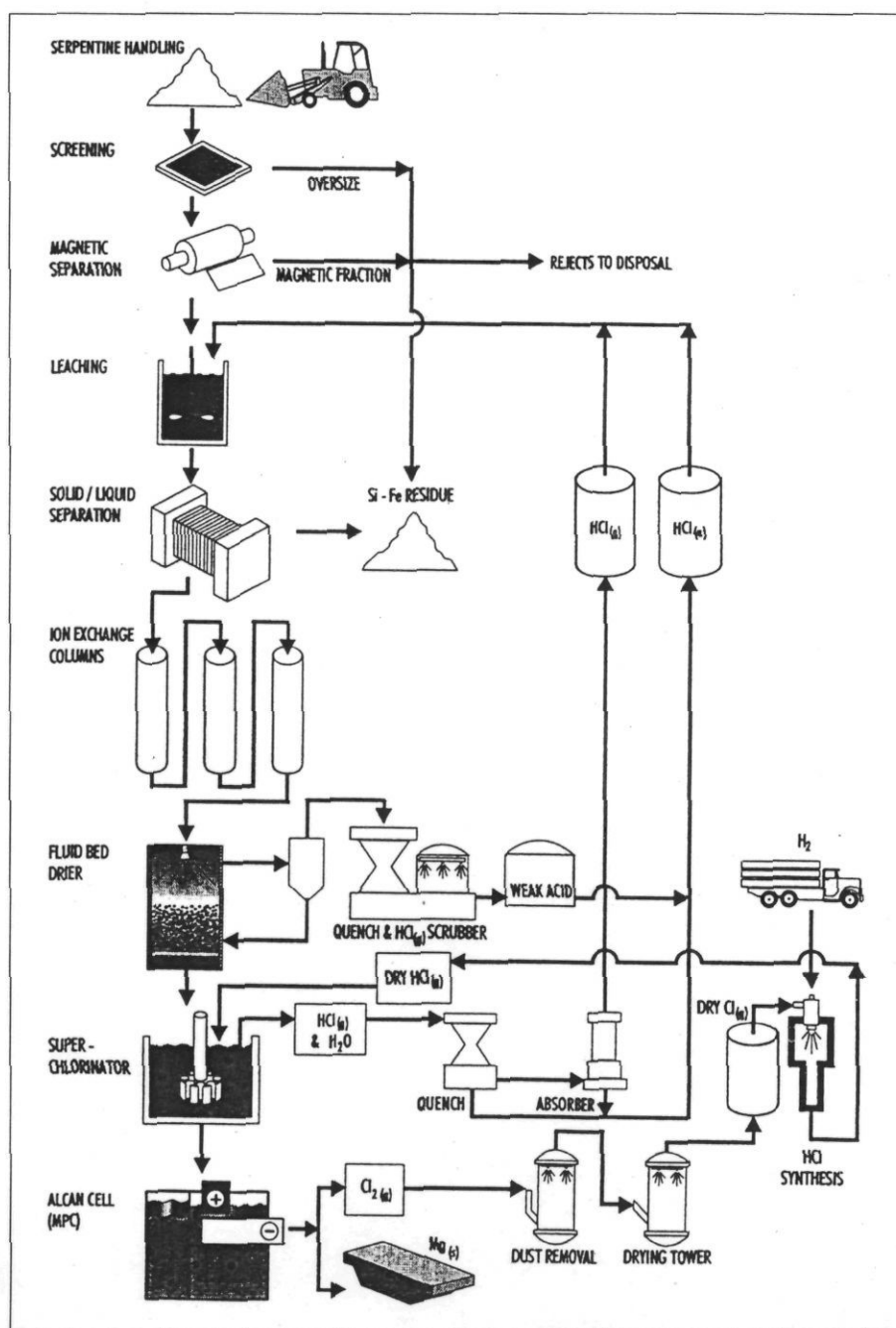
Leach, Purification and Ion Exchange

The slurried serpentine material ($3MgO \cdot 2SiO_2 \cdot 2H_2O$) is fed to the leach circuit at a constant addition rate where it is leached with a combination of hydrogen chloride gas and hydrochloric acid.

The excess hydrochloric acid in the final leach slurry is neutralized with MgO and the unleached residue is then filtered from the impure brine solution. The filtered residue is sent to a residue disposal pond.

The solution is purified by chlorine gas injection under agitation to oxidize such impurities as ferrous chloride, manganous chloride, and nickelous chloride.

MgO and 50% caustic is added for the final neutralization of the chlorinated solution. The precipitated impurities are then removed by filtration. The filtrate goes through a final purification in ion exchange columns to ensure removal to low levels of critical elements such as boron, nickel and manganese. The purified magnesium chloride brine solution is pumped to storage.

The process, based on leaching with gaseous hydrogen chloride and neutralization to produce impure brine has been thoroughly tested at the laboratory scale. These tests have confirmed the concept of the gas leach and provided data that were subsequently used to establish the pilot and commercial plant design values.

Element	% extraction from feed
Mg	80
Fe	80
Ni	55
Mn	70

Off-gases from all the leach, neutralization, chlorination and purification tanks are all collected and vented to the HCl scrubber. Dilute acid (7 wt% HCl) is produced by controlling the cooling rate of the recirculation cooler. This weak acid is returned to the process.

The process water recycle tank collects water streams from the leach and purification area and maintains the plant water balance. The primary use of the water is in preparation of the tailings feed slurry in the day tank. Excess water can be transferred to the effluent treatment plant but there should be none during normal operation.
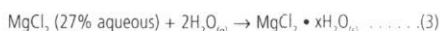
Brine Drying

A fluidized bed drier is used to dehydrate the purified, concentrated brine, containing about 350 g $MgCl_2$/l (equivalent to 27 wt% $MgCl_2$), to produce free flowing granules of hydrated magnesium chloride ($MgCl_2.xH_2O$). The concentrated $MgCl_2$ solution is sprayed through the nozzles over the fluidized bed of prills. The sprayed $MgCl_2$ solution is dried by evaporation when the droplets contact hot particles and gas within the bed. The granular particles grow in size by layering with fresh $MgCl_2$ and by agglomeration. Most of the $MgCl_2$ dust entrained in the off-gas from the fluid bed is recovered in the cyclone and returned to the fluid bed. The off-gases from the cyclone are scrubbed with water to remove the balance of the $MgCl_2$ and the HCl produced in the fluid bed dryer.

Hydrated magnesium chloride ($MgCl_2 . xH_2O$) free flowing granules, called prills, are produced and withdrawn from the fluid bed. The prills are then transferred to the chlorinator downstream. Storage and bagging capability is provided to be able to decouple the chlorinator from the fluidized bed drier.

The reactions occurring during fluidized bed drying are:

$$H_2O_{(l)} \rightarrow H_2O_{(g)} \dots \dots \dots (1)$$

$$HCl_{(a)} \rightarrow HCl_{(g)} \dots \dots \dots (2)$$

$$MgCl_2 \ (27\% \ aqueous) + 2H_2O_{(g)} \rightarrow MgCl_2 \cdot xH_2O_{(s)} \dots \dots (3)$$

$$MgCl_2 \cdot xH_2O_{(s)} \rightarrow MgO_{(s)} + 2HCl_{(g)} + (x-1) \ H_2O_{(g)} \dots \dots (4)$$

Equation 4 produces approximately 1 wt% to 2 wt% of MgO in the feed prills and must be removed from the $MgCl_2$ feed to the Alcan Multi Polar Electrolysis Cell.

Melt Chlorination

The partially dehydrated $MgCl_2$ prills are transferred to the 'Super-Chlorinator' where they are melted and contacted with $HCl_{(g)}$ (Fig. 3). The MgO free molten $MgCl_2$ feed is transferred continuously to the electrolysis cells.

The 'Super-Chlorinator' is an electrically heated (A.C.) solid-liquid-gas phase reactor, where the desired reactions are promoted by the dispersion of hydrogen chloride as small bubbles to react with solid magnesium oxide particles in the melt, and provide sufficient HCl partial pressure to suppress the hydrolysis of $MgCl_2$ with the waters of hydration of the prills.

The hydrogen chloride gas used in the chlorinator is produced in the synthesis unit described in a subsequent section. Sufficient HCl partial pressure in the freeboard of the reactor allows the water of hydration to flash to steam without generating MgO.

A typical granule feed composition consists of:

	wt%
MgCl2•xH2O	97-98
MgO	1-2
Impurities	<0.70

The impurities are mainly sodium and calcium chloride.

Two principle reactions take place within the chlorinator. In the first step, the prill feed dehydrates and melts in the chlorinator according to reaction 5:

$$MgCl_2 \cdot xH_2O \rightarrow MgCl_2 + xH_2O \dots \dots (5)$$
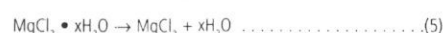
The second reaction is that between the MgO and the HCl and the equilibrium which is established within the chlorinator which determines the required operating conditions:

$$MgO + 2HCl \rightarrow MgCl_2 + H_2O \dots \dots (6)$$

A large number of tests have been performed in an experimental chlorinator to confirm the equilibrium conditions that prevent the hydrolysis of $MgCl_2$. The minimum molar ratio of HCl/H_2O in the gas from the chlorinator to suppress MgO formation has been determined from this work. Batch and continuous kinetic tests

Fig. 3. Magnola super-chlorinator.
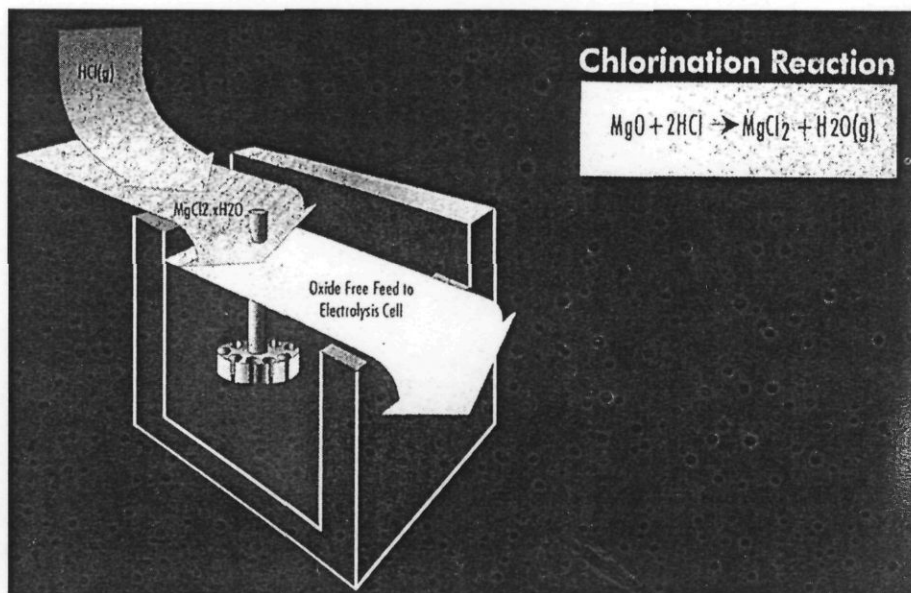
have also been performed to characterize the reaction type and the rates so that correlations that enable proper scale-up can be developed.

The chlorinator operates at a slight negative pressure and all the moist hydrogen chloride off-gases, air and dusts are ducted from the chlorinator to the thermal quench unit (described in another section).

Electrolysis

The design of the integrated pilot plant is centred around providing high quality feed to the Alcan Multi Polar Cell (MPC). Commercial scale cells are operating at Sumitomo SiTiX in Japan to produce magnesium to feed the Kroll process for production of titanium. The commercial sized MPC has been scaled down for testing in the integrated flow-sheet of the Magnola pilot plant. A photograph of the cells operating at Sumitomo SiTiX is shown in Figure 4. The cell operates at current levels between 90kA and 140kA.

In response to the oil crisis of the early 1970s, Alcan International and Sumitomo SiTiX, formerly called Osaka Titanium, invested a great deal of time and money to increase the efficiency and productivity of their magnesium electrolysis operations. The result of this investment was profound, the mono-polar electrolysis cell was replaced with the newly developed multi polar cell, resulting in a significant improvement in cell productivity and efficiency. The mono polar cells, which formally yielded ~100 tpy in 1962, produced more than ten times that by 1993 once the Multi Polar Cells technology was implemented. Improvements in power consumption have been equally dramatic. In 1962, the cell required more than 20 kWh to produce one kilogram of magnesium. The power requirement fell to ~10 kWh by 1993. Comparison with other cell technologies is illustrated in Table 4. The cell life is close to two years, after which some internal parts are replaced.

Table 4. Energy consumption in electrolytic magnesium cells

Company	Cell Technology	kWh / kg of Mg
Dow Chemical	Dow	18.5
MagCorp	Russian Diaphragmless	13-15
Dead Sea Mag	Russian Diaphragmless	13-15
Norsk Hydro Porsgrunn	I.G.Farben (Old)	12-13
Becancour	I.G.Farben (New)	12-13
Magnola	Alcan — MPC	10-12
Theoretical min.		7.0

Details of the Alcan-MPC can be found elsewhere (Johnston, 1957; Sivilotti, 1968, 1988; Sivilotti et al., 1976). The Alcan-MPC consists of an outer steel shell lined with a refractory brick layer resistant to salt attack by the molten salt electrolyte. A schematic is shown in Figure 5. The cell is divided into two areas:
- the metal compartment, into which anhydrous magnesium chloride is charged and from which magnesium metal is tapped; and
- the electrolysis or chlorine compartment where the electrolytic process occurs between a complex arrangement of vertical graphite anodes, graphite bipolar electrodes and steel cathodes.

Two main regulating devices are used to operate the cell:
- the thermal regulation of the cell is achieved by a 'heat exchanger' which is installed in the metal compartment; and
- the regulation of the liquid level is achieved by a 'submarine' that is submerged in the metal compartment under the electrolyte/metal level. This submarine maintains a constant melt level when feeding, tapping and continuous electrolysis occur.

The cell electrolyte is composed of: $MgCl_2$, NaCl, $CaCl_2$, and MgF_2. A sufficient bleed for impurity control is achieved when the magnesium metal is tapped from the cell because there is a slight loss of electrolyte.

The offgases from the electrolysis cell contains chlorine, infiltration air, and electrolytic dust which is all ducted to the chlorine cleaning system prior to synthesis with hydrogen for use in the rest of the plant. In the event of operating problems, all the gas can be diverted directly to the caustic scrubbers.

Electrolyte sludge is cast into moulds and transferred to disposal. Other solid wastes that may be produced include refractories and carbon pieces from the cell that are transferred to disposal. Dust from the drop out box is collected for disposal or recycled back into the process.

Casting

The Magnola pilot plant will be demonstrating the magnesium production process. Therefore, it has not been designed with the ability to refine the magnesium produced. The mag-

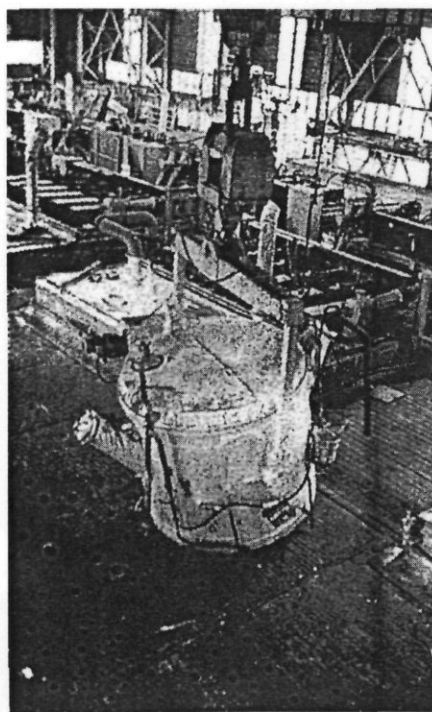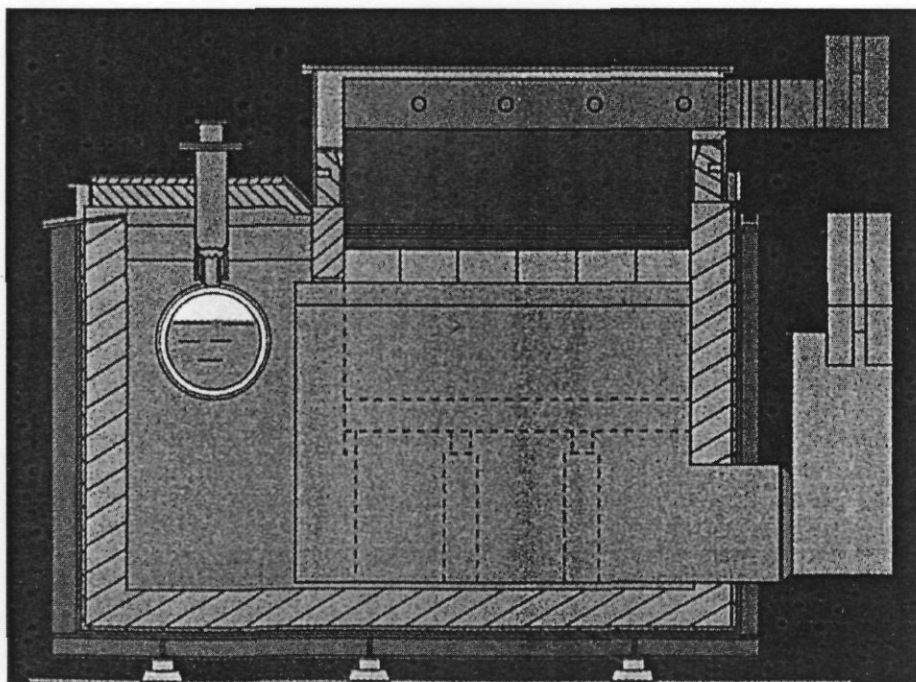**Fig. 4. Multipolar electrolytic cells.**

Fig. 5. Schematic illustration of the Alcan-MPC.

nesium will, however, be cast into loaf moulds in a safe environment. A preheated vacuum ladle will withdraw the magnesium from the electrolysis cell and cast it into 250 lb loaf magnesium ingot moulds under a fume canopy with gas protection. Cooled magnesium loaves will be removed from the moulds at the end of each shift and stacked for characterization and testing.

Chlorine Handling and Treatment

The gas produced by the electrolysis cell contains mostly chlorine and a small amount of electrolyte dust. This hot gas is cooled in the off-gas hood and in the duct system from the cell to the chlorine handling system. The chlorine handling system consists of a primary gas cleaning stage used to remove entrained dust by contacting the gases with counter-current process water in an unpacked tower. This stage is then followed by a drying step where the wet chlorine is scrubbed with counter-current sulphuric acid which absorbs the gas moisture content. The resulting dry and dust free chlorine is then fed to the HCl synthesis unit.

Thermal Quench and HCl Synthesis Units

The dry chlorine produced in the MPC is then reacted with purchased hydrogen to convert all the chlorine to $HCl_{(g)}$ in the synthesis unit. The plant make-up chlorine is added at the synthesis unit. The synthesis reaction is highly exothermic with heat being recovered by indirect cooling of the synthesis unit.

This gas is then sent to the super-chlorinator to perform the final dehydration stage where the HCl picks up a great deal of moisture. The off-gas from the super-chlorinator, containing HCl with H_2O, is fed to the thermal quench unit where the gas is cooled, thereby producing aqueous hydrogen chloride and gaseous HCl streams. The cooled aqueous stream containing strong HCl (~30 wt% HCl) is sent to storage to be reused in the leach area of the plant. The gaseous HCl stream, containing 70 vol% HCl is fed directly to the leach tanks.

The important feature of this section of the plant is that it closes the chloride balance for the process by converting the chlorine from electrolysis back to hydrogen chloride gas and aqueous hydrochloric acid. The plant areas that consume chlorine and hydrogen chloride and those that release them in main process streams are listed below:

Consumers	Producers
Leach/purification - $HCl_{(g)}$, $HCl_{(a)}$, Cl_2	Leach/purification - unreacted $HCl_{(a)}$
	Brine Drying - $HCl_{(a)}$
Melt chlorinator -, $HCl_{(g)}$	Melt chlorinator - unreacted $HCl_{(g)}$
	Electrolysis - Cl_2

Scrubbing System

The scrubbing system is designed to handle normal flows such as leach and purification tank vent gases. It is also designed for emergency scrubbing of all plant gases containing chlorine or hydrogen chloride in the event of operational problems. This ensures a safe working environment in the plant and that the stack gases meet environmental requirements.

The emergency mode of the caustic system is designed to recover the dust and scrub the chlorine or hydrogen chloride produced from the chlorinator, the cell, the HCl synthesis unit or thermal quench. Even if the power to the MPC rectifier is cut off (i.e., interruption of the electrolysis and hence the chlorine generation), when a problem occurs, the scrubber is still necessary to remove the chlorine containing gases from the plant gas ducting. For safety reasons, this critical equipment is provided with a secure electric power supply to ensure continued operation during power failures.

Effluent Water Treatment

The plant effluent water is collected in neutralization tanks, neutralized and filtered prior to discharge. Sludge from the clarifier is pumped to disposal.

Spent caustic containing sodium hypochlorite resulting from caustic scrubbing is stored in a tank prior to disposal. Part of this hypochlorite may used in the brine purification process.

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