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SUBJECT: The Reactions of Magnesium and Magnesium
Alloys with Gases at High Temperatures

TO: W. D. Manly

FROM: H. Inouye

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THE REACTIONS OF MAGNESIUM AND MAGNESIUM ALLOYS WITH GASES AT HIGH TEMPERATURES

(A Literature Survey)

INTRODUCTION

Magnesium and uranium are compatible up to the melting point of either metal. In addition to this feature, the attractive nuclear properties of magnesium make it an excellent cladding material for uranium fuel elements. Its use at elevated temperatures, however, raises the possibility of catastrophic oxidation if the can is suddenly exposed to excessive temperatures in the reactor atmosphere, or if the fuel elements are suddenly exposed to air as a result of reactor component failure. In order to assess properly this potential (fire) hazard, the variables affecting the reaction of magnesium need to be known. The results of a literature survey conducted for this purpose are presented in the following sections.

THE COMPATIBILITY OF MAGNESIUM WITH O_2

Magnesium oxidizes in three distinct ways depending upon the temperature.¹ At temperatures up to $450^\circ C$, the oxidation rates observed for pure magnesium in dry oxygen are slight.¹ The rate curves are adequately described by equations of the type $\Delta w = kt^n$ where Δw is the weight gain, k is a constant, t is the time, and n is an interger usually equal to or greater than 2. The MgO films in these cases are protective.

Beginning at about $475^\circ C$, the oxidation rate of magnesium in O_2 is linear, i.e., the rates are described by equations of the type $\Delta w = kt$. The MgO scales in these cases are rather thick and nonprotective due to their porous nature. The effect of temperature in the range in which this relation is valid is shown in Fig. 1.

At still higher temperatures, an acceleration occurs such that the rate curves can be described by two linear intersecting curves. If the temperature is high enough, the magnesium will auto heat to the ignition temperature

1 T. E. Leontis and F. N. Rhines, "Rates of High Temperature Oxidation of Magnesium and Magnesium Alloys," Trans. AIME, 166, (February, 1946), pp 265-292.

Fig. 1. The Oxidation Rate of Pure Magnesium in O_2 . (Ref. 1. Trans. AIME, 166, 265—292 Feb. 1946)

(the Constable Effect) as a result of the heat generated by the oxidation process.² Time-delay experiments of this type are shown in Table I. At a uniform heating rate, the ignition temperature for magnesium has been determined to be $623^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$. This temperature is defined as the explosion temperature, i.e., the temperature at which the rate suddenly becomes extremely rapid and flame appears.

Mechanism of Burning

Prior to the ignition of magnesium, the oxide coating is white in color; however, just an instant before ignition occurs, the oxide coating turns a dark brown or black color.³ Studies of burning magnesium ribbons in flowing oxygen show that the flame front travels at about 8 cm/sec.⁴

The products of combustion are MgO fumes and an ash which is still undergoing reaction as evidenced by its ability to ignite the ribbon at points of contact. Because of the high rate of evaporation of magnesium at the flame temperature, it has been postulated that the fumes originate from a reaction between the magnesium vapor and oxygen at a considerable distance from the surface. This hypothesis is supported by the fact that the fumes are composed of perfect MgO cubes which is strongly suggestive of their growth in space.

The Effect of O_2 Pressure

The ignition temperature of magnesium is oxygen-pressure sensitive. At atmospheric pressure the ignition temperature is 623°C and is increased to about 643°C as the pressure is increased to 10 atmospheres.⁵ Below atmospheric pressure, the ignition temperature decreases to a minimum of approximately 600°C at about 0.4 atmosphere of oxygen, but increases again at still lower pressures to about 645°C . Baur⁶ reports an ignition temperature of 591°C for magnesium in 7 atmospheres of oxygen.

2 W. M. Fassel, Jr., L. B. Gulbransen, J. R. Lewis, and H. Hamilton, "Ignition Temperatures of Mg and Mg Alloys," Trans. AIME, 191, 1951, p. 522.

3 Ibid.

4 R. F. Chaiken, et al, The Reaction of Molten Metals with H_2O , AGC-AE-35, March 29, 1957.

5 Fassel, et al, *ibid.*

6 J. P. Baur, D. W. Bridges, and W. M. Fassel, "High Pressure Oxidation of Metals," J. Electrochem. Soc., 102, 8, 1955, pp. 490-496.

TABLE I

TIME NECESSARY FOR MAGNESIUM TO "SELF-HEAT"
TO THE IGNITION TEMPERATURE

Constant Furnace Temp.(°C)	Sample Temp. at Ignition (°C)	Time Interval to Ignition (Min.)
623	623	0.00
602	623	34-1/2
587	623	213

The Effect of Alloying

The oxidation resistance of magnesium is generally impaired by alloying. Similarly, the ignition temperature is lowered by alloying elements provided that the melting point (liquidus) is lower than pure magnesium. This behavior is shown in Table II for alloys of aluminum and magnesium.

A study of fifteen other binary magnesium alloys shows that (a) nickel and copper are most effective in accelerating the oxidation rate, (b) the elements Fe, Ag, In, Mn, Ca, Si, and Nb have only a minor influence, and (c) Ce and La materially reduce the rate.

The most striking effect of an alloying element is exhibited by beryllium additions to aluminum-magnesium alloys. ^{7,8,9,10,11,12} In the range up to 0.05%, beryllium drastically reduces the oxidation rate and the pyrophoricity of magnesium in air. Tests of Mg-4 Al-1 Zn-0.01 Be alloy show that protection from inflammability of molten magnesium is effective for two hours at 780°C. A spectrographic study of the magnesium surface shows that this protective action is directly related to the fact that the surface skin is enriched some ten times in beryllium content due to its diffusion from layers immediately below it.

Since the oxidation resistance and the ignition temperature is usually lowered by alloying, it follows that contact of magnesium with other metals at high temperatures will have a similar effect due to alloying caused by interdiffusion. Examples of the lowering of the ignition temperature of magnesium in contact with a few metals are shown in Table III.

-
- 7 C. E. Nelson, "Secondary Magnesium," Metals Technol., 10, 1943, p. 77.
 - 8 A. Keil, "Spectrochemical Investigation of the Effect of Beryllium on (The Inflammability Hazard of Molten) Magnesium Alloys," Met. Abstr., 19, 1951-1952, p. 522.
 - 9 A. Keil and P. Bertin, "A Study of Grain Refinement and Protection Against Inflammability of Magnesium-Aluminum Alloys," Met. Abstr., 19, 1951-1952, p. 22.
 - 10 J. Calvet and V. Potemkine, "The Protective Role of Small Be Additions to the Oxidation of Magnesium-Aluminum Alloys," Met. Abstr., 20, 1952-1953, p. 707.
 - 11 K. E. Mann, "The Effect of Be Additions to Magnesium-Aluminum Alloys," Z. Metallkunde, 46, 1955, pp. 17-24.
 - 12 G. Long, P. T. Stroup, and W. T. Ennor, Explosions of Mg and H₂O, "NP-5471.

TABLE II

THE RELATIONSHIP OF THE OXIDATION RATE AND THE IGNITION TEMPERATURE
TO THE COMPOSITION OF MAGNESIUM-ALUMINUM ALLOYS

Alloy Wt % Al	M.P. of Alloy, °C (Liquidus)	Oxidation Rate at 475°C, Mg/cm ² /hr	Ignition Temp. °C
0 (Pure Mg)	650	0.010	623
1.78	643	0.025	605
3.18	635	0.051	580
7.23	620	0.305	558
9.12	605	7.7	543
18.6	547	17.0	503
63	453	-	462

TABLE III

THE LOWERING OF THE IGNITION TEMPERATURE OF MAGNESIUM
IN CONTACT WITH OTHER METALS IN OXYGEN

Contact Metal	Ignition Temperature °C
Magnesium	623
Silver	623
Steel	623
Nickel	588
70-30 Brass	538
Aluminum	523

THE COMPATIBILITY OF MAGNESIUM WITH CO₂

Commercial and special magnesium alloys have been exposed to CO₂ for periods up to 2500 hr. For a total gaseous-impurity content of 50 ppm, the reactions between dried CO₂ and magnesium are slight. No failures were observed below the melting point of magnesium or any magnesium alloy.¹³ The effect of CO₂ in suppressing oxidation is graphically shown in Fig. 2¹⁴ for a magnesium alloy (MJL-5), and in Table IV for pure magnesium.

After ten days of exposure to pressurized CO₂ at 450-500°C, magnesium specimens show an appreciable deposit of carbon, while specimens exposed at 425°C show slight but visible traces, and at 400°C only faint traces are evident. Over-all, the maximum oxidation resistance is obtained with pure magnesium since additions (except Be and Zr) appear to reduce its oxidation resistance slightly.¹⁵ X-ray and electron diffraction work on the films and loose scale produced at one atmosphere of CO₂ gives patterns corresponding to MgO in all cases. In pressurized CO₂, only magnesium and carbon are detected in the films.¹⁶

Thermodynamically, the formation of MgO and MgCO₃ are feasible at 400°C. Furthermore, the deposition of carbon on the same basis is favored by the lower temperatures. It has been postulated that the slight oxidation rate is due to the preferential adsorption of CO₂ on the metal. While decomposition of CO₂ to produce oxygen seems reasonable in view of the MgO deposits, the reaction of the magnesium with this oxygen is inhibited by the initial adsorbed CO₂ film. It is further assumed that the carbon present in the film derives from the catalytic decomposition of CO.

-
- 13 P. Murray, Reactions Between Reactor Materials, RHM (56) 119, April, 1956. (Confidential)
 - 14 I. A. Makolkin, "Oxidation of Magnesium and Its Alloys at High Temperatures," Shur. Priklad Khim., 24, 1951, pp.460-470.
 - 15 T. Grainger and A. B. McIntosh, "Studies of Production Techniques," J. British Nuclear Energy Conference, 2, April, 1957, p. 121.
 - 16 A. B. McIntosh and K. Q. Bagley, "Selection of Canning Materials for Reactors Cooled by NaK and CO₂," J. Inst. of Metals, March, 1956, p. 251.

TABLE IV

RATE OF OXIDATION OF PURE MAGNESIUM AT
550°C AT ATMOSPHERIC PRESSURE

Gas	Oxidation Rate Mg/cm ² /hr
Pure O ₂	0.24
O ₂ Saturated with H ₂ O at 28°C	0.61
Pure CO ₂	Negligible
Pure SO ₂	Negligible
O ₂ + 5% SO ₂	Negligible
O ₂ + 11% CO ₂	Negligible
1% O ₂ + 99% N ₂	0.026
50% O ₂ + 50% N ₂	0.059

Fig 2 The Oxidation Rate of MJ1-5(9.45Al-0.50Zn-0.29Mn) at 460°C
(Ref 12. Shur. Priklad Khim, 24, 460-470, 1951).

The ignition temperature values ranged from $642^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for pure magnesium to $638^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for AM503 (1.25 Mn). The alloys ZTI and ZREI containing Th (or rare earth), Zn, and Zr showed no tendency to ignite while in the solid state. Attempts to ignite molten Mg and its alloys in CO_2 flowing at $20 \text{ ft}^3/\text{hr}$ were unsuccessful. No ignition was observed at temperatures up to 700°C .

THE COMPATIBILITY OF MAGNESIUM IN CO_2 CONTAINING MOISTURE

Moisture in CO_2 increases the rate of attack on magnesium to form MgO . Failure is characterized by a relatively rapid attack spreading from points on the surface and producing an effect similar to pitting corrosion by aqueous media. In some cases the whole specimen is consumed from points of breakdown. In commercial alloys breakdown occurs as low as 500°C in times varying from 72 hr up to 2500 hr, while those alloys based upon the "Magneox E-1" (Al-Be-Ca-Mg) show no failure after 2500 hr at 615°C . The exposure of pure magnesium to CO_2 containing a high moisture content (8%) results in a measurable attack within six hours. The corrosion product in this case was identified as a carbonate.

All the alloys exhibit a rapid oxidation rate following film breakdown. For the alloys based upon Magnox E-1, failure occurs within 24 hr once the protective film ruptures. This phenomenon has been attributed to a vapor-phase reaction in which the magnesium leaving the metal as a vapor reacts with CO_2 within the porous oxide away from the metal surface to form more loose and non-protective oxide.

COMPATIBILITY OF MAGNESIUM AND MAGNESIUM ALLOYS WITH SO_2

The effect of SO_2 on the reactions with magnesium is essentially the same as that observed for CO_2 (See Fig. 2). Up to 600°C the scale which forms on the metal is composed of MgSO_4 and MgO . At 700°C , the MgSO_4 decomposes and only MgO is found.¹⁷

The ignition temperature of magnesium in O_2 - SO_2 mixtures increases from 623°C at 0% SO_2 to approximately its melting point at 1.7% SO_2 . The variations in the ignition temperature with the SO_2 content are shown in Table V.

17 A. Schneider and V. Esch, "Reactions Between Magnesium and SO_2 ," Z. Metallkunde, 32, 1940, pp. 173-177.

TABLE V

THE IGNITION TEMPERATURE OF Mg IN O₂-SO₂ MIXTURES

<u>% SO₂</u>	<u>Ignition Temperature °C</u>
0	623
0.5	630
0.75	638
1.0	646
3.0	648
4.0	649
5.8	648

DISCUSSION

The development of the Magnox alloys as the canning material for the Calder Hall reactors is based principally upon the beneficial effects of beryllium. Although the addition of one per cent aluminum will reduce the corrosion resistance of magnesium and lower the ignition temperature, it is added principally to overcome the difficulty of processing a high-purity metal and to increase the solubility of beryllium in magnesium above 0.005%.¹⁸ Further benefits realized by modest beryllium additions are noted in the fact that inclusions in the alloy are practically eliminated. Thus, it is seen that beryllium counteracts the harmful effects of alloying, renders alloys resistant to moisture-containing gases, and eliminates the fire hazards of the alloy up to 780°C.

The rapid failure of magnesium alloys in moisture-laden CO_2 after rupture of the protective film has been ascribed to the reaction of the CO_2 with magnesium vapor in the scale. It is worthy to mention that a similar behavior occurs during the oxidation of niobium in air in the neighborhood of 400°C. At this temperature, the vapor pressure of niobium is negligible and therefore can hardly be a factor in the sudden acceleration of the rate. Studies of the growth of the films on niobium show that acceleration coincides with the development of numerous cracks in the film which have been determined to be time and temperature dependent.

The important role of the vapor pressure of magnesium in ignition at elevated temperatures cannot be refuted. It should be pointed out, however, that per se, it will not determine the ignition temperature since factors such as alloying and flow rate of the gas over the hot metal have a large influence.

The vapor pressure of magnesium and magnesium-aluminum alloys increases with temperature as shown in Fig. 3.¹⁹ Comparison of these data with the ignition temperatures of the Mg-Al alloys listed in Table II shows the inverse relationship between vapor pressure and ignition temperature and the excellent

18 E. F. Emley, "Symposium on the Calder Hall Nuclear Power Plant," J. British Nuclear Energy Conference, Session 2, July, 1957, p. 254.

19 A. Schneider and E. K. Stoll, "The Vapor Pressure of Mg Over Liquid Mg-Al Alloys," F. Elektrochemie, 47, 7, 1941, pp. 519-526.

Fig. 3. The Vapor Pressure of Molten Mg-Al Alloys.
Composition in wt % [Ref. 17. F Electro Chemic, 47, (No.7),
519-526, 1941]

correlation of the latter with the lowering of the melting point of the alloy. This behavior in conjunction with the inability to ignite molten magnesium in flowing CO_2 and the time-delay experiments points to the dependence of the ignition temperature on the concentration of the vapor. Studies of the explosibility of magnesium powders²⁰ suspended in air show that a critical concentration of the powder is required for explosions and fires (See Table VI).²¹

TABLE VI

CRITICAL EXPLOSIVE CONCENTRATION OF MAGNESIUM POWDERS IN AIR

Alloy	ZK10		Cell Mg		AZ61	
Mesh size	-325	200/325	-325	200/325	-325	200/325
Concentration*	0.0316	0.0418	0.0371	0.0489	0.0465	0.0749
*gr/l or oz/ft^3 of atomized powder						

CONCLUSIONS

The ignition temperature of magnesium is influenced by numerous variables of which the most important appear to be alloying (or dissimilar metal contact) and the concentration of the magnesium vapor over the heated metal. Most alloying elements lower the ignition temperature; however, beryllium additions inhibit the pyrophoricity of alloys up to temperatures in excess of their melting point.

Although there is evidence that the ignition temperature of magnesium is oxygen-pressure sensitive, its effects are slight in comparison to the above factors. Similarly, the changes observed in the ignition temperature resulting from changing the corrosive gas to CO_2 and SO_2 are slight. The corrosion products resulting from decomposition of these gases are MgO and C for atmospheres composed of CO_2 , air, and O_2 , and MgSO_4 in the case of SO_2 atmospheres.

20 T. E. Leontis and F. N. Rhines, 2.

21 Explosibility of Magnesium, Pt. 2, AD-40736, Dow Chemical Company.

Moisture in the above gases accelerates the corrosion rate. In CO_2 , the rate is increased by moisture by a factor of 4 in one month to a factor of 2 after two months. There is no information on the effect of moisture on the ignition temperature; however, if the correlation between oxidation rates and ignition temperatures is still valid, it is expected that moisture will lower the ignition temperature.

For fuel elements of uranium canned in magnesium, it is believed that the real hazard in case of a can failure due to excessive temperature is the pyrophoricity of uranium, which ignites at 350°C in air.

ADDENDUM

To: Clark Goodman
From: Brady, London

Have spoken with Schonland, Stewart and others about CO₂-Mgnox Reaction. Active investigation now going on in high-temperature region. Results will be available AEC and direct contractors.

Results so far indicate slow reaction at temperature below melting point of 630°C. Ignition can occur at melting point in stagnant CO₂ but not in flowing. Fire easily quenched by increasing gas flow rate.

Have initiated formalities to obtain results so far available and other reports when written.

(Ralph Atwood's copy of telegram copied by W. D. Manly during his visit to ACF, January 7, 1958.)