A METHOD OF TESTING STRESS-CORROSION CRACKING IN MAGNESIUM ALLOY FS-1 EXTRUDED BAR

by

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TABLE OF CONTENTS

INDEX OF TABLES

INDEX OF FIGURES AND DRAWINGS

I. INTRODUCTION

Magnesium alloys, the lightest of all the structural metals, became leading strategic materials in the early days of the recent war, and are now firmly established in American industry. Their major advantage is a very high strength-weight ratio, supplemented by toughness, high resiliency, good durability, excellent machinability, and ease of fabrication. The peak magnesium wartime production capacity of nearly 600,000,000 pounds, attained in 1945, left a plentiful supply of this material available for peacetime uses, but the demand for the metal has dwindled to about one-tenth of the peak production capacity.

It would appear logical that the demand for these recently developed alloys would be increased by overcoming the problems which tend to discount their advantages. The major problems preventing many designers from selecting magnesium alloys for proposed construction are (1) their lack of corrosion resistance, especially in the presence of most acids and salts, (2) that their yield strengths are lower in compression than in tension, and (3) their low thermal capacities. The important problem of corrosion is of vital concern to the chemical engineer, since in the selection of materials of construction for the chemical industry, corrosion resistance is absolutely essential.

Many forms of corrosion can destroy magnesium alloy assemblies. One of the severest types of attack is stress-corrosion cracking, which occurs under a condition of high stress in the form of residual stresses due to fabrication, or other stresses caused by externally applied loads. Wherever such corrosion takes place, the metal usually fails completely. Studies of stress-corrosion cracking have been made in both the United States and in Germany, but nearly all of the research has been carried out on the sheet metal. Extrusions are also subject to high design and fabrication stresses, and study of these would therefore appear necessary.

The purposes of this investigation are (1) to develop an accurate but simplified method of accelerated stress-corrosion cracking testing of magnesium alloy FS-1 extruded rectangular bar, 1/8 x 3/4-inch nominal cross section, using standard tension test speciment having a two-inch gage length; (2) to determine the effect of pH on the stress-corrosion cracking limits for the alloy when exposed to an accelerated sodium chloride-potassium chromate solution; and (3) to determine the characteristics of the testing method.

II. LITERATURE REVIEW

Magnesium and Magnesium Alloys

Useful scientific and technical literature on magnesium and its alloys is limited. This is evidently due to the slow progress made in the production methods and technology of these metals in the United States prior to World War II, and later to wartime security measures which prevented publication of information during the period of the most rapid advancement. Since the recent war, an increased amount of information has appeared in the technical journals and trade literature.

Magnesium. Magnesium is a silvery white metal having an atomic number of 12; atomic weight, 24.32; isotopes, 23, 24, 25, 26 and 27; valence, 2; specific gravity, 1.74; melting point, 651° C.; and boiling point, 1100-1120° C.(47)(48). It was recognized by Black as an element in 1755, isolated by Davy in 1808 and prepared in a pure metallic form by Bussy in 1831(49). The first practical means of separation is attributed to Bunsen who, in 1852, derived pure magnesium by electrolysis of the fused chloride. This type of electrolysis is a basic process in the production of magnesium today.

Magnesium is the eighth most abundant element and the sixth most abundant metal found in the earth's crust, constituting about 2.1 per cent of the total. Dolomite and magnesite are the prevalent natural forms of the mineral. In sea water the availability of magnesium is about 5.5 pounds per 1000 gallons, occurring predominantly as the chloride. Various subterranean brines are more highly concentrated.

It is used primarily (1) in the finely divided form or at high temperature in flares, pyrotechnics, Grignard reaction and desulfurization of nickel, copper, zinc and aluminum alloys, and (2) as an alloying ingredient(35).

Production of Magnesium. Magnesium was first produced commercially in Germany about 1913 and until World War I was purchased in the United States for about $1.65 a pound(14). At that time its principal uses were in photographic lighting. American producers began pioneering in 1915, when the price of ingots had risen to $5.00 a pound and the war demanded new sources. The ten years following World War I marked the transition of magnesium from its principal use in flash bulbs to a dependable light alloy for use in structural forms and decorative applications, and from that time through World War II, the production gradually increased annually, with the exception of the periods 1930-1932 and 1935-1936. Stocking and Watkins(77) blame cartel arrangements for retarding the growth of the young industry which might have grown even faster. During the recent war the aviation industry made such great demands for the metal that the Government contracted with ten companies for its production, which reached a peak capacity of 586,000,000 pounds in 1945(14). Table I, page 5, gives the production of magnesium for specified years between 1915 and 1944 and Dow Chemical Company's

TABLE I

PRODUCTION OF MAGNESIUM AND DOW CHEMICAL COMPANY'S AVERAGE PRICES FOR MAGNESIUM INGOTS IN SPECIFIED YEARS, 1915-1945.

Year	Production(78)		Prices(79)	
	U. S., 1000 lb	Germany, 1000 lb	To AMC*, cents per lb	To Other Domestic Customers, cents per lb
1915	88	-	-	-
1918	294	-	-	-
1920	124	-	-	-
1925	245	-	-	-
1926	322	-	-	-
1927	366	-	55	73
1928	521	-	49	78
1929	1,330	-	49	81
1930	1,174	-	48	76
1931	581	-	42	54
1932	792	-	32	37
1933	1,435	5,000	28	35
1934	4,250	-	23	28
1935	4,241	17,000	19	28
1936	3,903	-	20	27
1937	4,540	26,576	20	27
1938	6,415	31,020	20	25
1939	6,700	36,300	19	26
1940	12,522	55,000	18	26
1941	32,500	77,000	16	26
1942	97,926	-	18	22
1943	367,168	-	21	22
1944	336,674	-	-	21
1945	-	-	-	21

*American Magnesium Corporation.

annual average ingot prices for the years 1927-1945.

Commercial magnesium ingots are graded as No. 0, containing 99.99 per cent magnesium minimum; No. 1, 99.85 per cent minimum; and No. 2, 99.0 per cent minimum(81).

Corrosion Resistance of Magnesium. Magnesium is resistant to attack of most alkalies, alkali metal arsenates, chromates, dichromates, fluorides, ammonia, pure chromic acid, concentrated hydrofluoric acid (5 to 60 per cent), and of most organic chemicals including hydrocarbons, some aldehydes, ketones, alcohols, phenols, amines, esters and most oils(2). Commercially pure magnesium resists the attack of most atmospheres more satisfactorily than does mild steel(11), due essentially to the formation of the protective hydroxide or carbonate, and unless moisture is trapped in crevices or low spots, no structurally serious corrosion will occur(4). Marine and seacoast locations are more corrosive and may cause severe attack(6)(12).

The protective film is not impervious but retards subsequent attack(15). It is preferable to supplement this natural coating with additional surface protection provided by paint systems, resin finishes and chemical treatment(12).

Natural Protective Coatings. Upon ordinary atmospheric exposure, a primary oxide film forms on the surface of magnesium but this is readily converted, though probably not wholly, to such products as the hydroxide, basic carbonate or basic sulfate(27)(33)(87)(88). The film is almost as protective as that on aluminum in atmospheric exposure but is much less protective when in contact with water containing carbon dioxide. Salt water penetrates these films and renders them non-protective(24)(26)(88)(89)(90). Bushrod(25) found that the hydroxide was destroyed at pH 6 or lower. Boyer(23) studied the behavior of this film in salt water and found that pure magnesium immersed in sodium chloride evolved hydrogen rapidly at first, with visible evolution of hydrogen continuing for 26 hours. It may be considered that the retardation of hydrogen evolution is due to a slowing down of the rate of supply of the reactants through the film to the surface of the metal itself(44). Boyer concluded that the overvoltage of magnesium coupled with the protection offered by the film was sufficient to stop hydrogen evolution. The effect of the oxide is to slow down diffusion rates and so aid in the change in nature of the solution.

Effect of Impurities on Rate of Corrosion. Hanawalt, Nelson and Peloubet(43)(44) prepared "super-pure" magnesium by distillation and carefully alloyed this metal with alloying constituents, which were likewise specially purified. Their conclusions were that copper chloride, ammonium chloride, ammonium fluoborate, sulfur, magnesium nitride, magnesium oxide, magnesium oxychloride, carbon, helium, nitrogen, carbon disulfide, acetylene, carbon monoxide, carbon dioxide, hydrogen, hydrogen sulfide, hydrochloric acid, chlorine, methane, sulfur dioxide, oxygen, natural gas, air, and flux inclusions have "no significant effect" on the corrosion of the high-purity magnesium and its alloys, provided certain metallic impurities were not introduced.

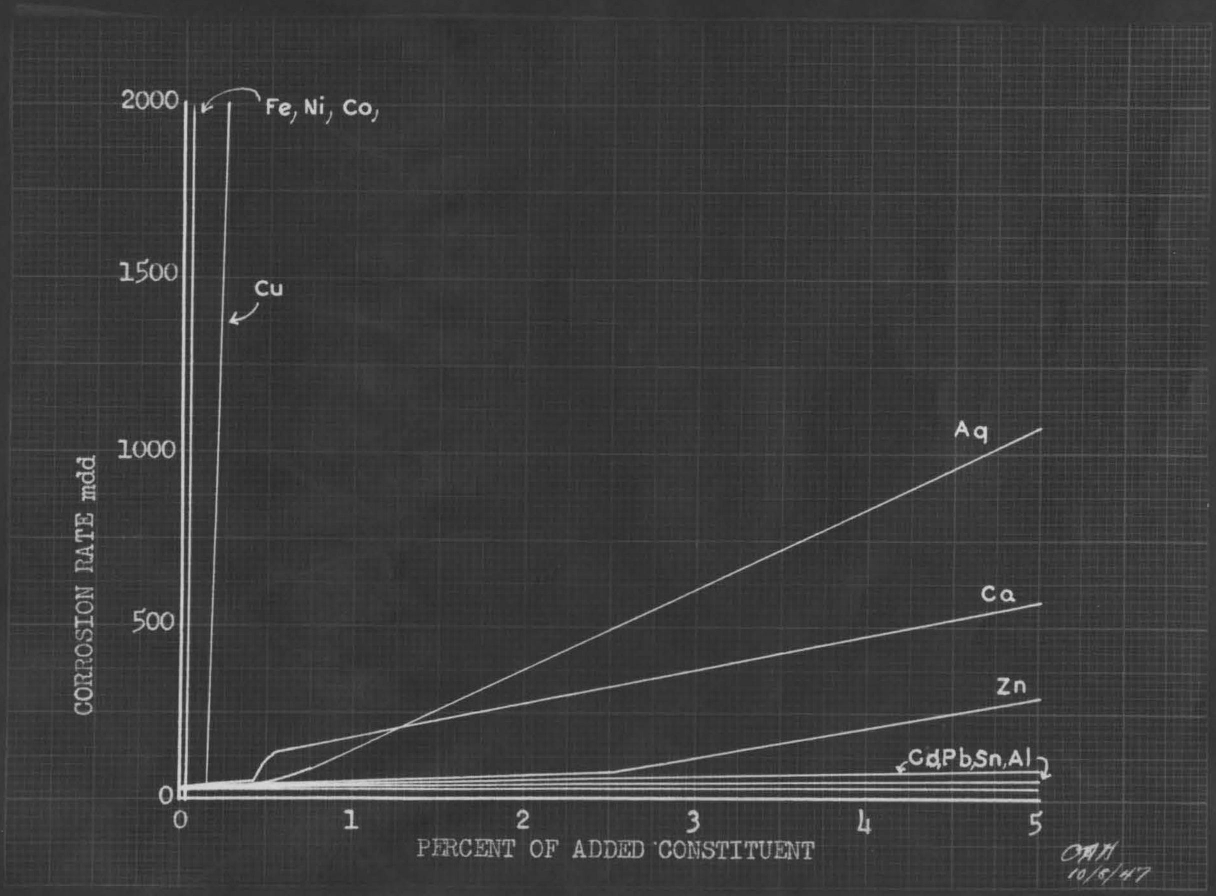
Tolerance Limits for Various Impurities. These investigators drew conclusions on tolerance limits for their alloys, finding that limits for iron, nickel and copper were 0.017 per cent, 0.0005 per cent and 0.1 per cent, respectively. These limits are markedly changed by the presence of commercial alloying constituents. Uhlig(84) gives the effect of certain impurities on the rate of corrosion of magnesium as shown in Figure 1, p. 9; and states that in salt water pure magnesium corrodes at about 0.012 inch penetration per year, whereas the rate for commercial magnesium is 100 to 500 times greater.

Fortunately, iron does not combine readily with magnesium or magnesium-base alloys and is precipitated in the production of practically all the magnesium alloys except the Mg-Mn type used in this country(63), therefore little danger is incurred by iron in combination. However, exclusion of free iron and iron oxides from the metal during production and in subsequent handling is essential.

Theory on Effect of Impurities. The reason for these higher corrosion rates due to impurities is attributed to the theory that cathodic areas are produced by the impurities, thus producing solution of the anodic magnesium(29). Hanawalt and his associates(43)(44) have somewhat justified this theory, although their data are of limited scope, by measuring the increase in potential of the external circuit, using the various metals which

FIGURE 1

EFFECTS OF VARIOUS IMPURITIES ON THE RATE OF CORROSION OF MAGNESIUM IN 3 PER CENT SODIUM CHLORIDE SOLUTION(84)



cause accelerated corrosion of magnesium when serving as cathodes in an electrolytic cell consisting of a platinum anode and a 3 per cent sodium chloride solution. The results indicate that the corrosive effect of the impurities is in the same relative order as that determined by the corrosion experiments.

Dix(29) cautions that many of the accepted conclusions on the effect of alloying constituents on resistance to corrosion are probably masked by the presence of unknown quantities of impurities when tests were carried out, and it is likely that the principal effect of the alloying elements may be the indirect effect on the tolerance limits of the impurities.

Magnesium Alloys. Like other pure metals, magnesium does not possess sufficient strength and corrosion resistance to warrant its use as a structural material(13). Its elastic limit is only about 2,500 psi(82). However, when alloyed with such elements as aluminum, manganese and zinc, strength is added to lightness to produce a very high strength-weight ratio. Table II, p. 11, compares the strength-weight ratio of magnesium alloys with those of other metals and alloys. Corrosion resistance is greatly improved by certain alloying elements. Commercial magnesium alloys are available as sand castings, permanent mold castings, die castings; forgings; rolled sheet, strip, and plate; extruded sections including bars, structural shapes and special shapes; and tubing(7). Aluminum, zinc and manganese are the chief alloying metals in magnesium alloys, and all commercial alloys contain one or more of these.

TABLE II

STRENGTH-WEIGHT FACTORS OF VARIOUS MATERIALS(53)

Material	Tensile Strength, psi	Average Specific Gravity	Strength-Weight Ratio
Steel, piano wire, cold drawn	400,000	7.84	51.0
Steel, very high alloy	250,000	7.85	31.8
Steel, 18-8 stainless, cold-rolled	200,000	7.93	25.2
Magnesium alloy, J-1h	47,000	1.80	25.9
Aluminum alloy, 24SRT	68,000	2.77	24.5
Steel, alloy, heat-treated	190,000	7.85	24.2
Spruce for aircraft	10,000	0.435	23.0
Duralumin	58,000	2.79	20.8
Aluminum, cold rolled	24,000	2.71	8.9
Steel, cold-rolled	60,000	7.84	7.6
Iron, ingot	40,000	7.87	5.1

Aluminum. Aluminum is used more exclusively than any other metal and practically all of the alloys contain an amount of this element. The maximum solubility of aluminum in magnesium is 12.1 per cent at the eutectic temperature of 436° C., and 2.6 per cent at room temperature(2). The decreasing solubility makes these alloys amenable to heat treatment and aging(85). Aluminum enhances both mechanical and corrosion-resistant properties, and in quantities up to 10 per cent it facilitates heat treatment(2).

Zinc. Zinc is added in quantities up to 3 per cent to increase the resistance to salt water corrosion and to improve mechanical properties in the heat-treated condition(2). This element stabilizes magnesium-aluminum alloys if small amounts of iron and nickel are present, but if these are absent zinc is not required. Also, in the ternary system, zinc improves the elongation(2).

Manganese. Manganese improves corrosion resistance when alloyed in quantities up to 0.3 per cent, although it has practically no effect on physical properties(85). It is added to commercial alloys to the extent of 0.1 to 0.5 per cent to improve corrosion resistance(2) and 1.0 to 2.5 per cent produces an alloy with superior welding properties. Manganese, according to Hanawalt(43)(44), does not increase the tolerance limit for iron and copper but does raise the limit for nickel from 0.0005 per cent to 0.001 per cent with 0.2 per cent manganese and to 0.015 per cent with 2 per cent manganese. Although it does not raise the tolerance limit for iron, manganese apparently does minimize the undesirable effects of this impurity.

Silicon. Silicon is not soluble in magnesium and is always present in the alloy as Mg_2Si which, precipitated in the form of lamellar crystals, increases the hardness, while tensile strength reaches its maximum value with approximately 1 per cent silicon(2).

Other Alloying Elements. Tin, zirconium and cerium are employed to a limited extent as alloying metals. Alico(2) gives, with references, the primary effects of other metals when alloyed with magnesium. These include antimony, arsenic, barium, beryllium, bismuth, cadmium, calcium, cobalt, copper, lead and silver.

Heat Treatment. Mechanical properties of some of the magnesium alloys can be improved by heat treatments, whose functions are (1) to harden and strengthen, (2) to relieve internal stresses as in castings, (3) to soften objects for further working, or (4) to anneal objects previously deformed cold(32).

Solution Heat Treatment. Solution heat treatment for magnesium alloys is carried out at temperatures between 500° and 800° F., depending on the alloy composition(3). The purpose of such treatment is to throw as much of the material as possible into solid solution in order to give higher tensile strength and maximum elongation or ductility.

Aging Heat Treatment. Aging heat treatment(62) takes place in the range of 300 to 500° F. after the solution heat treatment and is given to increase the yield strength and hardness of the alloys.

Stabilizing Heat Treatment. Stabilizing heat treatment(62) involves treatment from 425 to 550° F. and therefore falls under the same type of treatment as does the aging treatment. It is distinguished from that treatment since it may be applied to the as-cast metal or after solution heat treatment in order to obtain rapidly a complete precipitation. The function is to give almost complete stress relief and freedom from growth due to aging in service. Indications are that creep strength is highest in the stabilized heat-treated state.

Corrosion

Corrosion has been defined as the destruction or deterioration of a metal by chemical action of its environment(70).

Annual costs of corrosion and its prevention have been estimated to be in the neighborhood of $3,000,000,000, which marks it as a problem demanding continued investigation as to its causes and prevention. Whoever it was that first called it a "major industry - in reverse," put it well. The problem exists, more or less, wherever there are metals or alloys present. Variables which affect the susceptibility to and the rates of corrosion are numerous, and the problems become increasingly complex where many variables are active simultaneously. This is usually the case in service.

Data and literature on the subject are exceedingly voluminous, and only the most important and related subjects will be brought out in this review.

Essence of the Electrochemical Theory. The electrochemical theory(86)(92) is the generally accepted theory of corrosion. Other explanations have been offered but the superiority of this theory is seldom questioned.

According to the electrochemical theory, the complete corrosion reaction is divided into an anodic portion and a cathodic portion occurring simultaneously at discrete points on the metal surface. The anodic portion carries out the destructive action--the metal enters the environment as ions with the loss of electrons. At the cathode there is a gain of electrons, usually by hydrogen ions which are discharged as hydrogen gas, or as water upon reaction with dissolved oxygen or with hydrogen acceptors.

The flow of electricity may occur from local cells set up on a single metal surface or between dissimilar metals. It may also be externally applied, accidentally or deliberately.

The general mechanism of corrosion is shown by these simple equations(71) involving the formation of rust by iron in water in presence of air:

(1) $Fe + 2H^+ \rightleftharpoons Fe^{++} + 2H$

followed by either

(2a) $2H + \frac{1}{2}O_2 \longrightarrow H_2O$ (Destruction of film of hydrogen)

or

(2b) $2H \longrightarrow H_2$ (Removal of hydrogen as gas bubbles).

These allow (1) to proceed with accumulation of Fe^{++} in the solution, followed by

$$(3) \quad 2Fe^{++} + \tfrac{1}{2}O_2 + H_2O \longrightarrow 2Fe^{+++} + 2OH^- \rightarrow \begin{array}{l}\text{Insoluble}\\ \text{ferric}\\ \text{hydroxide}\\ \text{(Rust)}\end{array}$$

The rate of this corrosion process, as a whole, is determined by the rate of the slowest of these reactions under the particular conditions. In neutral or alkaline solutions, (2a) controls the rate of corrosion; in strongly acid solutions, (2b) usually predominates. Numerous other factors affect the corrosion rates, however.

Factors Controlling Corrosion. According to Speller(71), corrosion is controlled by (1) primary factors which affect the tendency of the metal toward solution and (2) secondary factors having to do with the subsequent rate of corrosion. Primary factors are associated with the metal; secondary, with the environment. The rate factors are the more important. He sums up the principal factors as follows:

Primary factors having to do mainly with the metal or alloy:

1. Effective electrode potential of the metal in solution
2. Chemical and physical homogeneity and texture of its surface
3. Its inherent ability to form an insoluble film
4. Overvoltage of hydrogen on the metal surface

Factors which vary with the environment:

5. pH of the solution
6. Effective supply of oxygen in the solution adjacent to the metal
7. Distribution of available oxygen on the metal surface
8. Specific nature, concentration, and distribution of other ions in the solution
9. Effective rate of flow of the liquid past the metal surface
10. Presence of solid particles (dirt), or of a coating of any kind as mill scale, on the metal surface, or contact with other conducting material in the presence of an electrolyte
11. Temperature
12. Whether the metal is under static stress conditions or exposed to cycles of alternating stress (the so-called corrosion fatigue)
13. Ability of environment to build up protective films on the metal.

McKay and Worthington(56)(57) consider these same points to be the most important in controlling the corrosion processes.

Effects of the Principal Factors. The effects of the principal factors controlling corrosion are briefly explained as follows:

Hydrogen-Ion Concentration. Increasing the hydrogen-ion concentration favors the tendency of the formation of atomic hydrogen, therefore metals tend to corrode more rapidly in acid solutions(71). An opposing effect, though less apt to occur, is that in some cases the hydrogen-ion concentration changes the solubility of corrosion products (such as hydroxides) and therefore the nature of the protective film. For iron or steel, the dividing line between rapid corrosion in acid solutions, and moderate or slow corrosion in nearly neutral or alkaline solutions, occurs at about pH 4.5. For the amphoteric metals, such as aluminum and zinc, highly alkaline solutions may be even more corrosive than acid solutions, causing profuse evolution of hydrogen(36). It has been demonstrated that the initial rate of corrosion in alkalies may be as great as that in solutions of lower pH, but the coating is built up very rapidly to decrease this rate.

Overvoltage of Hydrogen. On most surfaces hydrogen has a resistance, termed "overvoltage", to overcome before it can be evolved as a gas. The magnitude of this overvoltage depends mainly upon the nature and condition of the surface, and also upon temperature, pressure, nature and velocity of the corroding medium and the size and number of the hydrogen bubbles formed(71). Bancroft(18) considered the overvoltage to be due to polarization by electrically neutral monatomic hydrogen which must react to form molecular hydrogen in order for it to be discharged as a gas or dissolved in the solution. Electrical contact of dissimilar metals (one high and one low in the electromotive series) breaks down the protection of high overvoltage on the first metal and allows evolution of hydrogen from the second(71). The rate of corrosion under-water, in most cases, is directly dependent upon the rate at which some oxidizing agent can depolarize the hydrogen.

Oxygen Concentration. Dissolved oxygen may act as a promoter or inhibitor of corrosion. An increase in oxygen concentration generally causes a decrease in the area or probability of attack but tends to increase the intensity of any attack which does occur(21). One of the two main ways of removing hydrogen from the system (other than evolution as bubbles) is the reaction with dissolved oxygen to form water. The rate of corrosion in natural waters is determined by the rate of depolarization of the cathode areas, and therefore by the rate at which dissolved oxygen reaches the cathode surface(72). According to Speller(72), "The effective oxygen concentration at the metal surface is determined by many factors including (a) the concentration of oxygen in the solution as a whole which is influenced by the partial pressure of oxygen above the solution, the area of contact with the atmosphere ..., the rates of solution and diffusion of oxygen, the viscosity and the motion of the solution; (b) the temperature ...; and (c) the degree of permeability of films or coatings ...". Oxygen concentration cells have a very important effect on the rate of corrosion: two areas, as shown in Figure 2, p. 19a, having different oxygen concentration, cause

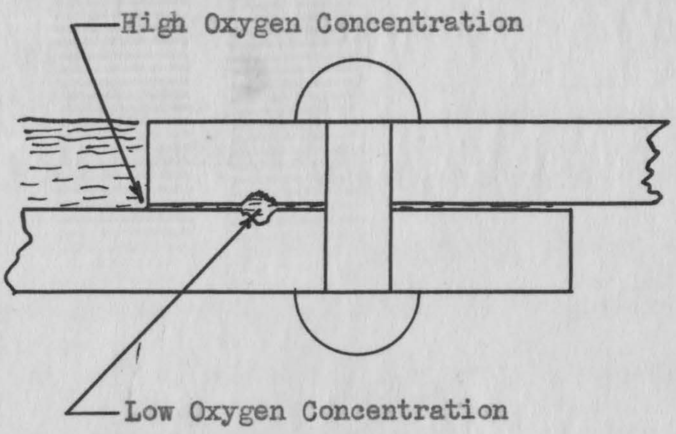


Figure 2. Localized action due to oxygen concentration cell effect.

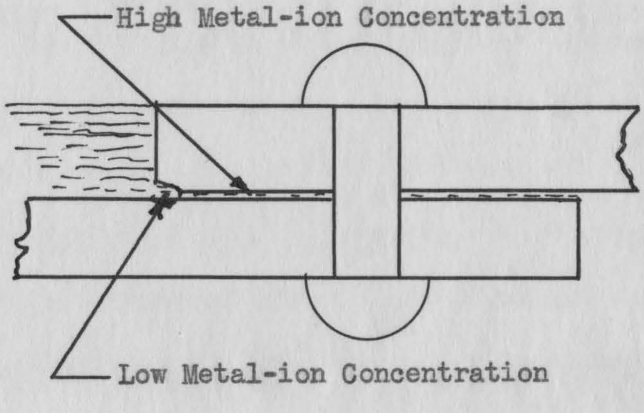


Figure 3. Localized action due to metal-ion concentration cell effect.

localized corrosion or pitting at the area which is less accessible to oxygen.

Metal-ion Concentration. As previously mentioned, the tendency of a metal to enter solution is greatly influenced by the concentration of the ions of that metal already in the solution adjacent to the metal. The lower the ion concentration, the greater is the tendency of the metal to dissolve. Therefore, any effect which removes the metal ions from the surface, such as flow of the liquid, will cause more rapid corrosion in those areas. Metal-ion concentration cells, as shown in Figure 3, p. 19a, are set up between areas of high and low metal-ion content, causing localized corrosion at the points of low concentration. The presence of ions of other metals influences the rate mainly by affecting the nature and distribution of corrosion products. They often break down protective films already formed.

Protective Films and Passivity. Some metals, including aluminum, magnesium, chromium, silicon and stainless alloys, form a very thin film over their surfaces under many conditions(71). This film is practically impervious and therefore protects the metal from further action by the corrosive environment; the metal thus becomes passive. The film is invisible and is thought to be made up of one or more layers of oxygen atoms attached to the surface metal atoms by ordinary forces of chemical valence(40)(71). This condition can be brought about by oxygen or other oxidizing agents such as nitric acid, chlorates, and chromates. The physical and chemical characteristics of metal films are factors of very great importance with respect to the life of the metal(45).

<u>Velocity</u>. An increase in the velocity of the corrosive medium past the metal surface tends to accelerate corrosion(5)(73), mainly because it causes more oxygen to be brought to the surface of the metal. Motion also tends to thin the liquid film on the metal surface, reducing the barrier for oxygen to cross. In the absence of oxygen and if velocity is not uniform, the area subjected to the higher velocity becomes anodic. This situation with oxygen present is reversed. Velocity can be sufficient to cause mechanical erosion, not of the metal itself, but of the corrosion products(74)(93).

<u>Temperature</u>. The major effect of temperature is to increase the rate of corrosion. The relationship between rate and temperature is seen in the following equation:

$$\log R = A + \frac{B}{T}$$

where: R = rate of corrosion
A and B are constants
T = absolute temperature

Temperature also has secondary effects on the solubility of gases, such as oxygen, which often results in a decrease in the oxidizing power of the solution(55). Also, temperature somewhat affects the solubility of corrosion products.

Galvanic Corrosion. Two-metal galvanic corrosion is rapid corrosion of a metal due to its being electrically connected to another metal in corrosive conditions(56). The effect depends entirely on the different chemical properties of the two metals. The circuit must be complete through conducting metals and solutions.

Principal factors influencing galvanic corrosion are the resistance of the solution and the metal-to-metal contact, the potential set up between the metals, polarization of the anode or cathode, relative areas of anode and cathode, and the geometrical relationship between the two metals(5).

Pitting. Pitting or localized corrosion may result from a number of situations. Mears and Brown(60) attribute this type of attack to the following possibilities:

1. Impurities
2. Grain boundaries
3. Differential grain size
4. Differential thermal size
5. Surface roughness
6. Local scratches and abrasions
7. Difference in shape
8. Difference in oxygen
9. Differential concentration of solution; also composition
10. Differential aeration
11. Differential heating
12. Differential illumination
13. Differential agitation
14. Contact with dissimilar metals
15. Externally applied potentials
16. Complex cells

Corrosion Testing

Speller(75) warns that designing a rational system of corrosion tests requires a good working knowledge of the corrosion reactions and of the various factors involved. These have been discussed. Borgmann and Mears(22) emphasize that no one test can be expected to evaluate accurately the "life" of the material except under conditions similar to those of the test itself. There is no standard or preferred way to carry out a corrosion test.

Aims of Corrosion Tests. The type of corrosion test to be used should be governed by the end desired(22). Tests are employed to achieve one of five different aims. These are:

(1) To study the mechanism of corrosion

(2) To select the most suitable material for resisting a certain environment

(3) To determine environments suitable for a given metal

(4) To develop new alloys

(5) To serve as a control test

Types of tests can be classified in general as laboratory tests, plant tests, field tests and service tests(5).

Value of Accelerated Tests. The most reliable method of determining the resistance of a material in a certain environment is to test it under exact service conditions to destruction(75). Such tests can seldom be carried out, primarily because of the cost of unsuccessful experiments and the long time usually involved.

Quick tests are essential even though the results do not always parallel those of service tests.

Accelerated tests are deemed permissible when they include all the important factors and conditions found in service, and these factors are related to one another as they are in service. Controlling factors must control; unimportant factors must remain unimportant. This can be attained by accelerating all the factors equally.

Different accelerated tests for different types of corrosion can usually be devised and can often be carried out to advantage in the laboratory, where the factors can be controlled. Such tests should be calibrated against service results wherever possible.

Submerged Corrosion Tests. The most important factor to be considered in an immersion test is the oxygen concentration. In most cases, oxygen concentration is limited by the saturation value under atmospheric conditions. The rate of absorption in quiet water is not great enough in most cases to insure complete saturation of the liquid(1). Aeration is usually desirable in immersion tests. Results are also dependent upon velocity, temperature and composition of the liquid.

For systematic testing these conditions and other factors such as slight mechanical abrasions, impurities and temperature fluctuations most be governed(39)(52)(67).

Stress-Corrosion Cracking

Stress-corrosion cracking may be defined as cracking resulting from the combined effect of tensile stress and corrosion(10). It is a specific case of the more general subject of stress corrosion in which a condition of high stress accelerates the corrosion of metals.

Perhaps the most familiar example of this type of corrosion is the so-called "season cracking" of cold-formed brass articles, a phenomonon brought about by the combination of high residual stresses inherent in the metal and presence of ordinary atmosphere or a mildly corrosive atmosphere of an ammoniacal nature. "Caustic embrittlement" found in boiler plates at stressed rivet holes is another common example. However, in recent years more and more types of stress-corrosion cracking have become evident where failures were thought to be of a purely mechanical nature, and it is probable that investigators will find solutions which will cause cracking of any known metal or alloy(30).

Generalized Theory of Stress-Corrosion Cracking. It has been pointed out by Dix(28) that in order for accelerated corrosion by high stresses to take place there must exist two conditions. These are (1) susceptibility of the alloy to selective corrosion along more or less continuous paths, as for instance, at the grain boundaries, and (2) a condition of high stress acting in a direction tending to pull the grains apart. Therefore, those metals which are subject to intergranular corrosion are usually susceptible to stress-corrosion cracking; and with few exceptions the path of stress-corrosion failure follows the grain boundaries. Mechanical failures due to overloading at room temperature are opposingly intragranular and can usually be distinguished from the corrosion type by this difference.

Intergranular Corrosion. A theory to explain the intergranular corrosion of certain types of heat-treated Al-Cu alloys, which is typical of most all the metals, was developed by Weber and Dix(28) about 1930. It is based on two facts: First, the microstructural condition of the alloy was found to contain discrete particles precipitated from the Al-Cu solid solution and that precipitation at the grain boundaries was in a more advanced stage than within the grains(31). Second, in chloride solutions pure aluminum is electronegative to many of its alloys(34). The theory advanced stated that there is a difference in solution potential between the material composing the grains and that composing the grain boundaries, and that the direction of this potential is to cause the solution of the metal at the grain boundaries to be greatly accelerated. The solution of a very small amount of metal in grain boundaries would proceed rapidly and the corroding solution would be drawn by capillary action farther and farther below the surface, following the more anodic grain boundaries. This causes the ductility and strength of the alloy to be rapidly destroyed.

Potential Measurements of Grains and Grain Boundaries. Brown, Fink and Mears(28) proved that in an Al-Cu 4 per cent alloy given artificial aging (to make it susceptible to intergranular corrosion) the grain boundaries of one sample were anodic to the grain areas of a sample from the same sheet, the difference in potential being 0.044 volt. Brown made further studies on this alloy and found that a difference in potential between the grain centers and grain boundaries in one sample averaged 0.20 volt. These notable experiments were performed with very exacting technique in which either the grains or grain boundaries were coated with Bakelite varnish for insulation and water-proofing; while the other was exposed to the solution and its solution potential measured.

Effect of General Corrosion on Stress-Corrosion Cracking. The presence of general corrosion lessens the chance of cracking taking place. It has been observed that as the number of localized paths of corrosion starting on the surface of a metal increases, the time required for complete failure is likewise increased(61). Experiments(24) have shown that the intensity of corrosion at any one point decreases as the number of corroded points in a given area increases, hence concentrated attack along a single path is less likely to occur in the event of general action. If there is sufficient general corrosion, stress-corrosion cracking may not occur at all. Another factor which lengthens the time to cracking due to a large number of anodic paths is the mutual effect of a number of notches tending to decrease the stress of any one notch. It has been demonstrated by photoelasticity that the stress is concentrated around the base of the notch(46), and that this stress concentration can be reduced by the introduction of similar notches in the vicinity of the original notch.

The Role of Stress. Once corrosive action has begun along a selective narrow path, it is quite obvious that a component of tensile stress acting normal to the path would set up high stress concentration at the base of the path. The deeper the attack and the smaller the radius at the base of the crack, the greater would be the stress concentration(16). This action exposes bare metal to further corrosion, since this bare metal is anodic(24) and as long as the condition of high stress exists, this corrosion will continue, accelerated, until failure of the metal occurs, provided anodic polarization is not large. Read, Reed and Rosenthal(64) recommend that in alloys where no solution precipitation has been observed microscopically, any electrochemical measurements indicating that the grain boundaries are anodic should be carefully scrutinized.

Internal Stresses. The stress required for stress-corrosion cracking may be internally or externally applied. Internal stresses (also called "initial", "locked-up", and "residual") are produced by cold-working, unequal cooling and internal structural rearrangement involving volume changes(28). Practically all cases of stress corrosion encountered in service have resulted from internal stresses caused by fabrication or during the assembly of structures(61) during which a piece is bent, straightened, or otherwise deformed. Common examples are stresses induced by internal structural changes, quenching, cold-forming or "sinking" of tubing(30). Stresses induced by assembly include those from welding, press or shrink fits, wrapping of sheet to fit a structure without preforming, or the joining of poorly fitting parts. The magnitude of these stresses can be determined by a tedious procedure, but except in simple shapes much uncertainty exists regarding their local intensity and direction.

Welded Materials. Welded areas are very susceptible to stress-corrosion cracking. Smith(69) has pointed out that cracking occurs in arc-welded samples of 24S aluminum alloy in the "overheated zone" which contains enlarged grains after cooling and in the "precipitation zone" on either side of the weld bead. Experiments indicate that heat-treated welds are more resistant than as-welded specimens. In heat-treated samples the welds themselves received greater attack than the two zones mentioned above.

External Stresses. Operating stresses (external) are not ordinarily dangerous since most designs specify stresses lower than those required for stress corrosion to occur, but these can be decidedly influential when added to the internal stresses(28)(30). The magnitude of the tensile stress in a purely externally loaded piece is the most important factor contributing to failure due to stress-corrosion cracking. Frequently, a difference of 1000 psi can mean the difference between cracking within a few minutes or no cracking at all. Usually there exists a critical stress below which no cracking will take place, or at which the curve of Applied Tensile Stress vs. Time to Failure levels off sharply, asymptotic to the Time axis. The level of this critical stress differs between alloys and for different corrosive media. It is often stated in terms of per cent yield strength, or simply as the minimum tensile stress read off the curve.

Value of Laboratory Tests. Dix(28) and Madden(59) agree that in order for stress-corrosion cracking tests to have any practical value, the test conditions must be selected with due regard to the service to which the metal will be subjected. Correlation between laboratory tests and service demands is difficult to obtain and often requires several years. Dix believes that stress-corrosion tests are warranted if they give satisfactory results when employed in connection with the following:

(1) In determining which alloy compositions are markedly susceptible to stress corrosion

(2) For alloys susceptible, in determining the fabrication conditions required to reduce or limit the tendency

(3) As means of technical control to insure proper fabrication treatment

(4) By comparison of tests with those of metals in general use which have good service records, the use of specific alloys can be determined.

Importance of Stress Determination. Apparently, the majority of investigators agree that stresses imposed on the specimens subjected to tests should be determined, since most of the published information includes tensile stresses in data. Madden(59) agrees, and aptly bases his statements on the facts that stress greatly affects the results and that it is susceptible to fairly accurate determination. As previously brought out, 1000 psi can be the difference between cracking in a short time or none at all. In order to determine critical stresses, therefore, the maximum applied stresses of the sections tested must be known.

Objects of Stress-Corrosion Cracking Research. Loose and Barbian(54) believe that from a consideration of the practical aspects of interest to designers two main objects should direct stress corrosion research. One is determining the lowest constant stress at which stress corrosion could occur over an extended period of time (the critical stress), and the other is finding the stress-time curve under conditions of residual stress where creep and other factors may influence the results. Sager, Brown and Mears(65) are more general in stating aims of useful stress-corrosion cracking tests. These are (a) to study the mechanism of the cracking, (b) to select suitable material for a specific application (from the user's viewpoint), (c) to determine those applications in which a specific metal or alloy can be satisfactorily used (the metal producer's viewpoint), (d) to help guide the development of new alloys, and (e) to serve as product control tests. Certainly no one test can satisfy all the objects and it is up to the research man to design his tests to suit his specific aims.

Types of Loading Used in Tests. For testing, specimens can be either internally or externally stressed or a combination may be used. Externally applied loads are easier to control and evaluate(65), and therefore are extensively used. As far as it can be determined, there are no standard methods or equipment for stressing the specimens, and it would seem that the choice again lies with the researcher. In external loading, test pieces may be subjected to either constant load or constant deformation. Constant loadings must be applied with springs or with sufficient quantities of weights, and generally such tests produce accurate results at the expense of high cost and bulky apparatus. Loads may be applied in direct tension or as in loading simple beams. Apparatus required for constant deformation or constant deflection, on the other hand, is inexpensive and simple in addition to producing very good results. Specimens have been deflected as columns or simple beams, deformed by fitting them in place against curved surfaces and in numerous other ways. Usually the stresses can be calculated by use of suitable formulae, but if they cannot the maximum surface tensile stresses are determined by use of electric strain gages(54)(59)(66). In tests where the applied stresses are within the plastic range, ordinary formulae do not hold and strain gages are employed so that direct measurement of stresses can be made. Strain gages are now widely accepted in industry(17).

Stress-Corrosion Cracking of Magnesium Alloys

Pure magnesium metal has not been known to stress-corrosion crack(50) but when aluminum is alloyed with it, stress-corrosion cracking ensues. Precipitation takes place within the grain body and probably also along the grain boundaries, and therefore the theories on stress-corrosion cracking offered by Dix and his co-workers(28)(29)(31)(61) are applicable to these alloys. All of the commercial magnesium alloys, except those containing 1.5 per cent manganese, are subject to this type of cracking under ordinary atmospheric conditions(19)(50)(54)(59)(68), in the presence of distilled water(41)(59) and to accelerated cracking in the presence of NaCl (or Na_2SO_4) - K_2CrO_4 (or $K_2Cr_2O_7$) solutions(54)(59)(61)(65) and in several other media(54).

Service results according to Loose and Barbian(54) indicate that stress-corrosion failures are due mainly to residual stresses and not to stresses applied during the use of the fabricated articles. However, in aircraft structural materials, Madden(59) reports that the stress corrosion problem is serious not only because of fabrication stresses, but also because of higher design stresses which must be used in aircraft. He also has stated that reluctance to use certain wrought magnesium alloys is justified by the fact that some have failed on weather exposure in 43 to 672 hours when stressed to 90 per cent yield strength, while aluminum alloys under the same conditions did not fail after 3600 hours.

Corrosion Environment. The possibility of stress-corrosion cracking is regarded to be a function of its corrosive environment. Much work has been done on wrought magnesium alloys(19)(41)(51)(54)(59)(68), and only a few corrosive environments, including those mentioned above, have given positive results.

Atmospheric Tests. Atmospheric-exposure tests have been extensively employed(54)(59) in evaluating the susceptibility of magnesium alloys, and in general they have given very reliable results(71). The main disadvantage of such tests is the comparatively long time required for their completion, but this is offset by the practical value of such tests. No standard apparatus has been employed and specimens have been subjected to loads of both the constant-stress and constant-deformation types(51)(54)(59)(65). Tests show that both rural and coastal exposures produce nearly identical stress-time curves for the same alloy(54). Factors contributing most to cracking are weather conditions. Rain, high humidity and high temperatures have positive effects.

Accelerated Tests. Accelerated tests commonly employ a solution composed of 20 grams per liter of potassium chromate and 35 grams per liter of sodium chloride(51)(54)(59)(65). This solution normally produces cracking in a short time when specimens are stressed above a certain critical stress or no cracking at all at stresses slightly below this threshold point. It is very useful in the investigation of commercial alloys, experimental alloys, and in examining methods of preventing stress-corrosion cracking(65). Ordinary chloride containing solutions employed in corrosion tests for magnesium proved to be inadequate for stress-corrosion work because of the presence of large amounts of general corrosion. The chromates in the salt-chromate solution render the metal surface passive to extensive corrosion, allowing only localized corrosion to take place under the action of the chloride.

<u>Mechanical Properties</u>. Mechanical properties of magnesium alloys affect its stress-corrosion cracking characteristics. Creep or stress relaxation is a major factor which prevents the maintenance of constant stress in bending(54). The creep rates are higher in these alloys than in most structural materials, and the factors affecting this property are not well enough known to quantitatively account for it in time to failure tests on these alloys. Most wrought magnesium alloys have compressive yield strengths lower than the tensile yield strengths. Therefore, in bending tests in which high tensile stresses must be obtained, the stresses on the compression side of the metal will be strained in the plastic range. Ordinary formulae used for calculating maximum bending stresses are workable only when the piece is strained in the elastic range, and therefore are useless where the compressive yield strength has been exceeded. Electric strain gages give direct indications of surface strains, and when

strains so obtained are used in conjunction with stress-strain curves, the desired stresses can easily be determined.

Test Results. Several experiments(54)(65) on magnesium alloys have produced the following results:

(1) No failures reported in alloy M.

(2) J-1 is the most sensitive commercial sheet alloy composition; FS-1 next.

(3) No cracking has occurred on FS-1h or J-1h when clad with alloy M.

(4) Percentages of failures in atmospheric tests are less than the percentages in accelerated tests.

(5) A tensile stress of about 100 per cent of tensile yield strength is required for cracking of FS-1 in salt-chromate tests.

(6) Stress-corrosion limits are usually lower in atmospheric exposure than in accelerated testing. Also the curves level off more gradually in the atmospheric tests.

(7) Types of loading have produced different stress-corrosion limits.

(8) No stress corrosion caused by rivets in aircraft structures for exposures of 1½ years.

(9) Painting greatly extends the time to cracking. Non-painted non-stress-relieved welded structures failed in 5 weeks, similar painted structures started to crack after 5 months.

(10) Specimens stressed to 100 per cent yield cracked when bare in about 10 days; when painted with four-coat system cracking occurred in 6 months.

(11) Rapid stress-corrosion of J-1 as-welded panels (5 weeks in rural and 12 weeks in coastal). Good protection by painting.

(12) J-1h boom (made from sheet), not stress-relieved, cracked at every tack weld; seam welds cracked at right angles to direction of welding.

(13) No cracking in stress-relieved boom after 8 months.

(14) FS-1h booms cracked in many places during welding.

(15) FS-1h booms, stress relieved, gave no cracking in 8 months.

(16) In aircraft structures fabricated by welding, riveting and bolting:
  Ailerons: no cracking after 1½ years on any of four tested.
  Two failures: one in non-stress-relieved tip section of elevator, having dichromate treatment and zinc-chromate primer; another on leading edge of stabilizer exposed bare.

Effect of pH in Accelerated Tests. In accelerated tests(65) carried out on alloy AM-C57S-H (J-1h) stressed as simple beams to 100 per cent yield strength by dead load, the effect of pH on time to failure indicated that pH 2 produced failure in about 2.5 minutes, pH's of 4 to 12 produced failure in 5 to 8 minutes and pH 13 produced no failure in 980 hours. This indicates that when the solution is sufficiently basic, cracking is inhibited. Hunter(51) showed that pH 6.2 initiated cracking in about a tenth the time required using pH 8.2 at various stresses on alloy J-1.

III. EXPERIMENTAL

Purpose of Study

Most of the stress-corrosion testing of magnesium alloys has been carried out on the sheet metal, although extrusions are also subjected to high design and fabrication stresses. The purposes of this investigation are (1) to develop an accurate but simplified method of accelerated stress-corrosion cracking testing of magnesium alloy FS-1 extruded rectangular bar, 1/8 x 3/4-inch nominal cross section, using standard tension test specimens having a two-inch gage length; (2) to determine the effect of pH on the stress-corrosion cracking limits for the alloy when exposed to an accelerated sodium chloride-potassium chromate solution; and (3) to determine the characteristics of the testing method.

Plan of Investigation

The following plan was used in this investigation:

Strain-Deflection Calibration of the Bending Jig. A curve of strain vs. deflection for a standard test specimen of magnesium alloy FS-1 extruded rectangular bar (having a 3/4 x 1/8-inch cross-section), placed in the bending jig was obtained by using

SR-4 type electric strain gages and strain indicator. Creep values were obtained during the calibration.

Obtaining the Stress-Strain Curve. A tensile stress-strain diagram was obtained for the alloy, using a standard A.S.T.M. tension test specimen. The accepted A.S.T.M.(95) procedure was used, and data were obtained using a 10,000-lb testing machine.

Obtaining the Stress-Deflection Curve. A stress vs. deflection curve was obtained for the range including most of the stresses used in this investigation by correlating the strain-deflection and stress-strain curves.

Preparation of the Test Solutions. Stock sodium chloride-potassium chromate test solutions were prepared by dissolving the chemicals in distilled water. This stock solution was separated into several sample bottles and the pH of each was adjusted with hydrochloric acid or a potassium hydroxide solution; pH adjustments varied from 0.5 to 12 for the different solutions.

Stress-Corrosion Cracking Tests. The tests were made with from one to six specimens placed in the jig. After bending a specimen the desired deflection, a short section of 11-mm glass tubing was sealed to the specimen at the point of maximum deflection with paraffin wax. A corrosive medium of a certain pH was placed in the tube, and the time required for failure of the specimen was recorded. Several specimens were run at different deflections with the same solution until the increment of deflection (usually 0.010 inch) was found above which cracking would take place in a short time (usually less than 10 minutes), or, below which cracking would not take place after an extended period of time (one hour or more). Within this increment of deflection was the critical deflection. This critical point was determined in terms of applied maximum tensile stress from a curve of maximum applied tensile stress vs. time to failure.

Materials

The following materials were used in this investigation:

Magnesium Bar, FS-1 Alloy, Rectangular. Extruded. 1/8" x 3/4". No surface treatment. Composition(8): aluminum, 3.0 per cent; manganese, 0.2 per cent; zinc, 1.0 per cent; silicon, 0.3 per cent (max.); copper, 0.05 per cent (max.); nickel, 0.005 per cent (max.); iron, 0.005 per cent (max.); other impurities, 0.3 per cent (max.); magnesium, remainder. Physical properties: minimum tensile strength, 32,000 psi; tensile yield strength, 22,000 psi; compressive yield strength, 13,000 psi; modulus of elasticity, 6,500,000 psi; elongation, 8 per cent. Used as stock in cutting and milling of test specimens. Obtained from White Metal Rolling and Stamping Co., Brooklyn, N. Y.

Test Specimens. (208). (Cut from the magnesium bar). Standard A.S.T.M. Tension Test Specimen(94). Dimensions: overall length, 8 inches; cross section of ends, 1/8" x 3/4"; cross section of reduced section, 0.125" x 0.500"; length of reduced section, 2-1/2"; gage length, 2.000"; radius of curved portions, 2-7/16". Cut and milled from random lengths of rectangular FS-1 alloy magnesium bar in a universal milling machine by Comas Cigarette Machine Works, Salem, Va.

Potassium Chromate. Lot No. 112732. C. P. reagent. Meets A.C.S. standard. Used in preparing salt-chromate corrosive solutions. Manufactured by J. T. Baker Chemical Co., Phillipsburg, N. J.

Sodium Chloride. Jackfrost Fine Kiln Dried, Evaporated. Used in preparing salt-chromate corrosive solutions. Manufactured by Mulkey Salt Co., Detroit, Mich.

Acid, Hydrochloric. C. P. reagent. HCl, 37.0 per cent (minimum) by weight. Specific gravity at 60° F., 1.1878 (minimum). Used in making pH adjustments of salt-chromate solutions. Manufactured by E. I. duPont de Nemours and Co., Inc., Grasseli Chemicals Department, Wilmington, Del.

Potassium Hydroxide. U. S. P. Lot No. 2143. Used in preparing 1.542 N. solution for use in making pH adjustments of salt-chromate solutions. Manufactured by J. T. Baker Chemical Co., Phillipsburg, N. J.

Papers, pH. Hydrion A, pH range 2 - 10 (even) and Hydrion B, pH range 1 - 11 (odd). Used for determination of approximate pH of corrosive solutions. Made by Micro Essential Laboratory, Brooklyn, N. Y.

Wax, Parrafin. "Essowax". Used in sealing solution tubes to test specimens. Distributed by Esso Incorporated, New York, N. Y.

Emery Cloth. No. 2/0. "Grit-Lok Bond". Used in preparing surface of test specimens. Made by Minnesota Mining and Manufacturing Co., St. Paul, Minn.

Paint, Tygon, White. Code No. TP-61. Control No. 7154. Used for protecting portions of specimens from corrosive solutions. Manufactured by U. S. Stoneware Co., Akron, Ohio.

Acetone. Used as thinner for Tygon paint. Obtained from Phipps and Bird, Inc., Richmond, Va.

Wire, Magnet, Copper. Plain enameled. Gauge No. 26. Used in making leads for strain gages. Manufactured by Philadelphia Insulated Wire Co., Philadelphia, Pa.

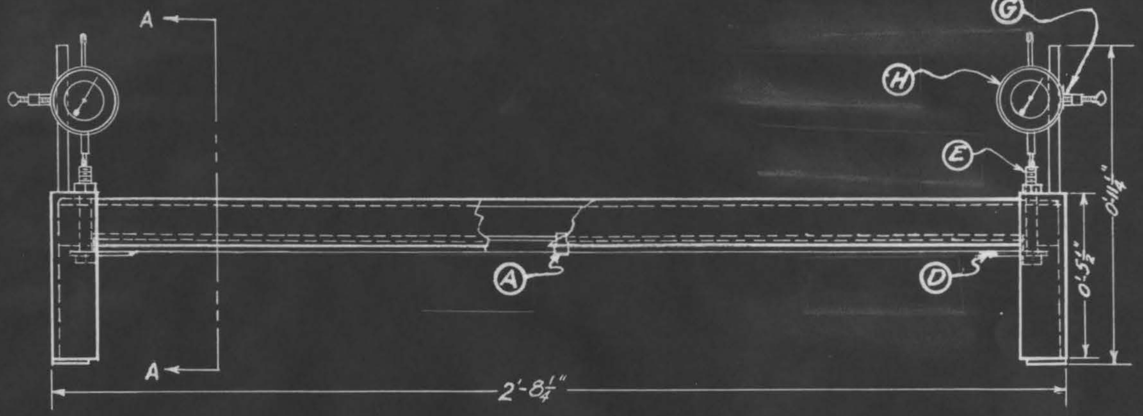
Kit, Cement. For SR-4 strain gages. Contents: One can each of pre-coat cement, cement, pre-coat solvent and cement solvent; one brush. Used for cementing strain gages on specimens. Obtained from Baldwin Southwark Division, Baldwin Locomotive Works, Philadelphia, Pa.

Apparatus

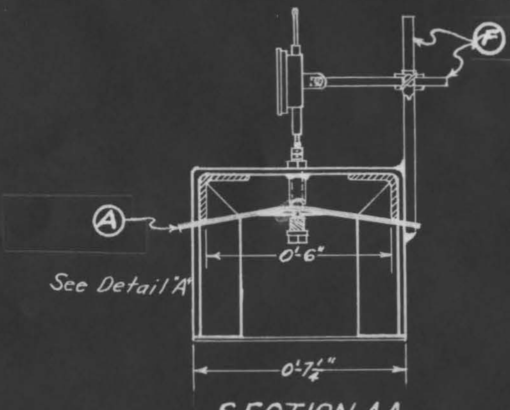
The following apparatus was used in this investigation:

Bending Jig. (Refer to Drawing No. 1, p. 43). Used for applying stresses to test specimens. This apparatus was constructed as follows:

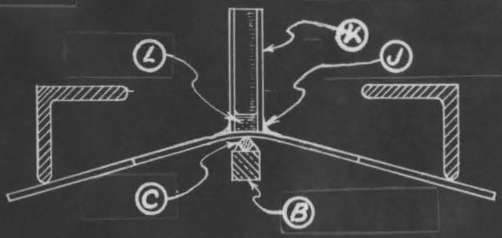
A 29-1/2-inch length of 5/8-inch square steel bar (B) and the same length of 3/8-inch round steel bar (C) were cleaned with emery cloth and tack-welded together with an arc welder. Care was



JIG ASSEMBLY



SECTION A-A



DETAIL "A"

Showing specimen under test.

NOTES:

1. ALL-WELDED CONSTRUCTION.
2. BEARING SURFACES POLISHED.
3. ALL ANGLE IRON $1\frac{1}{2} \times 1\frac{1}{2} \times \frac{3}{16}$"

LEGEND

- Ⓐ Specimen, stressed
- Ⓑ Movable bar, steel, 5/8" sq.
- Ⓒ Bearing surface, 3/8" steel bar, round.
- Ⓓ Bar, rectangular, steel 1/4 x 1"
- Ⓔ Adjustable bolt, 1/2 x 3 1/2", steel, machine
- Ⓕ Indicator supports, 1/2" rd. steel bar
- Ⓖ Ringstand clamps
- Ⓗ Indicator, Ames
- Ⓙ Seal, paraffin wax
- Ⓚ Solution container, 11mm. I.D. glass tubing
- Ⓛ Corrosive solution

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

ASSEMBLY AND DETAIL OF

BENDING JIG

DRAWN BY: ABS DATE: 9/20/47
CHECKED BY: DATE:
APPROVED BY: HOB DATE: 10/1/47

SCALE: NONE
DRAWING NO. 1

taken to be certain that the round bar was centered accurately on the face of the square bar. Two 2-1/4-inch lengths of 1/4 x 1-inch rectangular steel bar (D) were cut and 1/2-inch holes drilled 3/8-inch from one end. These were welded to the bottom side of the square bar so that the edge of the hole was 1/16-inch outside the end of the bar. Two 1/2 x 3-1/2-inch machine bolts (E) were put through the holes and welded in place. Two 30-1/2-inch lengths of 1-1/2 x 1-1/2 x 3/16-inch angle iron were welded at ends, as shown in the drawing, to two lengths of angle iron bent in form of a U. The vertical portions of these bent pieces served as legs for the jig.

Holes, 17/32-inch, were centered and drilled in the horizontal side of the end (bent) angles for insertion of the bolts which were welded to the bar. Two 1-1/4-inch pieces of 17/32-inch steel tubing were welded to the lower sides of the angles, concentric with the holes, and were filed down to allow about 3/4 inch of vertical movement of the adjustable bar.

Two 1/2-inch round steel bars (F) 7-1/2 inches long were welded vertically to the two legs on one side of the jig, with centers 1/2-inch from the end, so that about 6 inches protruded above the angle iron. Ring stand clamps (G) were placed on these steel bars.

A one-inch portion, at the end of each of two 1/2-inch round steel bars, was ground off flat on one side and a 9/32-inch hole was drilled through the flat surface 1/4 inch from the end.

Ames dials (H) were bolted on to these with 1/4 x 1-inch stove bolts and the bar placed through the ringstand clamp.

The bearing surfaces (the 3/8-inch round bar and the lower edges of the long angles) were polished with emery cloth and oiled so as to provide smooth bearing surfaces for the specimens when subjected to bending.

Indicators, Ames. Model 262. Plain bearings. Dial: 0 - 100 with 0.001-inch graduations. Limit: 0.500 inch. Used on bending jig for measurement of specimen deflection. Manufactured by B. C. Ames Co., Waltham, Mass.

Gages, Strain, Electric. SR-4, Type A-1. Lot MH. Gage factor 2.05 ± 1 per cent. Resistance 120. Used in measuring strain of specimens for strain-deflection data. Purchased from the Baldwin Southwark Division, Baldwin Locomotive Works, Philadelphia 42, Pa.

Gage, Strain, Olsen. 2-inch gage length. Used in measurement of strain for stress-strain data. Made by Tinius Olsen Testing Machine Co., Philadelphia, Pa.

Indicator, Strain. Baldwin SR-4 type. Model K. Serial No. D 95374. Used for measurement of strain of specimens for strain-deflection data. Purchased from the Baldwin Southwark Division, Baldwin Locomotive Works, Philadelphia 42, Pa. Made by the Foxboro Co.

Power Supply, A.C. Serial No. 70805-9. 115 volts, 60-cycle a.c. Used as power supply for SR-4 strain indicator.

Purchased from the Baldwin Southwark Division, Baldwin Locomotive Works, Philadelphia 42, Pa.

Testing Machine. 10,000 lb capacity. Used in obtaining stress-strain data on test specimens. Made by Tinius Olsen Testing Machine Co., Philadelphia, Pa.

Electrometer, pH. Model 30. Used for determining pH of corrosion solutions. Manufactured by Coleman Electric Co., Inc. Maywood, Ill.

Timer, Electric "Time-It". One-tenth-second indication. Used in timing short times-to-failure. Purchased from the Fisher Scientific Co.

Hotplate, Electric. 115 v., a.c. Type 40102. Used for heating paraffin wax. Purchased from the Fisher Scientific Co.

Balance, Torsion. 610-gram capacity, 0.1-gram increments. Used in weighing chemicals. Purchased from the Fisher Scientific Co.

Miscellaneous laboratory glassware. Used for preparation and handling of corrosion solutions.

Milling Machine. No. 1B Universal. Comas equipment No. 90. 4-7/8-inch side mill with 1-inch face used. Used in cutting and milling rectangular magnesium bar into test specimens. Manufactured by Kearney and Tucker, Milwaukee, Wis.

Method of Procedure

The method of procedure used in this investigation was as follows:

Strain-Deflection Calibration of the Bending Jig. Two standard specimens (A and B for convenience) were polished in a longitudinal direction between the gage marks to remove burrs, transverse scratches, machine marks, and surface impurities with number 2/0 emery cloth. The polished surfaces were wiped off with a cotton cloth saturated with acetone. A thin coat of SR-4 pre-coat cement was brushed on each specimen over the polished surface and given 3 - 5 minutes to dry. A liberal coating of SR-4 cement was brushed over the pre-coat and an SR-4 type A-1 (gage factor 2.05 ± 1 per cent) electric strain gage was then mounted over the cement so that the longitudinal center line of the strain gage passed through the gage marks and the transverse center line was equidistant from the gage marks. A cork stopper was placed so that it covered the felt strip of the strain gage and a one-pound weight was set on the cork. After the cement became sticky the weight was removed and the cement was allowed to air dry for 24 hours. Leads were soldered to the strain gage wires with acid-core solder. These leads were each approximately 20 inches of number 26 enameled copper wire.

The bending jig was set up near a Baldwin SR-4 type, Model K strain indicator. The center bar was adjusted to the zero position by placing specimen A and another specimen in the jig, one near each end, and bringing the center bar up so that both it and the parallel angle irons just contacted both specimens. The Ames indicators were set at zero. The leads of specimen A were connected to the poles on the strain indicator marked "measuring gage". Caution was taken to prevent the wires from touching any metal objects. Specimen B was placed flat on the table and its leads connected to the "compensating gage" poles.

Standard directions accompanying the strain indicator were followed for obtaining the initial zero reading at the balanced position of the indicator, and in determining the values of the subsequent strains.

Data for the strain-deflection curve were obtained by deflecting the specimen (by simultaneously turning the two nuts equal amounts) in steps of 0.020 inch, balancing the strain indicator and taking a reading in each step. The deflections were recorded to the nearest thousandth of an inch, and the corresponding strains, in microinches (millionths). At higher strains, the effect of creep or stress relaxation was encountered, producing an unstable reading on the strain indicator, which increased with time at a constant deflection. In such cases, the initial readings were recorded along with a reading taken after that period of time when the increase in strain reached a very low rate (about 10 microinches per minute, or less). The difference between these values was recorded as creep. Data were taken up to a specimen deflection of 0.630 inch and a plot was made of Maximum Strain (inches per inch) vs. Specimen Deflection (inches). This curve is shown in Figure 4, p. 59.

Correcting for Creep in the Strain-Deflection Curve. In order to correct the strain-deflection curve for creep, the summation of creep up to and including a certain deflection was subtracted from the final reading taken at that deflection. The difference was recorded as "Corrected Initial Creep". Corrected values were obtained at each deflection and the curve was corrected for creep by plotting these values against the corresponding deflections, as shown in Figure 4, p. 59.

Obtaining the Stress-Strain Curve. A standard test specimen was lightly polished with number 2/0 emery cloth on top and bottom between the gage marks to remove surface irregularities. Average width and thickness of the gage length were obtained with a micrometer to the nearest thousandth of an inch, and the average cross-sectional area was calculated.

The specimen was fitted with an extensiometer attached to the gage marks. The specimen was clamped between the jaws of a 10,000-pound testing machine with more than 2-3/4 inches of the ends of the specimen between the grips. The machine was manually operated. The crank was fitted to the low-ratio gear chain and was turned, for application of tension until the dial on the extensiometer indicated a slight, but positive tension.

The dial was set at zero and after the weight on the machine was balanced, a reading of applied force, in pounds, was taken off the machine. The force was increased by operating the crank and setting the weight in steps of approximately 100 pounds, and readings in thousandths of an inch strain were simultaneously taken off the gage when the weight was balanced. This stepwise operation was continued until the applied tensile stress was approximately 33,000 psi, which is well within the plastic range of the alloy (yield strength of FS-1, as given in literature(8), is 22,000 psi minimum).

Again creep was apparent and in order to obtain corresponding readings of force and strain, readings were taken simultaneously with as little time as possible being lost between readings. From the data obtained, a plot was made of Applied Tensile Stress (psi) vs. Strain (inches per inch). This is shown in Figure 5, p. 61. Stress, in psi, was obtained by dividing the average cross-sectional area of the reduced section into the force. After fitting the curve it was necessary to move the stress axis to the left in order for the curve to start at the origin, since the first point obtained was a positive stress against zero strain.

Obtaining the Stress-Deflection Curves. A correlation was made between the strain-deflection curve and the stress-strain curve, between deflections of 0.360 and 0.600 inch, and plots of

Stress vs. Deflection were made covering this range. These curves are shown in Figure 6, p. 63. To show the effect of creep, one curve was drawn using the original recorded initial strains, the other, using the corrected initial strains.

Preparation of the Salt-Chromate Solutions. Twenty grams of potassium chromate (C.P.) and 35 grams of sodium chloride were weighed to the nearest 0.1 gram and placed in a clean 1000-ml volumetric flask. The solids were dissolved by adding about 500 ml of distilled water, then the flask was filled to the mark. Two such stock solutions were prepared.

Eleven 200-ml samples of the stock solution were placed in sample bottles. The pH of these samples was adjusted with 1.542 potassium hydroxide solution and/or 23 per cent hydrochloric acid to approximately 0.5, 2, 4, 6, 6.5, 7, 7.5, 8, 10, 11.5 and 12. These values were estimated with Hydrion pH papers. Accurate pH values (to the nearest 0.01) for the solutions were established with a pH electrometer. The standard buffer solution used consisted of 40 ml of 0.1N sodium hydroxide solution, 50 ml of 0.1M potassium acid phthalate solution and distilled water to make 100 ml, and was used in contact with a glass electrode. Adjustments of the pH were made by adding acid or base, as before, so that the values would be within 0.25 of the desired pH.

Stress-Corrosion Cracking Tests. Two kinds of tests were carried out. These can be distinguished by the type of curves plotted from the data obtained, which were (1) Stress (maximum tensile stress) vs. Time to Failure and (2) Time to Failure vs. pH (at a constant stress). The specimens were prepared by polishing a portion of about one inch at the center with number 2/0 emery cloth and wiping off the loose particles with a clean cotton cloth. Two specimens (A) were placed in the jig (See Drawing No. 1, p. 43), one near each end, and the adjustable center bar, (B) and (C), was raised until both it and the parallel angle irons contacted the specimens. The Ames dials (H) were set at zero.

The specimens were subjected to bending by simultaneously turning the control nuts with two end wrenches until the deflection corresponding to a desired stress was obtained (as taken from the Stress-Deflection Curve). Parrafin was melted in a small galvanized tray on a hotplate. Two and one-half-inch lengths of 1/2-inch glass tubing (K) were sealed with wax (J) vertically on the specimens at the point of maximum deflection. The lower surface of the specimen adjacent to the center bar was coated with Tygon paint. One-half milliliter of the salt-chromate solution (L) was put in each of the tubes through a graduated pipette. The timer was started as soon as the liquid contacted the first specimen; the solution was put on the second specimen approximately 30 seconds later. When cracking took place, the time recorded was the moment when the first visible crack appeared. The specimens were removed from the jig after cracking was evident across the width of the specimen. Some were left in the jig until they failed completely. Others were removed shortly after cracking began.

Due to relatively rapid evolution of hydrogen from the specimens exposed to pH 2 and 4, 2.0 ml and 1.0 ml of the solution, respectively, were used instead of 0.5 ml samples. After 10 minutes these were replaced with fresh solution. The 0.5 pH solutions were used in 3-ml quantities and the solution were changed every 5 minutes.

Test (1) was carried out with each of the solutions of pH 0.5, 1.92, 3.91, 6.2, 6.6, 6.87, 7.7, 8.25, 10.25, 11.4 and 11.85. Specimens were run in duplicate. The procedure given above for bending the specimens was followed. The deflection applied to the first specimen subjected to pH 1.92 solution corresponded to a stress well above the yield strength. If cracking occurred within a short period of time (usually less than 10 minutes), subsequent runs using the same pH were made at lower deflections, the interval between successive deflections usually being 0.010 to 0.020 inch. This order of runs was made until a deflection yielding no cracking after an extended period of time (greater than 60 minutes) was found. The last two runs, in which a specimen subjected to one stress produced cracking in a short time and a specimen subjected to a slightly lower stress gave no cracking after a relatively long period of time, determined approximately the range of the critical stress. This critical range was more definitely fixed by applying a deflection half way between these last two.

Two or three checks were made of the "boundary" deflections on either side of this critical range. The critical range determined was an interval of 0.010-inch deflection difference or about 150 to 250 psi tensile stress difference (depending upon the magnitude of the stress).

In case the initial run did not give short-time failure, larger deflections were tried until one did produce quick cracking, and the remainder of the procedure for determining the critical range was similar to that given above.

Curves of Maximum Tensile Stress vs. Time-to-Failure were drawn from the data for each pH. These are shown in Figure 7, p. 65. The portion of the curve asymptotic to the abscissa was drawn approximately half way between the points terminating the critical range. A curve, shown in Figure 8, p. 67, was drawn of Critical Tensile Stress vs. pH, the critical stresses being taken as the stresses mid-way between the lowest which produced short-time cracking and the highest which did not. (Assuming a 200-psi critical range, which was the average, the critical stresses determined had a maximum deviation of 100 psi.)

Test (2) involved stressing a number of specimens at the same deflection, exposing them to solutions of pH 0.5, 2, 4, 6, 7, 8, 10 and 12, and finding the time to failure of each. Several specimens were run at each pH. The deflection used was 0.513

inch corresponding to a maximum tensile stress of 30,200 psi or 130 per cent of the yield strength. This arbitrary value was chosen since it was above the critical stresses determined over most of the pH range, and would therefore produce positive results.

Data and Results

In this section the data and results of all experimental work are presented in tabular and graphical form. Each table is followed by a corresponding curve.

Table III gives complete data taken while using the electric strain gage and indicator for the strain-deflection calibration of the jig, along with the calculated initial strains corrected for creep. Data for a check calibration are given in Table IV. Plots of strain vs. deflection (strains taken from columns (F) and (K) and deflections from column (A) of Table III) are presented in Figure 4. The stress-strain data for the alloy are given in Table V and the stress-strain diagram is shown by Figure 5. Table VI correlates stress with deflection from the data of Tables III and V, and the stress vs. deflection curves are presented in Figure 6. The lower curve has been corrected for creep.

Table VII gives all the data and results of the stress-corrosion cracking tests. These data were used in plotting the stress vs. time-to-failure curves presented for various pH values in Figure 7. Critical stresses for various pH values, obtained from Figure 7 and from Table VII, are tabulated in Table VIII and plotted in Figure 8.

TABLE III

DATA AND RESULTS OF STRAIN-DEFLECTION CALIBRATION OF BENDING JIG USING STANDARD TEST SPECIMEN OF MAGNESIUM ALLOY FS-1

	Strain Indicator Dial Readings		Indicator Settings		Strain				
(A) Deflection of Specimen, inches	(B) Initial, micro-inches per inch	(C) Final, micro-inches per inch	(D) Reference Switch	(E) Range Extender Switch	(F) Initial, micro-inches per inch	(G) Final, micro-inches per inch	(H) Creep, microinches per inch	(J) Σ Creep, microinches per inch	(K) Initial Strain, Calculated, microinches per inch
0.000	60	-	6	0	0	-	-	-	-
0.010	240	-	6	0	180	-	-	-	-
0.030	605	-	6	0	545	-	-	-	-
0.040	792	-	6	0	732	-	-	-	-
0.060	1135	-	6	0	1075	-	-	-	-
0.080	1468	-	6	0	1408	-	-	-	-
0.100	1843	-	6	0	1783	-	-	-	-
0.120	1260	-	7	0	2200	-	-	-	-
0.140	1630	-	7	0	2570	-	-	-	-
0.160	1020	-	8	0	2960	-	-	-	-
0.180	1465	-	8	0	3405	-	-	-	-
0.200*	1045	1050	9	0	3985	3990	5	5	-
0.220	1583	1600	9	0	4523	4540	17	22	4518
0.240	1090	1117	10	0	5030	5057	27	49	5008
0.260	1670	1700	10	0	5610	5640	30	79	5561
0.280	760	790	2	A	6155	6185	30	109	6076
0.300	1198	1218	2	A	6593	6613	20	129	6484
0.320	1660	1765	2	A	7055	7160	105	234	6926
0.340	1255	1305	3	A	7650	7700	50	284	7416
0.360	1664	1714	3	A	8059	8109	50	334	7775
0.380	1075	1125	4	A	8470	8520	50	384	8136
0.400	1530	1575	4	A	8925	8970	45	429	8541
0.420	1110	1160	5	A	9505	9555	50	479	9076
0.440	1555	1600	5	A	9950	9995	45	524	9471
0.460	1020	1070	6	A	10415	10465	50	574	9891
0.480	1450	1500	6	A	10845	10895	50	624	10271
0.500	1000	1210	7	A	11395	11605	210	834	10771
0.520	1650	1700	7	A	11835	11885	50	884	11001
0.540	1128	1320	8	A	12523	12715	192	1076	11639
0.560	1717	1795	8	A	13212	13290	78	1154	12136
0.580	1230	1270	9	A	13625	13665	40	1194	12471
0.600	1605	1800	9	A	14000	14195	195	1389	12806

*Creep started at this deflection.

TABLE IV

DATA AND RESULTS OF CHECK STRAIN-DEFLECTION CALIBRATION OF BENDING JIG USING STANDARD TEST SPECIMEN OF MAGNESIUM ALLOY FS-1 COVERING RANGE USED IN CRACKING TESTS (0.400-0.600-INCH DEFLECTION)

Check Calibration										First Calibration*	Deviation	
	Strain Indicator Dial Readings		Indicator Settings		Strain							
(A) Deflection of Specimen, inches	(B) Initial, micro-inches per inch	(C) Final, micro-inches per inch	(D) Reference Switch	(E) Range Extender Switch	(F) Initial, micro-inches per inch	(G) Final, micro-inches per inch	(H) Creep, micro-inches per inch	(J) Creep, micro-inches per inch	(K) Initial Strain, Calculated, micro-inches per inch	(L) Initial Strain Calculated, microinches per inch	(M) Initial Strain, micro-inches per inch	(N) Initial Strain, Per Cent
0.000	1700	-	5	0	-	-	-	-	-	-	-	-
0.400	1800	-	4	A	8555	-	-	-	8555	8541	14	0.16
0.450	1820	-	5	A	9575	-	-	-	9575	9682	-107	1.17
0.500	1980	1112	6-7	A	10735	10867	132	132	10735	10771	-36	0.30
0.550	1390	1520	8	A	12145	12270	125	257	12013	11888	125	1.05
0.600	1420	1500	9	A	13175	13255 +	80 +	337 +	12918	12806	112	0.88
												0.71 Av.

*From Table III, column (K), p. 57.

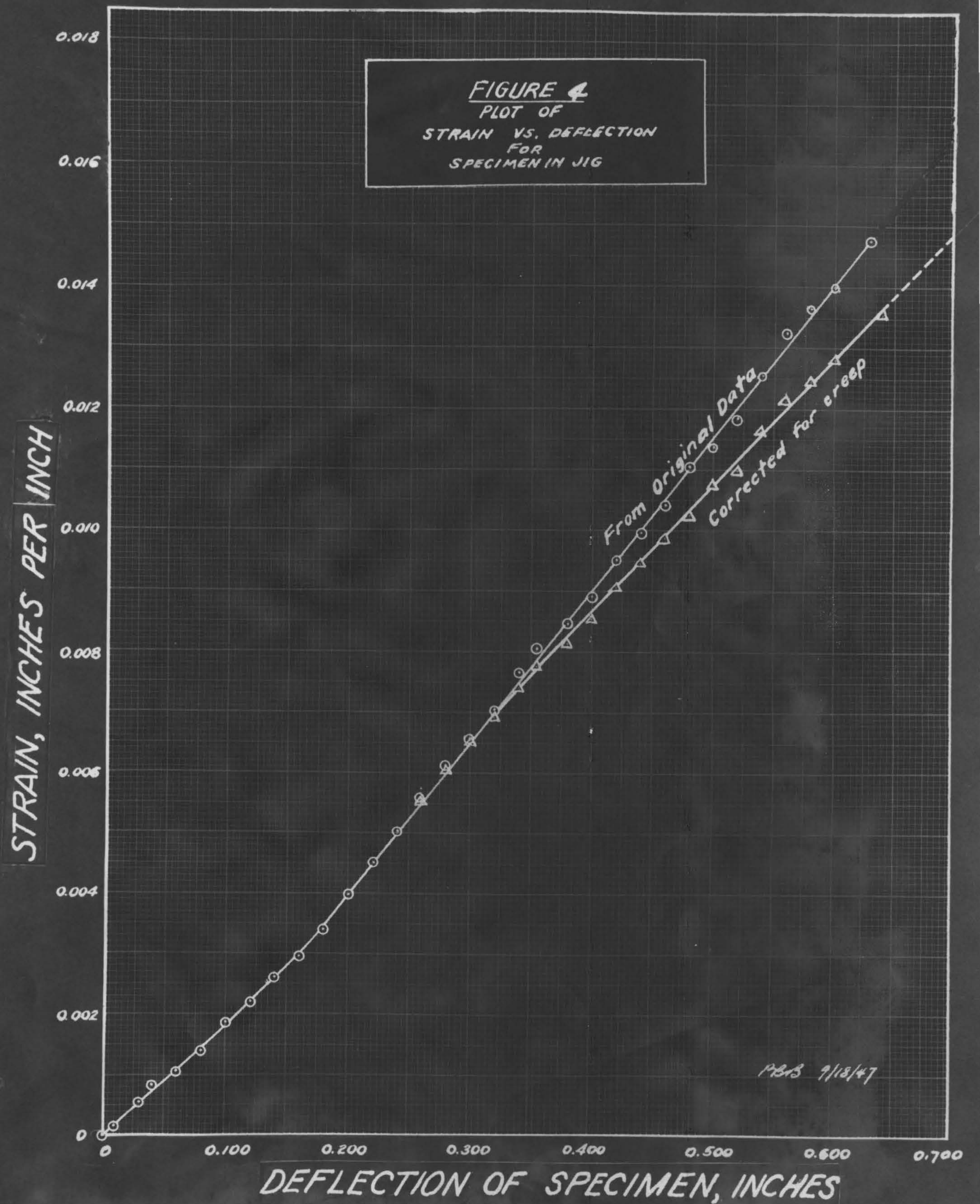


FIGURE 4
PLOT OF
STRAIN VS. DEFLECTION
FOR
SPECIMEN IN JIG

TABLE V

TENSILE STRESS-STRAIN DATA FOR MAGNESIUM ALLOY FS-1*

Applied Force, lb	Tensile Stress, psi	Strain, in. per in.
211	3,400	0.00000
377	6,080	0.00040
494	7,970	0.00055
683	11,000	0.00125
801	12,920	0.00160
900	14,500	0.00205
983	15,850	0.00275
1092	17,600	0.00345
1160	18,700	0.00410
1270	20,480	0.00465
1360	21,920	0.00505
1460	23,550	0.00555
1550	25,000	0.00620
1650	26,600	0.00715
1750	28,210	0.00860
1850	29,820	0.01060
1950	31,430	0.01320
2050	33,050	0.01700

*Standard tension test specimen, having 2-inch gage. Dimensions of reduced section: width, 0.495 in.; thickness, 0.1232 in.; area, 0.0620 sq. in.

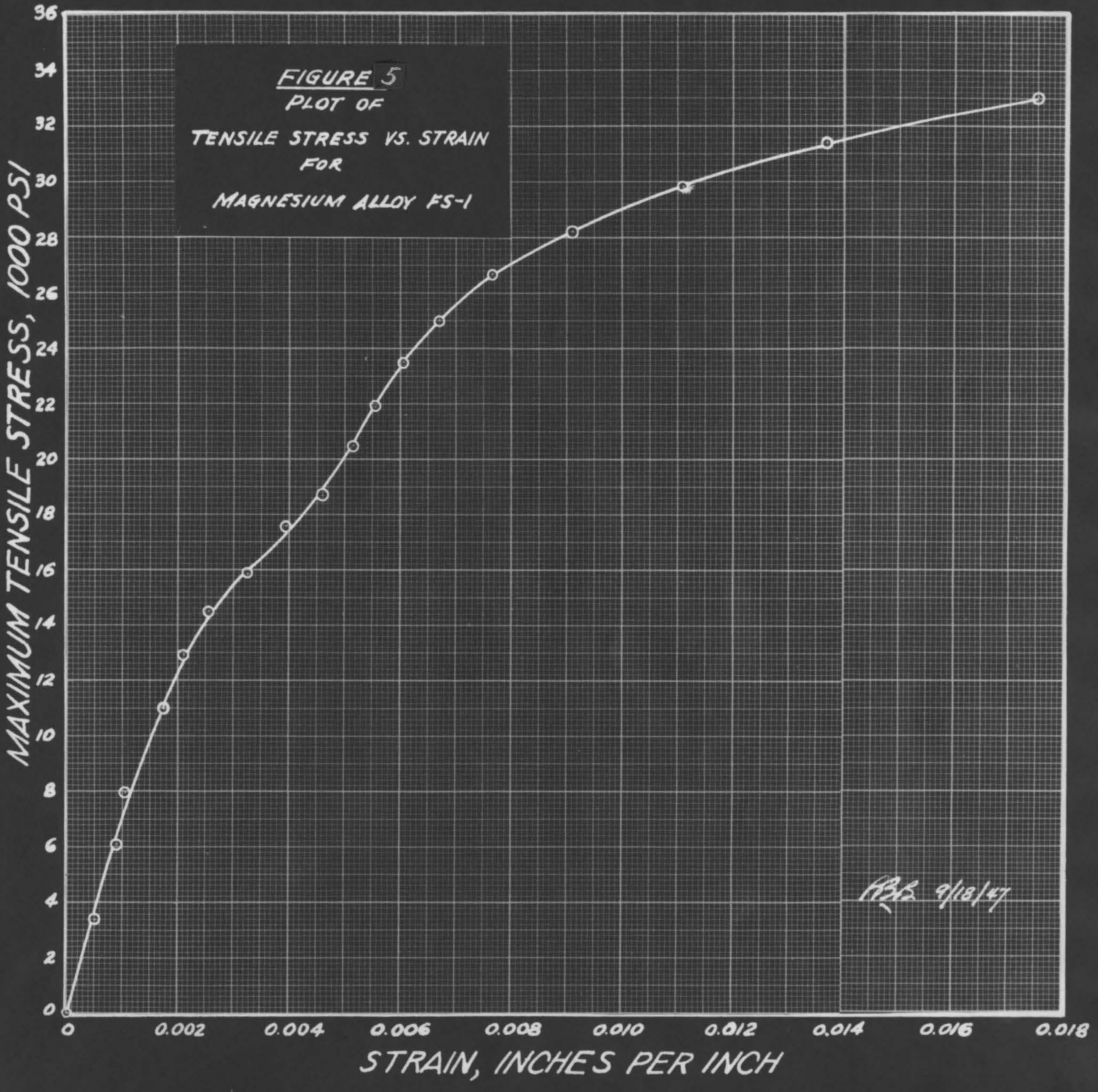


FIGURE 5
PLOT OF
TENSILE STRESS VS. STRAIN
FOR
MAGNESIUM ALLOY FS-1
MAXIMUM TENSILE STRESS, 1000 PSI
36
34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
0
0.002
0.004
0.006
0.008
0.010
0.012
0.014
0.016
0.018
STRAIN, INCHES PER INCH
ABS 9/18/47

TABLE VI

DATA FOR STRESS-DEFLECTION CALIBRATION OF BENDING JIG USING STANDARD TEST SPECIMEN OF MAGNESIUM ALLOY FS-1

	Strain		Tensile Stress		
(A) Deflection of Specimen, inches	(B) Initial Reading, micro-inches per inch	(C) Initial Reading (Corrected) microinches per inch	(D) Initial, psi	(E) Initial (Corrected) psi	(F) Difference, psi
0.360	8000	7700	27,200	26,700	500
0.380	8550	8150	27,700	27,200	500
0.400	9050	8600	28,300	27,700	600
0.420	9550	9050	28,700	28,300	400
0.440	10050	9500	29,000	28,600	400
0.460	10550	9900	29,400	28,900	500
0.480	11050	10350	29,800	29,300	500
0.500	11550	10750	30,100	29,600	500
0.520	12050	11200	30,500	29,900	600
0.540	12550	11650	30,800	30,200	400
0.560	13050	12150	31,100	30,500	600
0.580	13550	12500	31,400	30,750	650
0.600	14000	12950	31,600	31,000	600
					Av.520

FIGURE 6
PLOTS OF
TENSILE STRESS VS.
DEFLECTION
FOR CALIBRATION OF JIG

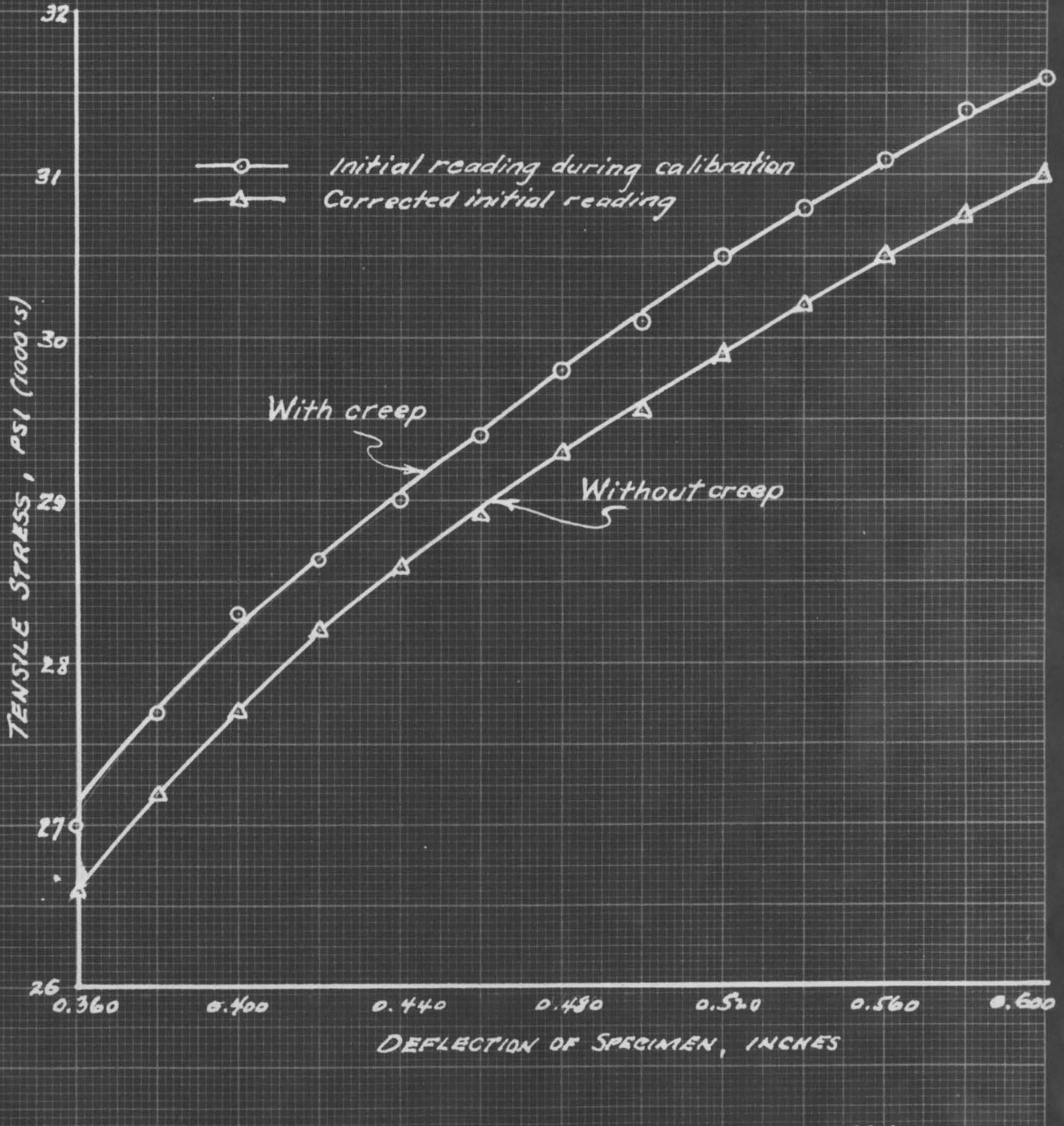


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TABLE VII

DATA AND RESULTS OF STRESS-CORROSION CRACKING TESTS ON MAGNESIUM ALLOY FS-1 IN SALT-CHROMATE SOLUTION

Specimen No.	Solution pH	Deflection of Specimen, inches	Initial Maximum Tensile Stress, psi	Time to Failure, min.	Time Exposed Without Failure, min.	Remarks
159	0.5	0.780	32,600*	1.5	-	Vigorous H_2 evolution
161	0.5	0.730	32,150*	5	-	"
162	0.5	0.720	32,050*	-	180	"
160	0.5	0.700	31,900*	-	60	"
158	0.5	0.640	31,200*	-	60	"
157	0.5	0.560	30,500	-	60	"
156	0.5	0.540	30,200	-	60	"
92	0.5	0.513	29,800	-	180	"
93	0.5	0.513	29,800	-	180	"
1-4	0.5	0.243	**	-	1800	Jig overloaded (27 specimens)
67	1.92	0.513	29,800	8	-	Slight deflection of adjustable bar
68	1.92	0.513	29,800	18	-	"
69	1.92	0.513	29,800	38	-	"
154	1.92	0.513	29,800	3.7	-	
129	1.92	0.440	28,600	3.0	-	
131	1.92	0.440	28,600	-	15	Small cracks evident
133	1.92	0.420	28,200	4.0	-	
151	1.92	0.410	27,950	4.5	-	
152	1.92	0.410	27,950	3.5	-	
153a	1.92	0.410	27,950	3	-	Specimen No. 153 was deflected to 0.410 in.
135	1.92	0.400	27,700	-	55	
142	1.92	0.400	27,700	-	25	
153	1.92	0.400	27,700	-	25	
139	1.92	0.390	27,450	-	1000	
137	1.92	0.380	27,200	-	600	
120	3.91	0.575	30,700	2	-	
121	3.91	0.530	30,050	2.5	-	
70	3.91	0.513	29,800	23	-	Slight deflection of adjustable bar
71	3.91	0.513	29,800	24		"
72	3.91	0.513	29,800	4	-	"
155	3.91	0.513	29,800	2.7	-	
126	3.91	0.480	29,300	1.5	-	
127	3.91	0.460	28,950	2.0	-	
128	3.91	0.440	28,600	3.0	-	
130	3.91	0.440	28,600	3.0	-	
132	3.91	0.420	28,200	4	-	
147	3.91	0.410	27,950	-	70	
148	3.91	0.410	27,950	-	70	
149	3.91	0.410	27,950	5	-	
150	3.91	0.410	27,950	6	-	
134	3.91	0.400	27,700	5.5	-	
143	3.91	0.400	27,700	-	25	
144	3.91	0.400	27,700	13	-	
145	3.91	0.400	27,700	-	100	
146	3.91	0.400	27,700	-	40	
138	3.91	0.390	27,450	7	-	
140	3.91	0.390	27,450	-	100	
141	3.91	0.390	27,450	-	100	
136	3.91	0.380	27,200	-	660	
110	6.2	0.557	30,450	1.5	-	
111	6.2	0.557	30,450	1.5	-	
94	6.2	0.513	29,800	3	-	
95	6.2	0.513	29,800	-	1	
96	6.2	0.513	29,800	3	-	
100	6.2	0.513	29,800	3.7	-	
106	6.2	0.513	29,800	1.5	-	
64	6.2	0.500	29,600	-	20	Numerous cracks detected after test, with 24x microscope.
65	6.2	0.500	29,600	4	-	
66	6.2	0.500	29,600	-	20	Numerous cracks detected after test, with 24x microscope.
101	6.2	0.500	29,600	2.2	-	
102	6.2	0.480	29,300	2.2	-	
105	6.2	0.480	29,300	2.2	-	
108	6.2	0.475	29,200	-	50	
104	6.2	0.465	29,050	-	300	
103	6.2	0.450	28,750	-	1000	
47	6.6	0.550	30,350	5	-	
48	6.6	0.550	30,350	4	-	
49	6.6	0.550	30,350	3	-	
53	6.6	0.525	30,000	10	-	
54	6.6	0.525	30,000	4	-	
55	6.6	0.525	30,000	7	-	
56	6.6	0.513	29,800	4	-	
57	6.6	0.513	29,800	5	-	
58	6.6	0.513	29,800	5	-	
41	6.6	0.500	29,600	-	2640	Minute cracks detected after test, with 24x microscope.
42	6.6	0.500	29,600	4.5	-	
46	6.6	0.500	29,600	-	25	Minute cracks detected after test, with 24x microscope.
59	6.6	0.500	29,600	70	-	
60	6.6	0.500	29,600	5	-	
61	6.6	0.500	29,600	9	-	
9-12	6.6	0.260	**	-	85	
31	6.6	0.243	**	-	1800	
73	6.87	0.513	29,800	-	40	Minute cracks detected after test, with 24x microscope.
74	6.87	0.513	29,800	14	-	
75	6.87	0.513	29,800	7	-	
87	6.87	0.513	29,800	13	-	
62	6.87	0.492	29,500	-	150	
63	6.87	0.492	29,500	-	150	
98	6.87	0.450	29,750	-	720	
99	6.87	0.450	29,750	-	720	
99a	6.87	0.450	29,750	-	720	
50	7.7	0.550	30,350	12	-	
51	7.7	0.550	30,350	3	-	
52	7.7	0.550	30,350	12	-	
43	7.7	0.500	29,600	460	-	
44	7.7	0.500	29,600	-	2640*	Minute cracks detected after test, with 24x microscope. Galvanic corrosion at center load.
163	7.7	0.492	29,500	5.5	-	
169	7.7	0.492	29,500	4.0	-	
164	7.7	0.480	29,300	-	180	
165	7.7	0.480	29,300	-	180	
166	7.7	0.480	29,300	-	180	
167	7.7	0.470	29,150	-	180	
168	7.7	0.470	29,150	-	180	
13-16	7.7	0.243	**	-	1800	
112	8.25	0.557	30,450	3.3	-	
113	8.25	0.530	30,050	2.7	-	
114	8.25	0.520	29,900	1.5	-	
76	8.25	0.513	29,800	17	-	
77	8.25	0.513	29,800	3	-	
88	8.25	0.513	29,800	-	720	One small crack visible.
97	8.25	0.513	29,800	-	30	
107	8.25	0.513	29,800	-	50	
115	8.25	0.513	29,800	2	-	
116	8.25	0.490	29,450	3	-	
117	8.25	0.475	29,200	510	-	
118	8.25	0.460	28,950	-	720	
125	10.25	0.585	30,800	2	-	Very small amount of surface corrosion.
122	10.25	0.575	30,700	2	-	"
124	10.25	0.560	30,500	2.2	-	"
123	10.25	0.550	30,350	-	160	"
119	10.25	0.530	30,050	-	130	"
78	10.25	0.513	29,800	-	1980	"
79	10.25	0.513	29,800	-	1980	"
82	10.25	0.513	29,800	-	720	"
83	10.25	0.513	29,800	-	720	"
84	10.25	0.513	29,800	-	720	"
90	10.25	0.513	29,800	-	150	"
91	10.25	0.513	29,800	-	180	"
17-20	10.6	0.243	**	-	1800	
36	11.4	0.600	31,000	-	180	No surface corrosion.
21-27	11.4	0.243	**	-	1800	"
172	11.85	0.790	32,600*	-	60	"
171	11.85	0.750	32,300*	-	60	"
170	11.85	0.700	31,900*	-	60	"
80	11.85	0.513	29,800	-	1980	"
81	11.85	0.513	29,800	-	1980	"
85	11.85	0.513	29,800	-	720	"
86	11.85	0.513	29,800	-	720	"

*Tensile Stress extrapolated from Figure 6, p. 63.

**Tensile stress values at dip in Stress-Strain Curve, therefore not dependable. See Figure 5, p. 61.

FIGURE 7

PLOTS OF

MAXIMUM APPLIED STRESS

VS

TIME TO FAILURE

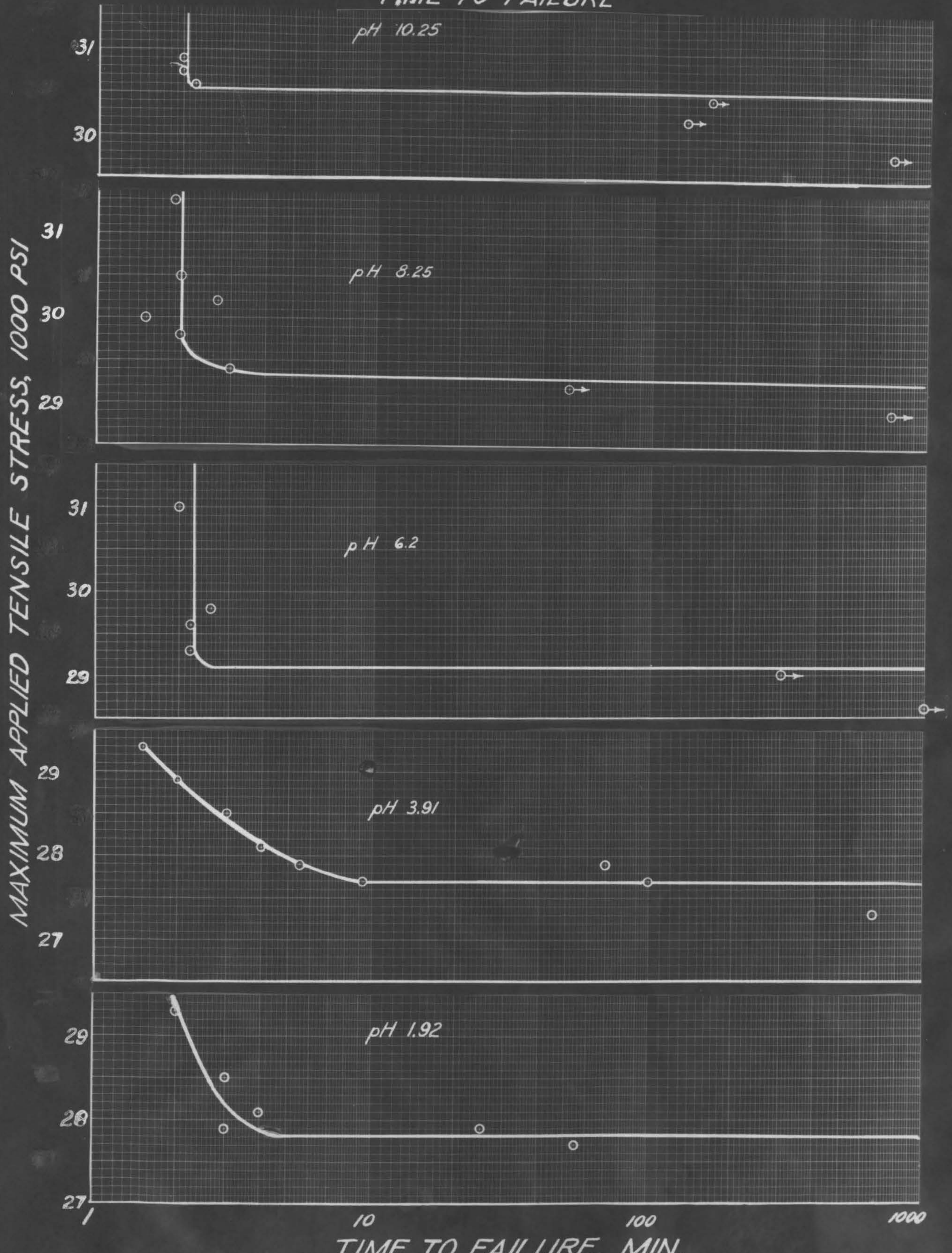

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TABLE VIII

THE EFFECT OF pH ON CRITICAL STRESSES FOR STRESS-CORROSION CRACKING OF MAGNESIUM ALLOY FS-1 EXTRUDED BAR IN SALT-CHROMATE MEDIUM

Solution pH	Critical Stress, psi
0.5	32,000
1.92	27,900
3.91	27,700
6.2	29,300
7.7	29,350
8.25	29,200
10.25	30,350
11.85	32,600

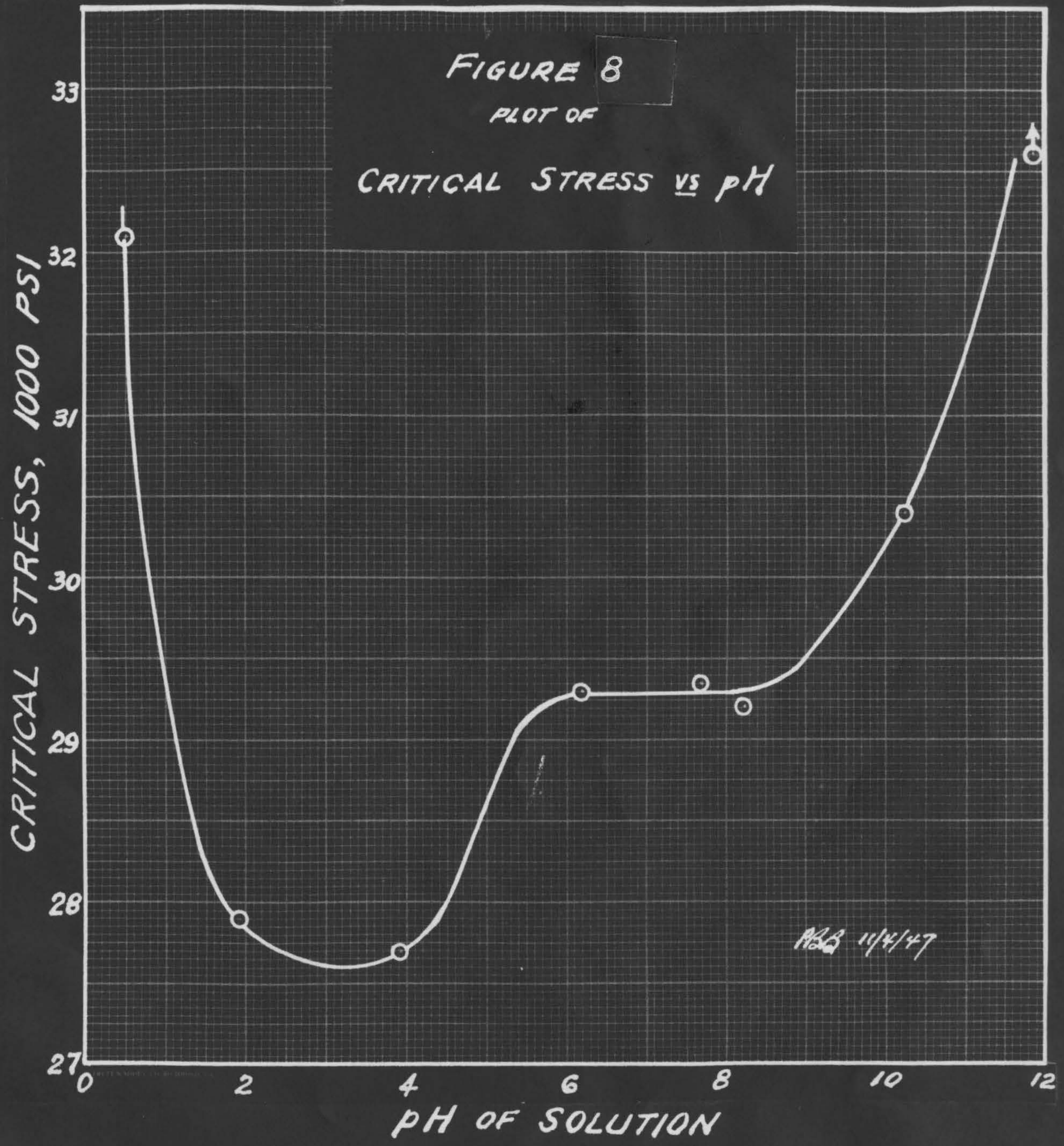


FIGURE 8

PLOT OF

CRITICAL STRESS vs pH

Sample Calculations

(1) Calculation of strain from strain indicator readings and settings (see Table III).

$$\text{Strain (in microinches)} = X + (R_2 - R_1)1000 + D_2 - D_1$$

where X = a constant for change in range extender switch setting (for a change from 0 to A, $X = +9455$; from 0 to B, $X = -9455$)

R_1 = Initial reference switch setting
R_2 = Final reference switch setting
D_1 = Initial dial reading
D_2 = Final dial reading

At a Deflection of 0.280 inch:

$X = 0 \text{ to } A = +9455$
$R_1 = 6$
$R_2 = 2$
$D_1 = 60$
$D_2 = 760$

$$\text{Strain} = +9455 + (2-6)1000 + 760 - 60$$

$$= 9455 - 4000 + 760 - 60$$

Strain = 6155 microinches

(2) Calculation of initial strain, calculated (or corrected). (See Table III, column K).

Initial strain, calculated (in microinches) = Final strain (column G) - cumulative creep (column J)

At a Deflection of 0.280 inch:

Initial strain, calculated = 6185 - 109
= 6076 microinches

(3) Calculation of tensile stress. (See Table V).

$$\text{Stress (psi)} = \frac{\text{Applied load (lb)}}{\text{Cross-sectional Area (sq in.)}}$$

For an applied load of 900 lb:

$$\text{Stress} = \frac{900}{0.0620} = 14{,}500 \text{ psi}$$

IV. DISCUSSION

A. Bending Jigs

Apparatus Requirements. As in any investigation, the objects of this work demanded certain requirements in the design of the apparatus. The two main requirements to be met were, first, that the tensile stress applied to a specimen be variable over a wide range, and second, that this applied stress be measurable. Other requisites were (1) that an inert material of construction or a satisfactory protective coating be used for the apparatus if total immersion tests were to be made; (2) that the jig be capable of handling several test pieces at a time, since approximately 200 specimens were to be tested, excluding numerous trial test specimens; (3) that loading the jig be simplified as much as possible; (4) that stress-corrosion cracking of a specimen be detectable at the moment it occurred; (5) that the various factors affecting general corrosion be controlled, viz. temperature, velocity and aeration; and (6) that the material of construction be sturdy enough to withstand the forces developed.

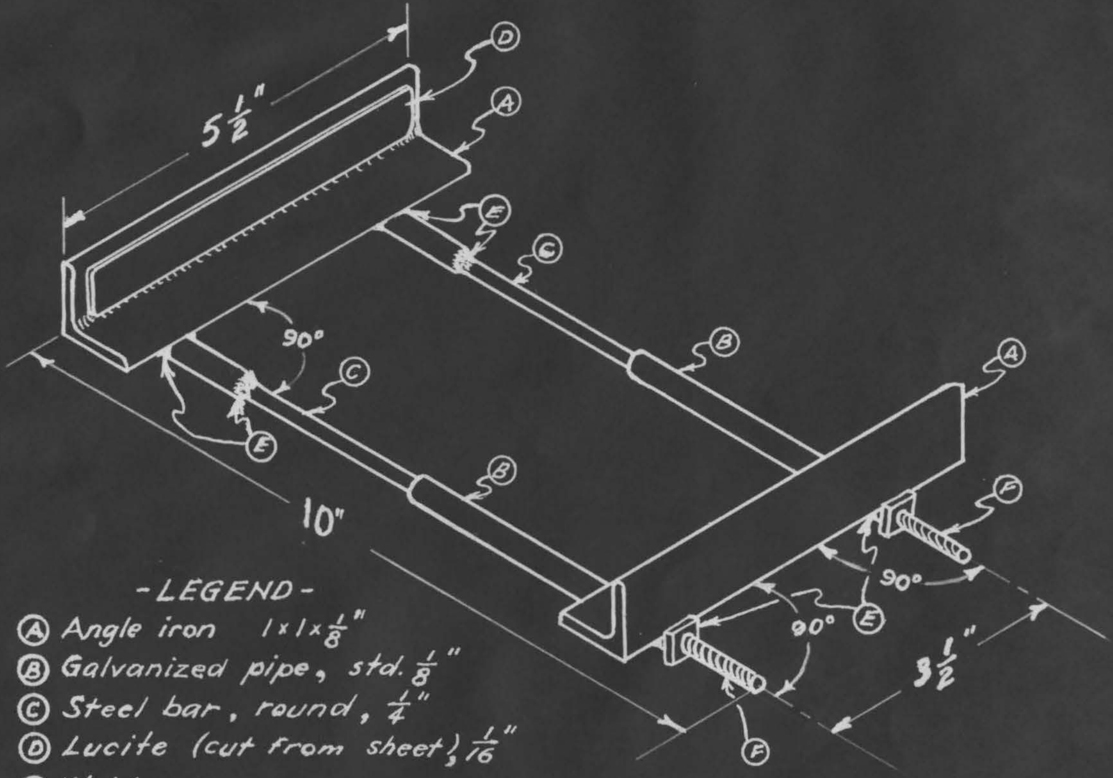
Bending vs. Direct Tension Methods of Stressing. In order to meet the two main requirements it was at first thought that stressing the pieces in direct tension would be satisfactory. This method, however, would involve bulky and burdensome apparatus.

For instance, a single specimen of FS-1 alloy loaded to its yield strength of approximately 22,000 psi would require a total force of about 1,400 lb. Loading with dead weights would require 1,400 lb of weights or an equivalent quantity in case a system of levers was employed. Spring loading would require a heavy frame as well as a large spring. Construction of equipment to supply such loads was impractical, since only one or two specimens could be tested in each unit. On the other hand, subjecting the pieces to bending stresses held much in its favor; since the problem of bulky apparatus could be eliminated, a number of specimens could be tested at the same time, and the method of loading the specimens could be made rather simple. For these reasons the bending type of apparatus was decided upon.

Original Bending Jigs. The bending jig which was used in this investigation was constructed only after another type was tried and found to be inadequate. The original jig was designed for bending the specimens under axial end loads. It was to be adjustable so that any degree of tensile stress might be applied and to be suitable for immersion testing. As shown in Drawing No. 2, p. 71, the jig was constructed of two pieces of angle iron, between which the specimens were bent, and two, 1/4-inch rods threaded at one end and placed through short lengths of 1/8-inch standard galvanized pipe, which were welded to the angles at 90°, and parallel to each other. The unthreaded ends of the rods were welded to the pipe; the threaded ends were left free to move.

DRAWING No. 2

ORIGINAL BENDING JIG



-LEGEND-

Ⓐ Angle iron 1 x 1 x 1/8"
Ⓑ Galvanized pipe, std. 1/8"
Ⓒ Steel bar, round, 1/4"
Ⓓ Lucite (cut from sheet), 1/16"
Ⓔ Weld
Ⓕ Threaded

PBB 11/18/47

The welds were ground off smooth on a grinding wheel. Nuts were screwed on the rods to adjust the span distance between the vertical faces of the angles. Six of these frames were made. Each was given an acid pickling treatment of 30 minutes in 6 per cent by weight hydrochloric acid at 120° F., followed by limewater and tap water dips, drying with a cotton cloth, and final drying over a hotplate.

Measurement of Stress in Original Bending Jigs. When the original jigs were constructed, it was thought that the applied maximum tensile stress of a specimen, bent to a known deflection or span distance, could be calculated with a suitable formula. All ordinary formulae, however, are based on the assumption that the material is stressed within its elastic limit, which was not the case in this investigation. Magnesium alloys are different from nearly all other alloys in that most of their compressive yield strengths are considerably lower than their tensile yield strengths. The FS-1 alloy has a tensile yield strength of 22,000 psi and a compressive yield strength of 13,000 psi (minima). This would mean that, in bending, the inner surface of a piece could be strained within the plastic range while the outer surface was still stressed within its elastic limit. Such a condition was expected in this work, since tensile stresses approaching the yield strength were anticipated. Therefore, ordinary formulae could not be employed for calculating the applied stresses, and a direct measurement was required.

Measurement of bending stresses, due to axial loading, depend upon very exacting conditions for accuracy. Slight discrepancies between the physical conditions of two similar test specimens and the way they are placed in the bending jig have a magnified effect on the resulting maximum stresses. In the original jig, it was difficult to put each specimen between the jaws in exactly the same manner without adjusting them after bending, since the ends were not securely held in a fixed position. Also the jigs were not made with the precision desired, nor were they machined; the jaws were not exactly parallel. When the jigs were loaded with three specimens, the rods tended to bow slightly. It was decided that all these factors would bring about erroneous and inconsistent results.

B. Protective Coatings Used on Original Bending Jigs

Silicone Treatment. Five of these jigs were given protective coatings of 40 per cent by weight DC 804 silicone resin in toluene, containing 0.30 per cent cobalt naphthanate dryer based on the solid resin content. The silicone treatment consisted of applying three brush coats of DC 804 resin to all portions of the jigs except the threaded ends of the rods. The first coat was thinned to 20 per cent resin by weight with toluene, so that the resin would impregnate the tiny crevices and pits which were present in the metal after grinding the welds and pickling the surface. Each coat was followed by a 4-hour air-drying period and a 16 to 20-hour baking period in an infra-red drying oven. The approximate temperature in the oven, as measured by a bimetallic Weston thermometer placed above the specimens and inside the dryer, was 450° F. After baking, the first coating put on the specimens appeared to be continuous when viewed under a stereoscopic microscope (24x). However, the second coats would not completely wet the surface, leaving voids of 1/16 to 1/8-inch in diameter, with the first coat exposed. Since the surfaces were kept perfectly free from grease, lint and other contaminants, the non-wetting effect was probably due to lack of surface adhesion between the polymerized silicone surface and the unpolymerized liquid resin. It was also noticed that the surfaces which were exposed normal to the direct rays of the lamp in the oven were also those which would not take continuous second or third coats. It seems possible that these surfaces could have received a higher degree of polymerization due to a higher temperature existing on them because of the perpendicular exposure, and that this more highly baked portion would not offer a sufficient attraction for adherence of the liquid.

Tygon Treatment. The sixth jig was given three brush coats of the "Tygon" paint. The first coat was thinned to approximately 50 per cent by weight of the original paint (as received), and the other two were thinned to approximately 80 per cent. An air-drying period of about 1 to 2 hours was allowed between applications.

Resistance of the Silicone Coating to a Salt-Dichromate Solution. When three specimens were sprung into a coated jig and the jig was exposed to a medium of 20 grams per liter of potassium dichromate and 35 grams per liter of sodium chloride (pH approximately 3) for 24 hours at a temperature of 125° F., none of the coatings held up satisfactorily. The silicone coatings, after exposure, were affected to such an extent that the top coat could be flaked off with the fingernail. This indicated again the lack of adhesion between successive coats of the resin. The base coat, for the most part, could be removed easily with a knife, although in some portions adherence with the metal was very good. It was obvious that the solution had penetrated the coatings, possibly through tiny pinholes which could not be detected under the microscope. The force of the specimens against the jig broke the coatings, causing subsequent galvanic corrosion at the ends of the specimens. The coatings appeared to be discolored, especially at the areas where they could easily be flaked off.

Resistance of the Tygon Coatings to a Salt-Dichromate Solution. The Tygon coatings also became discolored, yellowing, indicating some absorption of the solution. The formation of blisters ranging from about 1/8 to 3/4 inch in diameter was apparent on the Tygon-coated frames. This was probably caused by some acetone remaining in the paint due to incomplete drying. The specimens had broken through the coatings, but a coating applied at these contact surfaces prevented any galvanic action.

The silicone coatings were scraped off the frames, and the frames were then given three dip coats of "Tygon" paint. Six-hour air drying was allowed between coats. Strips of 1/16-inch Lucite sheet were then attached to the angle irons (with acetone) to act as bearing surfaces for the specimens. These coatings held up very well against the salt-dichromate solution, but the pressure of the specimen forced the Lucite out of position. The plastic was also too soft to provide an adequate bearing surface, and would not stand up under continued use.

Chemical Resistance of Valdura "Sarva". Still another specimen holder was cleaned of the silicone coating and given one dip coat of Valdura "Sarva" corrosion-resistant black paint and allowed to air dry for 72 hours. This offered poor resistance to the salt-dichromate solution and the coating broke down in many places. The coating became hard and brittle after drying, cracking in many places.

C. Selection of the Final Method

Reasons for Discarding the Original Method. The reasons for discarding the original method of testing and selecting some other type of apparatus have been discussed in the foregoing paragraphs. They may be summed up as follows:

1. The jigs lacked the precision required for accurate results in axial loading.

2. Protective coatings that were used did not hold up well in the few tests given them. Bearing surfaces, particularly, were subject to coating failure.

3. The jigs were not sturdy enough for continued testing.

Reasons for Selecting the Final Method. Since the original method of testing had been proven inadequate, it was necessary to devise some other type of apparatus and method of procedure which would produce accurate results. The difficulties inherent in the axial-loading methods of stressing could be eliminated by subjecting the specimens to transverse loads, so it was decided that the latter type of loading would be used. Variable stress could easily be applied to a specimen by loading it at three points with an adjustable center load and two fixed end loads. This would also hold the specimen securely in position while tests were being carried out. Ames indicators could be used for measurement of deflection, and with the use of an electric strain gage attached to the points of maximum stress, a curve of strain vs. deflection could easily be plotted. Correlation between this curve and a tensile stress-strain curve for the alloy would give a direct relationship between any indicated deflection and the applied stress. The problem of protective coatings could be eliminated by providing no contact between the corrosive medium and the jig itself. Therefore, it was decided to contain the corrosive medium in a short piece of glass tubing sealed to the specimen at the point of maximum tensile stress.

D. The Final Method Used in This Investigation

The Final Bending Jig. The jig used in carrying out the stress-corrosion cracking tests served its purpose very well. Specimens could easily be placed in the jig and almost immediately be given the desired stress. It is believed that the jig can be used for testing thin samples of any metal or alloy which does not require a large volume of solution for its tests. One difficulty was encountered in the operation of the jig: the adjustable bar was not heavy enough to resist bending when loaded with more than six specimens, three placed near each end. Also, there was a slight convergence between the two angle irons, and between the angle irons and the adjustable bar. This, however, was not thought to be serious since duplicate results could be obtained by testing specimens near either end of the bar. About seven inches of the bar at the left end sloped upward, approximately 1/8 inch, and this portion of the jig was, therefore, useless. It is believed that welding caused this bend in the bar.

Advantages of Limiting the Solution-to-Metal Contact Surface. The advantages of localizing the corrosion as was done with the glass tubes in this investigation are believed, in final analysis, to outweigh the disadvantages. The method, however, would not be recommended for tests of surface corrosion. The advantages are:

(1) Confinement of the corrosive action to the area at which the stress is known.

(2) Proper selection of tube diameter can keep the edges out of contact with the liquid. Due to the bending, the outer surface of the specimens become concave, causing an unknown concentration of stress on the edge fibers.

(3) No chance of a high stress differential causing accelerated corrosion can exist, since the fibers in the exposed area are all subjected to very nearly the same stress.

(4) Cracking can readily be detected at the outset, and its procedure can be watched.

(5) Elimination of protective coatings required on the bending apparatus.

<u>Possible Disadvantages of Limiting the Solution-to-Metal Contact Surface</u>. Some of the possible disadvantages of the final method are discussed below:

(1) <u>Lack of velocity control</u>. In the accelerated tests the rate of hydrogen evolution was high with solutions having pH 4 and below; less, with pH between 6 and 8; and almost nil, for pH above 8. The velocity of the liquid was mainly a function of hydrogen gas evolution, although convection currents may have given a

slight motion to the apparently quiet solutions. It is not believed that velocity is a controlling factor in stress-corrosion cracking, since only the minute quantity of metal at the base of the crack is attacked in the process, and that as separation of the grains takes place, bare metal is continuously exposed and corrosion proceeds. It is believed that some degree of motion of the liquid is necessary for cracking, but that the minimum velocity required is extremely low. This minimum flow can possibly be initiated by the movement of the metal as the crack opens, or by hydrogen gas bubbles passing up through the crack. Velocity in the bulk of the solution would not necessarily disturb the portion contained in the capillary of the crack. It appears also that the degree of stress and the nature of the solution are so great in controlling susceptibility to cracking, that the effect of velocity is thereby minimized. Another point is that a quiet solution would produce less chance for formation of concentration cells than would one in motion, therefore increasing the surface corrosion which would decrease the possibility of stress-corrosion cracking.

(2) <u>Lack of control of aeration</u>. The dissolved oxygen contents of the solutions were unknown. It is thought that they were near saturation since all the solutions were shaken during their preparation. The samples were run into the tubes with a pipette (for the tests) and air was mixed into the liquid at that time also. However, they may have been subjected to temperatures

high enough to cause a loss of oxygen during the time between preparation of the solutions and the tests. Oxygen would hardly affect the corrosion in the solutions of pH 8 and below, since hydrogen in these solutions is readily discharged as gas. Although aeration greatly stimulates most corrosion, it has practically no effect when sufficient chromate is present to inhibit(96), since the chromate ion acts as a hydrogen acceptor. From these facts it would seem that testing in an unaerated solution has more merit than testing in an aerated one.

(3) Leaks. Leaks developed between the wax seals, at the bases of the solution-containing vials, and the test specimens. This occurred after extended exposure (15 hours or more) regardless of the pH of the solution.

(4) Low solution-volume-to-metal-surface-area ratio. As in the case of velocity, the ratio is believed to be unimportant where the solution pH is 3.91 or higher, because of the small amount of corrosion products present to contaminate the solution. Before the tests were made, this was somewhat proved by wiping a thin film of the 6.2 pH solution over a stressed specimen; cracking of the piece took place in less than five minutes. After extended periods of time, however, the small volume of solution was certainly increased due to evaporation. This latter condition might have distorted some of the results. Surface corrosion products did contaminate the solutions of pH 1.92 and 0.5, requiring frequent additions of fresh solution.

(5) Lack of close temperature control. All tests were carried out at room temperature, which was 80° ± 5° F. No information

or data indicating the effect of temperature of the salt-chromate tests was found in the literature(60)(61)(71). Most of the previous investigations were also carried out at room temperature, although the temperatures were not given.

Effect of General Corrosion. General surface corrosion, as distinguished from stress-corrosion cracking, had a definite effect on retarding the progress of cracking. Some of the specimens which showed no visible signs of cracking during the runs and were recorded in the data as not failing, were later examined under a 24x stereoscopic microscope and found to contain numerous minute cracks, all perpendicular to the direction of stress. In every case the stress was above the critical for the respective pH. Surface corrosion was also present, mainly localized along scratches caused by the longitudinal polishing before testing. Many of the tiny cracks terminated at these lines of surface corrosion, apparently unable to pass through them. It may be surmised that this cracking would have resulted in failure had the surface corrosion been absent, and the cracks allowed to proceed unhindered. As the number of small cracks increased, the stress concentration at the base of each was, in all probability, correspondingly lowered. This lowering of stress concentration limited the chances for failure. Also, from a pH of 2 to a pH of 0.5, where surface corrosion was excessive, the critical stress vs. pH curve increased very sharply from about 27,600 psi to 32,100 psi. These effects agree with the literature(5)(72) on the subject.

Description of the Failures. The onset of cracking was evident during the tests when the evolution of hydrogen bubbles took place along a line perpendicular to the direction of stress. Gradually, the crack increased in depth and width, with continuous hydrogen evolution, until complete failure resulted. Usually only three or four cracks were formed, rarely as many as six or seven. Occasionally, when the liquid covered the entire width of the surface, the first crack started at the edge of the specimen where a relatively high, unknown stress existed. This effect would tend to mask the results since the stresses at the edges of the specimens were unknown.

Metallographic Tests. An attempt was made to determine whether the failures were transgranular or intergranular by metallographic examination. However, the cold work effects caused by either extruding (during manufacture), or bending past the yield strength during the tests, distorted the grains too much to allow any conclusions to be drawn as to which path the cracks followed. Preparation of the samples for examination involved cutting out the failed portions of specimens which had been subjected to solutions of pH 1.92, 6.85 and 10.25; casting them in a Bakelite mold under hydraulic pressure; cutting the mold and samples down in a lathe; polishing with emery papers Nos. ½, 1, 2, 0, 00 and 000 (in this order) using a gasoline-paraffin lubricant, and finally on a wheel covered with Vel-Chamee cloth using a powdered alundum (grain size 600)-water abrasive mixture; and etching, rinsing in distilled water, and drying in a blast of compressed air. (The air itself was not completely dry, however, for water vapor was seen in the blast.) Etches used prior to several examinations were:

(1)	Diethylene glycol	75%, by volume
	Distilled water	24
	Concentrated nitric acid	1
(2)	Citric acid	5 g.
	Distilled water	95 cc
(3)	Glacial acetic acid	50%, by volume
	Distilled water	50

None of these etches proved satisfactory. They were given inadequate trial, however, since the attempts were stopped after the cold-work effect became apparent. The possibility of the cold-work distortions being due to the polishing procedure was eliminated, since they were still evident after about 1/100 inch of the metal surface was dissolved with nitric acid (concentrated acid diluted 1:1 by volume with distilled water). The visible examinations of the samples were made under a metallographic microscope (100x and 500x).

<u>Effect of pH on Critical Stress</u>. Figure 7, p. 65, shows the relationship obtained between pH and stress-corrosion cracking limits, or critical stress, for standard test specimens of magnesium alloy FS-1 extruded rectangular bar (3/4 x 1/8-inch cross-section). Specimens were bent in constant deflection as end-supported, center-loaded beams; and were exposed to a solution of

35 grams per liter of sodium chloride and 20 grams per liter of potassium chromate. The constant critical stress of 29,250 psi existing in the curve between pH 6 and pH 8 indicates that neither slight acidity nor slight alkalinity has appreciable effect on the susceptibility to attack under these conditions of exposure. However, it is apparent that solutions above pH 9 and solutions below pH 2 tend to decrease the susceptibility to stress-corrosion cracking. From pH 9 to pH 11.3 (approximately), the critical stress increases to 32,600 psi; from pH 2 to pH 0.5 this stress increases from 27,800 psi to 32,100 psi. It is possible that above pH 12 this extruded alloy is immune to cracking, since the curve appears to become asymptotic to this ordinate. This increased resistance in highly basic solutions would be expected since both the chromate and hydroxide films are more protective against alkaline environments.

The higher critical stress at very low pH agrees with the findings in other investigations(24)(61), which show that surface corrosion inhibits the susceptibility to stress-corrosion cracking. In the tests carried out using a solution of pH 0.5, surface corrosion was very rapid; this was evidenced by vigorous evolution of hydrogen gas, discoloring (darkening) of the solution, an obvious decrease in the thickness of the specimen, and large amounts of corrosion products covering all of the exposed area. The possibility of the corrosion products protecting the metal from stress corrosion was discounted when two stressed specimens which had not cracked after a 60-minute exposure in a solution of pH 0.5 were caused to fail within two minutes upon exposure to a solution of pH 3.91 at the same stress (the stress was 31,200 psi in both specimens).

The fact that the critical stress begins to decrease below pH 6, rather than at some other pH, is probably due to the fact that the protective hydroxide film is destroyed below this pH. The increase of critical stress beginning about pH 9, rather than at some other pH, might be due to the fact that pure chromate (without the dichromate) exists at a concentration of 35 grams per liter(94) and that this ion is more protective than the dichromate ion, which exists with the chromate below pH 9.

Minimum Critical Stress. The greatest susceptibility of the alloy to stress-corrosion cracking under the conditions of exposure used in this investigation was at a pH of about 3. This minimum critical stress is 27,600 psi, and the minimum critical strain is 0.008500 inch. Considering a tensile yield strength of 22,000 psi for the alloy, this is 125 per cent of the yield strength. Other investigators(54) found that for annealed FS-1 sheet alloy the critical stress for direct-tension tests in the salt chromate medium was about 100 per cent of the tensile yield strength (22,000 psi).

Effect of Creep in Stress-Deflection Diagram. Creep in the specimens was first detected when data for the strain-deflection diagram were being taken. At a specimen deflection of

0.200 inch, the strain reading increased slightly (5 microinches), although the deflection was held constant. At higher deflections the rate of this elongation was increased. The rate of creep was not quantitatively determined, but it was evident from observation that it was increased by higher deflections. Since the strain readings were taken during stepwise increases of deflection, it is apparent that each successive reading of strain included the total value of creep obtained in the previous steps. Therefore, in order to obtain the corrected value for initial strain, the final strain reading at each deflection was decreased by the summation of creep at that deflection plus creep at all previous deflections. The initial strain values thus obtained are slightly higher than actual values, since the creep which took place during the operations of bending the specimens from one deflection to the next and of balancing the strain indicator for a reading, was not taken into account. It is believed, however, that this slightly higher value for strain would be closer to that actually present in the specimens at the time of making the stress-corrosion cracking tests than would the true initial values, since creep was taking place during bending of the specimens and in sealing the solution containers on them. In order to determine accurately the true strain on the specimens at the time of applying the solutions for the cracking tests, it would be necessary to obtain relationships between creep and time for each initial strain.

Stress-Strain Curve. The stress-strain curve obtained for the test specimens is characterized by a dip between strains of 0.0025 and 0.0070 inch. Data for two other curves were taken to determine whether or not some error in data-taking or in testing procedure had been made; but these curves also contained a dip. This would seem to indicate that the structure of this alloy gives it the ability to lose strength at certain strains but to regain its strength when the strain is increased. This is not uncommon in metals, although the property is not usually exhibited until after the yield strength is passed. In this test, the dip occurred at a range which prevented accurate determination of the yield strength, therefore the generally accepted minimum of 22,000 psi was taken for this value.

Recommendations

The following recommendations are offered for future investigations:

Atmospheric Exposure Tests. In order for accelerated tests to be of value, data obtained from them must be correlated with that obtained from exposure to other environments, especially service environments. Atmospheric exposure of the FS-1 extrusions is recommended since it is the most common type of exposure encountered in the use of these alloys. No suggestions are made as to the method of stressing to use but it is suggested that several specimens be exposed at the same time. These specimens should be subjected to a range of stresses from the highest critical stress obtained in this work (32,200 psi) down to 30-40 per cent of this critical stress. The critical stresses for weather exposure of FS-1 sheet have been found to be much lower than those required in accelerated tests on this alloy.

Heat Treatments. In conjunction with the atmospheric tests, it is recommended that the effects of various heat treatments on increasing the critical stresses be examined, since annealing is known to reduce stress-corrosion cracking susceptibility. The reports of investigations by Hunter and his co-workers(51) are recommended for reference in connection with work of such a nature.

Tests on Welded Specimens. Stress-corrosion cracking tests on welded magnesium alloy FS-1 extrusions are recommended, since welding is known to increase the susceptibility to cracking. Welding should be done by welders who are experienced in magnesium welding technique. Poor and inconsistent results in welding cannot be tolerated, since these would add unknown variables to the experimental results. Pitting due to flux inclusions and incomplete melting of the welded joint are two of the more prominent difficulties which should be prevented. Weld tests should be carried out in both the accelerated and atmospheric media. Heat treatment of the welds in preventing stress-corrosion cracking should be examined, since this is considered to be the most effective preventive method(28)(30)(50)(51)(54)(59)(61)(65)(69).

Further Use of the Bending Jig. If further use of the bending jig is made, it is recommended that the adjustable bar be replaced by one possessing greater resistance to bending in order that a greater number of specimens might be tested at the same time. The present bar would not resist bending when loaded with more than six (three near each end) of the standard test specimens of magnesium alloy FS-1 used in this investigation, when deflected to about 0.400 inch cr more.

Investigation of the Creep-Time Relationship. A relationship between creep (elongation of the surface stressed in tension of the specimens under constant deformation or constant stress) and time should be made, for in this investigation and in others(54) it was found to have an important bearing on the accuracy of the results. A higher degree of accuracy in determination of strain at the time of exposure to the corrosive medium would be obtained if (1) the time at which each strain reading is taken during a strain-deflection calibration is recorded, and (2) creep vs. time curves are obtained for various constant deflections within the range of deflection used in this investigation, and (3) in the cracking tests, the time interval between attaining a deflection and placing the corrosive medium in contact with the metal, be recorded. The working range is not definitely known until after the stress-corrosion cracking tests are made, therefore it is recommended that the creep vs. time curves be obtained only after sufficient experimental work has been carried out. A correlation could be made between the above relationships so that the exact strains applied to a specimen at (1) the time of first contact between the corrosive medium and the specimen, and (2) the time of cracking would be known.

Adoption of the Bending Jig as a Standard Apparatus. The bending jig was capable of producing any tensile stress between 0 and 32,600 psi and any tensile strain between 0 and 16,600 microinches per inch. This maximum stress is near the ultimate strength of the alloy (36,000 psi) and well above most design stresses.

The strains at any deflection were known by use of the previously obtained strain-deflection curve, the accuracy of the strains depending upon the accuracy of the equipment used for measuring the strains and the indicators for measurement of deflection. These apparatus used in this investigation were precision instruments. The stresses at any deflection were determined by use of a previously drawn stress-strain diagram for the alloy in conjunction with the strain-deflection curve. The accuracy of the stresses was therefore dependent upon both the accuracy of the testing machine and the accuracies of the strain- and deflection-measuring equipment. Any determination of bending stresses near the tensile yield strength in magnesium alloys requires this correlation since their compressive yield strengths are lower than their tensile yield strengths, causing ordinary stress formulae to be inadequate.

The jig itself was inexpensive, simple to construct, and simple to operate and control. With a sturdier center bar, it could be used for stressing about twenty-five specimens of 3/4-inch width for a single loading. A center bar could be stair-stepped, with individual bars placed across each flight, so as to deflect several specimens different amounts. This bar could be made interchangeable with the straight bar. Specimens of larger cross-sections could be tested if heavier bars and angle iron were used. The larger jig could be made according to the same design, however.

Because of these advantages of the bending jig and since it is believed that no other type of jig described in the literature(51)(54)(59)(65) is any better suited for stress-corrosion cracking testing in bending, it is recommended that this basic design be adopted as a tentative standard apparatus for accelerated laboratory stress-corrosion cracking tests of magnesium alloys by the American Society for Testing Materials.

Limitations

Test Specimens. The only specimens tested were standard ASTM tension test specimens eight inches long, having a gage length of two inches. These were cut from commercial magnesium alloy FS-1 rectangular extruded bar having a cross-section of 1/8 x 3/4 inch. It was originally planned to test sixty welded specimens, but the welding job on the pieces was very unsatisfactory. Apparently the flame used in welding was not hot enough, since incomplete melting of the metal was obvious. The flux was not removed after welding and this caused severe pitting on the weld and in the region surrounding it.

Corrosive Medium. The corrosive medium used was an aqueous solution containing 35 grams per liter of sodium chloride and 20 grams per liter of potassium chromate. Adjustments of pH to various values between 0.5 and 12 were made with 23 per cent hydrochloric acid, or with a 1.542 N solution of potassium hydroxide. One-half ml of the corroding medium was used for pH of 3.91 and above; 2 ml, for pH of 1.92; and 3 ml, for pH of 0.5.

Stress. Bending stresses were used, and these could be varied from 0 to 32,600 psi on the tensile side of the test piece. The maximum deflection of the pieces was 0.790 inch, and the span (distance between the fixed end supports) was fixed between 6-3/16 inches, for a specimen without deflection, to 6-1/16 inches, for a specimen deflected to the maximum. Accuracy of the stresses determined was limited by the accuracy of the stress-strain and the strain-deflection diagrams.

Temperature. Temperature was not controlled, but variations were limited to the range of 75° to 85° F.

Aeration. No aeration of the corroding solutions was used in any of the tests. The solution, however, probably absorbed some oxygen when shaken during their preparation; and while exposed to the air during the tests.

Velocity of Corrosive Medium. No velocity control was used. The solutions were quiescent except for agitation caused by hydrogen gas evolution for all solutions between pH 0.5 and pH 6.2.

Exposed Surface. The area of the test specimens exposed to the liquid was a circle of 11-mm diameter located midway between the gage marks on the surface under tensile stress. This area included the region of maximum tensile stress.

V. CONCLUSIONS

From accelerated stress-corrosion cracking tests employing an aqueous sodium chloride-potassium chromate corrosive medium and standard test specimens of commercial magnesium alloy FS-1 extruded bar stressed in constant deflection, the following conclusions were drawn:

1. At pH values of 0.5, 1.92, 3.91, 6.2, 7.7, 8.25, 10.25 and 11.85, the critical stresses, in psi, were 32,000, 27,900, 27,700, 29,300, 29,350, 29,200, 30,350 and 32,600, respectively.

2. The critical stress was constant at 29,300 psi from pH 6 to 8.5.

From the calibration of the bending jig used in this work, employing a standard test specimen of commercial magnesium alloy FS-1 extruded bar, the following conclusions were drawn:

3. The bending apparatus was capable of producing tensile strains from 0 to 16,600 microinches per inch and tensile stresses from 0 to 32,600 psi at deflections from 0 to 0.790 inch (maximum).

4. Creep of the alloy took place at the region of maximum stress, on the side of the specimen stressed in tension, at a deflection of 0.200 inch and higher, and at strains of 3985 microinches per inch and higher.

VI. SUMMARY

An important factor preventing a more widespread acceptance of magnesium alloys in the chemical industry is its susceptibility to various forms of corrosion. Stress-corrosion cracking, occurring under conditions of residual fabrication stresses or high external stresses, is a severe localized corrosion type which can bring about complete failure in most wrought magnesium alloys. Previous investigations concerned with stress-corrosion cracking of magnesium alloys have dealt with its occurrence only in the sheet metal.

This investigation involved accelerated laboratory stress-corrosion cracking tests on a commercial extrusion. Standard tension test specimens of magnesium alloy FS-1 extruded rectangular bar were subjected to bending in constant deformation by loading them transversely as end-supported center-loaded beams. The apparatus was constructed of angle irons for end supports and steel bars for an adjustable center load. Maximum tensile strains were measured with electric strain gages and corresponding stresses were determined from the tensile stress-strain diagram for the alloy.

A corrosive medium of 35 grams per liter of sodium chloride and 20 grams per liter of potassium chromate was placed in small sections of glass tubing sealed to the specimen surface at the region of maximum tensile stress. The effects of the magnitude of tensile stress and pH on time to failure were investigated and the stress-corrosion cracking limits (critical) stresses) were determined at various pH values from 0.5 to 11.85.

The apparatus was satisfactory for laboratory testing and the basic design was recommended to be adopted as a standard. The effect of creep was corrected to some extent, since creep rates were very high.

The minimum critical stress for the alloy was found to be 27,600 psi, or 125 per cent of the tensile yield strength, at a pH of 3.0-3.5. Values of pH between 6.0 and 8.5 have no appreciable effect on the critical stress, although pH values below 3.0 and above 8.5 increase the stress-corrosion limit. From pH 6.0 to 3.5 critical stress is decreased from 29,300 psi to 27,600 psi. Creep was evident at strains of 3985 microinches and higher.

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