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A STUDY OF LIGHT METALS AND ALLOYS
AT LOW TEMPERATURES

By

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AT LOW TEMPERATURES

I Introduction.

Although the determination of the strength of metals and alloys at ordinary temperatures is an everyday matter, very little work has been done to determine their strength and elasticity at very low temperatures. Mallock 1) and Schaefer 2) have investigated certain metals at the temperature of liquid air, and have deduced formulae to express the relation between elasticity and temperature, while Andrade 3) has given an expression for the flow of metals under large constant stress. This work was intended in part to find out if their formulae would apply to the very light alloys used here.

Another object was to find, if possible, a still lighter alloy than any on the market, which would be strong enough to be used as an engineer-

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1) Mallock: Proc. Roy. Soc., A 95, 1919; 429 ff.

2) Schaefer: Deutsch. Phys. Gesell., 2, 11, 1900; 122 ff.

3) Andrade: Proc. Roy. Soc., A 90, 1914; 329 ff.

ing material, and not be attacked too strongly by water and weak acids. It was proposed to make this alloy of magnesium and lithium, since both of these metals are very light, and magnesium has lately been proved to be a practical engineering material. It was hoped that the proper alloy would be stronger and less reactive than either of its constituents. The strength of this alloy at low temperatures was also desired.

The problem had many interesting features, since the metals used were so reactive, and the liquid oxygen, used for cooling, so easily evaporated. These factors led to the construction of the apparatus, and to the employment of the method, described below.

II Apparatus.

The testing apparatus used is illustrated in Figs. I, II, and III. The novelty in this apparatus lies in the use of the ordinary paper drinking-cup B as a container for the liquid oxygen used to keep the test specimen cooled down while its strength is being ascertained. It was found that this container obviated the use of a Dewar flask for this purpose, and thus greatly simplified the apparatus.

In explanation of the diagrams, the following points are to be noted. In Figs. I, II, and III, in which the lettering has been made the same, K is a heavy iron standard, securely fastened to the wooden block W by means of the braces O, C, and J. In a slot milled in the top of this standard, the bar L is fastened to D as a fulcrum. At the other end of L an upward force can be applied by means of the spring balance T and the hook U, the upper end of which is threaded to fit the thumb-nut V. The threaded portion of the hook has a slot milled in one side which engages a projecting pin on the iron plate on which V turns.

Thus a large force can be exerted on the test specimen which is held between the brass blocks G and H (see Fig. II), the uppermost of which, G, being connected to L at E, while the lower block, H, is connected to W, and can be adjusted by means of the wing-nut S.

At F and I are connections in which the metal immersed in the liquid oxygen in B is insulated from the ends of the rod, in order to avoid too great contraction of the metal parts owing to the cold,

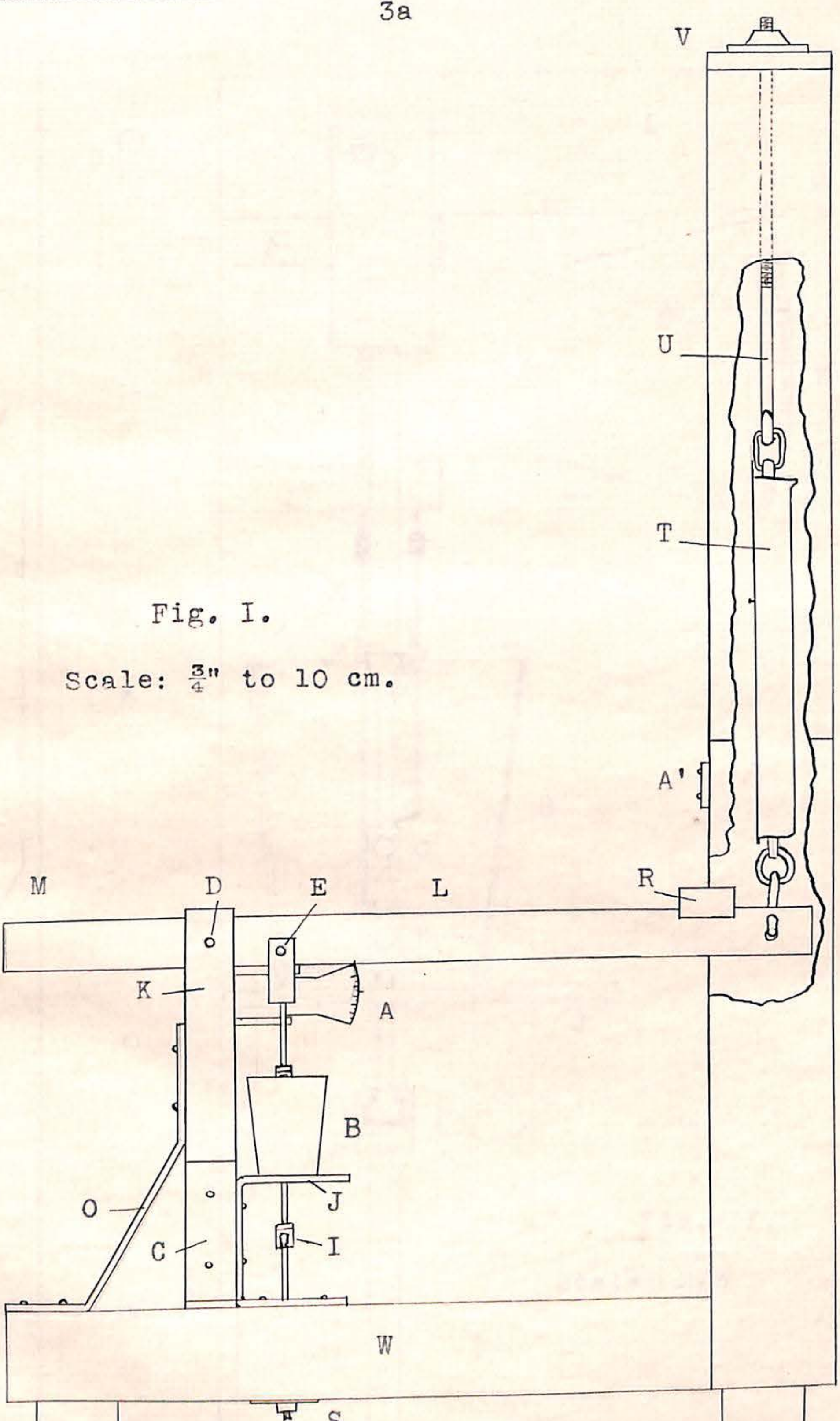

Fig. I.

Scale: $\frac{3}{4}$" to 10 cm.

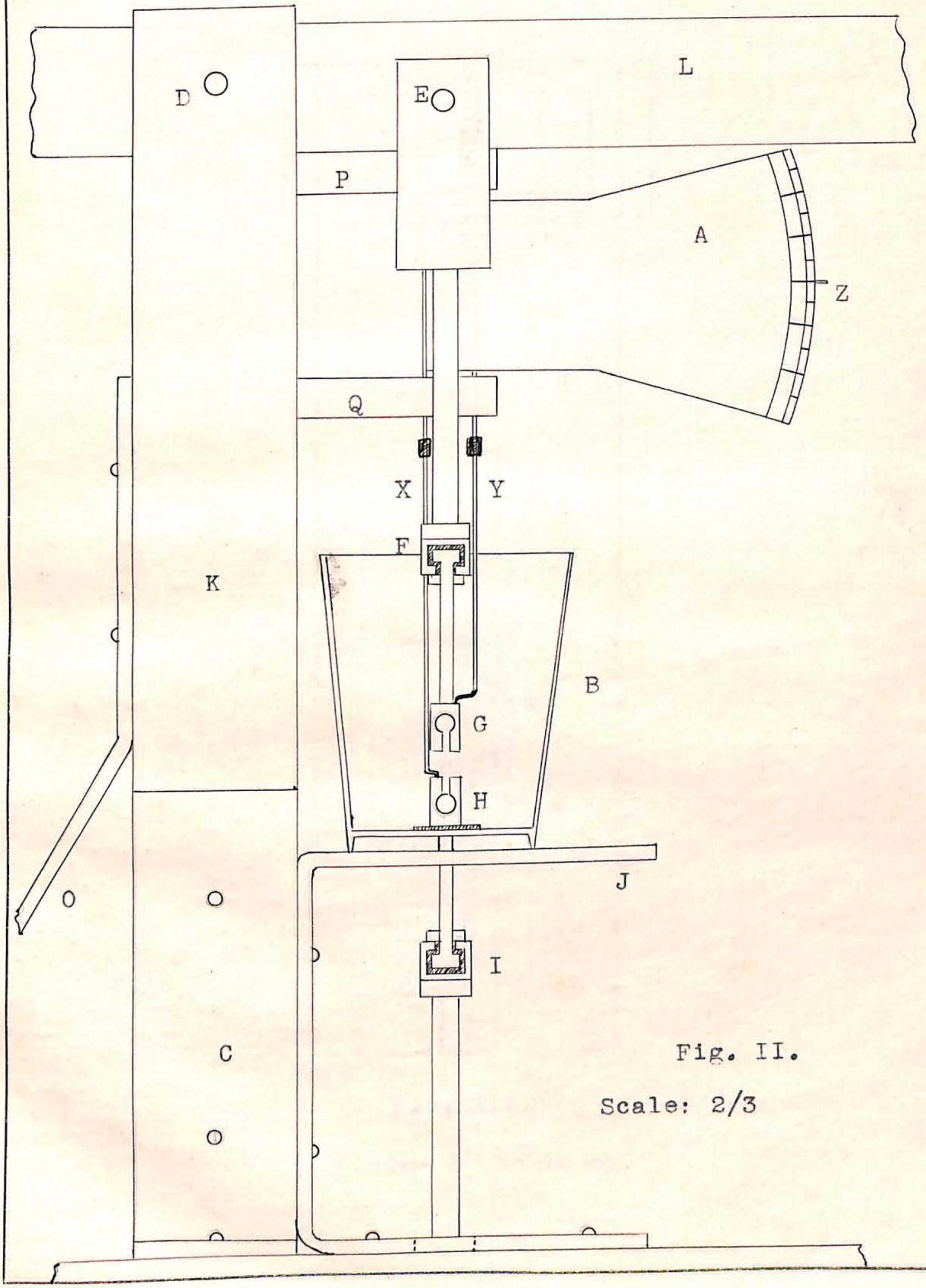

Fig. II.

Scale: 2/3

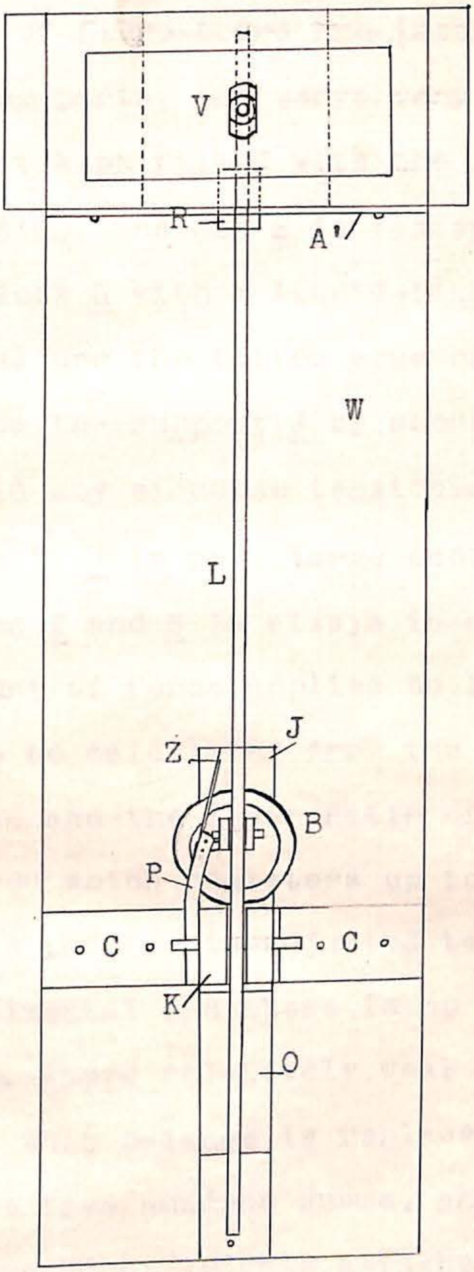

Fig. III.

Scale: 1" to 10 cm.

and also to prevent heat conduction to the specimen. Small pieces of fibre-board are inserted between the connecting parts, and serve very effectively if the cup is not kept filled with the liquid for too long at one time. The cup B is fastened to the bottom of the block H with a liquid-tight joint, so that in actual use the bottom edge of the cup is separated from the support J by about a millimeter.

To avoid any sidewise tension on the specimen, the hole in J is made large enough so that the system between E and S is always in a straight line.

The amount of force applied to the specimen can of course be calculated from the pull of the spring balance and the lever ratio of L. A spring balance is used which registers up to fifteen kilograms, and its pointer is adjusted to zero position when L is horizontal and there is no specimen in the apparatus. Where relatively weak materials are to be tested, this balance is replaced by a smaller one reading to five hundred grams, and the lever is counterbalanced by weights attached at M.

In Fig. I, the piece R is a block of rubber, used to absorb some of the shock when the specimen breaks and L suddenly snaps up against A', a steel bar screwed in place to prevent the sudden movement from going far enough to injure the rather delicate levers used to measure the elongation of the specimen being measured.

This latter part of the apparatus is best shown in Fig. II. The thin steel rods X and Y slide vertically through holes in the bars P and Q, and their pointed lower ends rest in depressions bored in the tops of H and G respectively. The pointer Z is attached to X by a small screw, and rests upon another small screw in Y. The metal scale A is soldered to X, so that any relative motion between X and Y, that is, between H and G, is measured on A, which is graduated in millimeters.

The more difficult problem than that of a testing apparatus was the design of an apparatus for the production of the desired alloys. This had to be such that the metals could be melted and stirred together under an atmosphere of hydrogen. The form finally adopted is that shown in Fig IV a.

This consists of a straight steel tube A, into the bottom of which is driven the iron plug B. The rod D is a piece of iron wire used to stir the melted metals. E is a piece of rubber tubing, closed by the pinchcock F. The tube A is turned down to the smaller

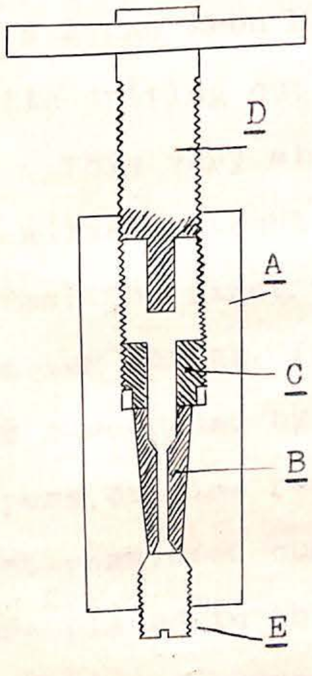

Fig. IV b.

Full Scale.

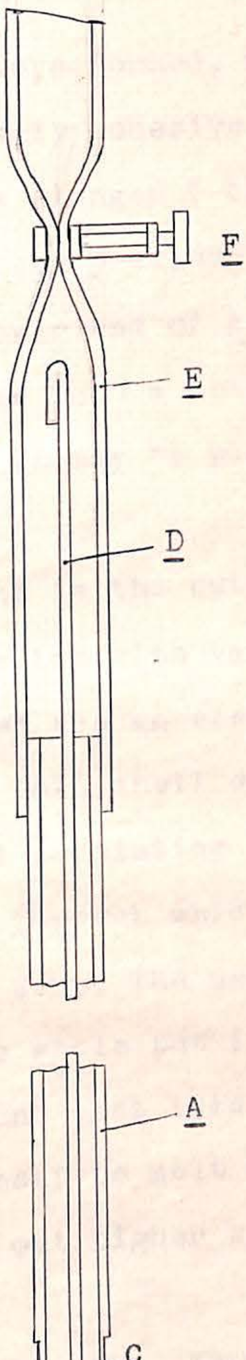

Fig. IV a.

size at C in order that the alloys formed, which were found to be generally strongly adhesive to iron, may be driven from the tube by a plunger (the rubber tubing and stirring rod having been previously removed), without deforming the lower end of A. That is, the small part at C just fits into a hole drilled in a large iron block, in which it may be placed while driving out the alloy.

This very simple arrangement is the outgrowth of a large amount of experimentation with various forms. The first to be considered was an electrical heating device. In this form, a thin shell of iron was surrounded by a heating coil consisting of two layers of fine resistance wire, through which a rheostat-regulated current could be sent. The metals were placed in this cup, and the whole put in an atmosphere of hydrogen. It was found that this arrangement would not give sufficient heat to melt magnesium, so after some time spent trying out higher currents, etc., it was discarded.

The next form is shown in Fig. V a. Here the steel tube A had a cup B fastened to the welded bottom C. This cup contained the split mold shown in detail in Fig. IV b. The top of the large tube was

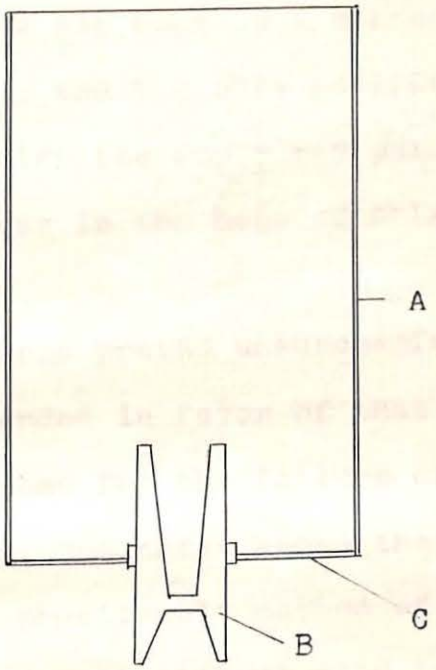

Fig. V a.

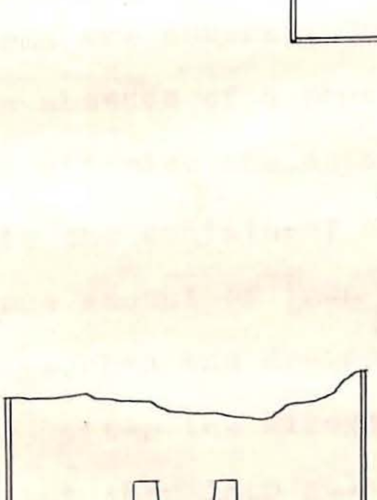

Fig. V b.

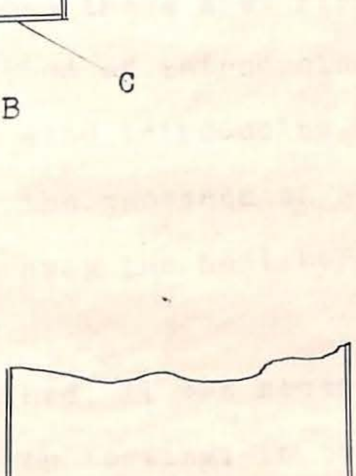

Fig. V c.

Full Scale.

covered with a sheet of mica, through holes in which hydrogen was passed in and metals could be introduced. The heating was done by a Bunsen burner underneath B. Figs. V b and V c show modifications of this arrangement in which the cup B was put in slightly different positions in the hope of obtaining more rapid melting.

All these forms proved unsuccessful, however, and so were discarded in favor of that first described. The reasons for the failure of the earlier forms are several, but chief among these are: First, the absence of a practicable method of introducing and stirring the metals without also introducing air into the container; and Second, the presence of a large amount of iron to conduct away the heat before it reaches the desired spot.

After the alloys were obtained, it was necessary to put them into suitable form for testing. In the case of the harder alloys, this was done by making them in ingots about a quarter-inch in diameter, which were then turned down to the proper dimensions on a lathe. For the softer alloys, the mold shown in Fig. IV b was employed.

Here we have a cylindrical block of iron, A, turned inside so as to hold the split mold B in the position shown. The threaded portion above this mold holds the ring C, which holds the mold in place, while the plunger D can be screwed down through it to force the ingot placed there into the mold. At the bottom of A is the plug E, acting as the end of B, and, when turned in as far as possible, serving to force B from its position after the specimen has been forced in.

III Procedure and Results.

Considerable time was first spent in trying to make metallic lithium in practical quantities by the electrolysis of fused lithium salts. The storage batteries in the laboratory were arranged so that they could be connected in parallel to give as much as 100 amperes at 10 volts. For this purpose, very large conductors were necessary, # 4 copper wire being used here. In

In the electrolysis, the method of Ruff and Johannsen[4] was followed. A mixture of 85 % lithium bromide and 15 % lithium chloride was fused in a

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4) Ruff and Johannsen: Zeitschr. Elektrochem., 12, 1906; 186 ff.

copper crucible, and the fusion electrolyzed with a carbon anode and an iron kathode. For several reasons, notably the lack of a suitable arrangement for the carrying off of the chlorine and bromine formed during the electrolysis, this method was unsuccessful as carried out here. After a few trials, it was decided to abandon the attempt, as time was scarce, and sufficient lithium was ordered from the Henry Heil Chemical Co., St. Louis, Mo. Since this did not arrive for some time, the apparatus first described above, page 2 ff., was tried out, and the original faults were corrected so far as possible.

In making the tests, the specimen was placed in position in the machine, and the tension applied, one kilogram at a time, until breaking occurred. After each additional kilogram was applied, the elongation was read, and again a minute later. Thus some idea of the elongation as compared with the stress could be obtained.

The first metal to be tested was pure aluminium, obtained in the form of an extruded rod, and turned down to the required size. The results of this and the following tests are given in the table on page 9a. Aluminium seemed to stretch considerably before

Composition of alloy	Al ext.	88%Mg 12%Al	88%Mg 12%Al	96%Mg 4% Al	Li ext.	75%Mg 25%Li	Duralumin	Hard Brass	Hard Steel
Specific gravity	2.75	1.82	1.82	1.737	0.534	1.461	2.766	8.363	7.833
Tensile str. lbs/in^2--25	18662.	50123.	65085.	35515.	230.9	1135.5	59753.	50450.	75000.
Tensile str. -182.4	32404.	51612.	34812.	55242.	1889.5	3325.	72381.	---	---
%Increase in str. at-182.4	73.9	2.09	-46.5	55.6	718.	193.	21.13	---	---
% elongation at 25	32.06	2.08	6.64	2.09	11.82	negl.	negl.	10,1	18.22
% elongation at -182.4	20.70	4.57	1.26	5.96	15.18	"	"	---	---
strength / sp. grav.	6912.	27540.	35758.	20099.	432.4	777.03	21599.	6032.5	9592.5

breaking, and the area at the actual point of fracture was greatly reduced. This test was carried out more as a trial of the apparatus and for the acquirement of a technique of operation, than for the value attached to the results obtained. The arrangement for measuring the elongation was found to be in such a position that it and the scale of the spring balance could not be read at the same time, a fault which was not corrected here, but which should be done away with by any subsequent investigator.

As soon as the minor defects in the apparatus had been corrected, three commercial alloys of magnesium and aluminium, obtained from the American Magnesium Corporation, Niagara Falls, N. Y., were tested at ordinary and low temperatures. The first of these was an alloy containing 12 % of aluminium, and had been extruded into a rod. This showed remarkable strength for its weight. The elongation was not large. At the low temperature its strength was increased by only 2.09 %.

The second of these three was of the same composition as the first, but had been given a heat treatment, first at 425 deg. C. for a short time, and then for a considerable time at between 150 and 250 deg.

This treatment causes hardening due to the precipitation, from the solid solution, of small particles of a compound of aluminium and magnesium. Consequently the greater strength found was not unexpected. The elongation was somewhat greater than in the preceding case. At the low temperature, however, the strength was <u>decreased</u> by 46.5 %. This was most unexpected, and was the only case, among all the alloys tested, where a decrease in strength at low temperatures occurred. It is probably due to the fact that the particles precipitated in the heat treatment, while bonding together the solid solution at ordinary temperatures, tend to segregate and render the alloy markedly less homogeneous at the boiling point of liquid oxygen.

The third of these commercial alloys to be tested contained 4 % of aluminium. This was not nearly so strong as the other two at room temperature, but at the low temperature it was very strong, the increase being 55.6 %. The elongation was practically the same as that of the first.

As soon as the first of the lithium ordered arrived, work was started on the production of alloys. The earlier forms of heating apparatus had meanwhile been tested and discarded, and the final form was now used.

The procedure here was to weigh out the metals in the proportions desired and put them into the tube shown in Fig. IV a. The iron stirring rod was then placed in the tube, the rubber tubing slipped over the open end, and the tube, clamped in an inverted position, filled with hydrogen. At first a Kipp generator was used for this purpose, but as this proved to be too slow, a tank of compressed hydrogen was later used.

After making sure that the tube was completely filled with hydrogen, the pinchcock was applied to the rubber tubing, and the steel tube, in a slightly inclined position, was brought to a bright red heat over a Bunsen burner. The rubber tubing was prevented from burning by a piece of asbestos wire gauze, through a hole in which the lower end of the tube extended.

When the metals were completely melted and mixed, the stirring rod was drawn up out of the molten alloy, and the tube allowed to cool. When fully cooled, the r rubber tubing was removed and the alloy driven from the tube by a tightly-fitting plunger.

In this way were made up over twenty different alloys, using varying proportions of magnesium, aluminium, manganese, zinc, and antimony, with lithium. Few of these proved of interest, as they were mostly either as soft as lithium, or very brittle, or of a crumbly composition.

Of the lithium magnesium alloys, the most intersting was the one containing 75 % of magnesium. If more than this proportion of magnesium is used, the alloy becomes too brittle, while if less is used, it becomes too soft. Even this alloy was rather brittle, and consequently could be turned only with great care. Its low tensile strength, as shown in the table on page 9a, was probably due to this brittleness.

An alloy containing 15 % manganese and 85 % lithium showed interesting strength when first made up, but when later made in a large enough quantity for an actual test, failed to be as strong as was expected. The second casting was made more carefully than the first, so that there is some doubt as to the cause of the strength in the first case. It is known that a small percentage of manganese causes surprizing increase in strength in many alloys.

In the table on page 9a are given the data on only one lithium alloy, the object being to compare it with the magnesium-aluminium alloys, and these with hard steel and hard brass, and with Duralumin. The strength of the latter was measured in the same manner as that of the magnesium-aluminium alloys above, while the strengths and densities of the steel and brass were taken from Marks' Mechanical Engineers' Handbook.

In the work by Mallock, referred to on page 1, the following relation is given:

$$\frac{M_{\theta_2}}{M_{\theta_1}} = \frac{(\text{Melting point in deg. C.}) - \theta_2}{(\text{"} \quad \text{"} \quad \text{"} \quad \text{"} \quad \text{"}) - \theta_1}$$

where M_{θ_2} and M_{θ_1} are the elastic moduli at the temperatures θ_2 and θ_1 respectively. As a check on this formula, it was decided to freeze the alloy containing 77 % potassium and 23 % sodium, which is liquid above -12.5 deg. C., and to find its strength and elasticity. Accordingly a small amount of the alloy was melted up under petroleum ether, and it was then poured into a small test-tube about one cm. in diameter, which was immersed in liquid oxygen until the alloy was at the temperature of the liquid surrounding it. The tube was then withdrawn and the glass broken with a hammer, leaving the solid alloy, a piece about one cm. in diameter and three cm. long.

This was examined for a short time, being held by small tongs. As it was feared that it would soon melt, it was replaced in the liquid to cool down again. It was then taken out, and a small portion cut from one corner to determine its relative hardness. Its consistency was found to be much the same as that of lead.

After about a minute it was again placed in the liquid oxygen. When, a few minutes later, it was taken out into the air, it exploded spontaneously with a loud report, small drops of the liquid alloy flying in every direction.

The next day another attempt was made. This time, instead of being poured into the small test-tube, it was put directly into the split mold described on pages 7 and 8, a small metal cup holding the halves of the mold together. This cup was carefully lowered into the liquid oxygen, allowed to cool completely, and then lifted out. Immediately on being brought into the air, the alloy again exploded, only a small portion of the oxidized alloy remaining in the bottom of the mold.

A third attempt was made, a glass shield being used for the protection of the experimenter. This time care was taken to keep the alloy under the liquid all the time, since it appeared that it would explode only when exposed to the air, after being immersed in the liquid oxygen. A special cup was made in which to transfer the specimen, under liquid oxygen, from the container in which it was cooled in the mold to the cup on the testing apparatus. The same procedure was followed as in the second case, except that the mold was kept under the surface of the liquid. After an immersion of a few minutes, it exploded once more, while under the liquid oxygen.

No further attempt was made to test this alloy, except that a small piece cooled in a glass tube was hammered to test its hardness once more. It flattened out under hammering almost exactly as lead does.

The above phenomena are difficult of explanation, since the alloy, when taken out of the liquid after the first immersion, did not explode, but did so on every subsequent attempt to examine it. It is probable that some obscure factor governing the reaction was absent in the first case, but present every other time. In these other cases it appears that the alloy, which is more or less porous, absorbs enough of the liquid oxygen to react with it to a slight extent, the heat generated by even a slight reaction, or perhaps merely from the absorption, being sufficient to cause the whole mass to explode instantaneously. The alloy is, of course, extremely reactive at ordinary temperatures, and oxidizes readily. A small drop of it on the skin causes a deep burn unless the skin is perfectly dry.

This concluded the experimental work. It is regrettable that more could not be done, but the above results show that with a detailed and lengthy investigation, making up a full series of binary alloys of magnesium and lithium, and perhaps a series of ternary alloys including manganese with these two metals, it is probable that a useful alloy, fairly strong yet very light, could be obtained.

In concluding I wish to thank Dr. R.H.Goddard for his constant advice and encouragement, and Mr. N.A.Riffolt for his invaluable aid in the design and construction of my apparatus.

IV Summary.

1. A new apparatus for determining the tensile strength of small test-specimens at low temperatures is described in detail.
2. A study is made of alloys of Al and Mg, and of Li with Mg, Al, Mn, Zn, and Sb, to determine their strength and elasticity at low temperatures. A table is given showing the results of the tests on the more interesting alloys, and comparing them with hard steel and hard

brass.

3. A detailed account is given of the surprising discoveries attending an attempt to measure the strength of the alloy containing 77% K and 23% Na, at the boiling point of liquid oxygen.
