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**PROCESS FOR DRAWING AND HEAT TREATING
MAGNESIUM WIRE**

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No Drawing. Application April 15, 1952,
Serial No. 282,482

9 Claims. (Cl. 148-4)

This invention relates to a method of heat treatment of magnesium and magnesium base alloys to render them more ductile so that they can be readily drawn into wire. The present application is a continuation-in-part of our copending United States patent application Serial No. 610,290, filed August 11, 1945, now abandoned.

The conventional wire drawing procedures suitable for steel or copper wire are not applicable to the drawing of magnesium wire. Magnesium has a hexagonal crystalline structure which is relatively non-ductile. It does not cold-work easily since its crystals can slip in only one direction at a time as compared to the crystalline structure of metals such as copper or aluminum or steel which have a cubic type of structure whose crystals can deform or slip in three directions. Therefore, magnesium cannot be cold-drawn through a series of dies to increase its tensile strength since it rapidly becomes too brittle to have any useful amount of tensile strength. Nor is magnesium amenable to annealing or strain relieving treatments following cold-working since such treatment never develops sufficient tensile strength and elongation properties for commercial requirements.

As a result of an extended investigation, we have found that by a heat treatment involving a succession of coordinated heating steps, first at a relatively high temperature during the initial drawing reductions, and then at a considerably lower temperature during the final drawing reductions, a magnesium wire of commercially practicable tensile strength and elongation can be produced using commercially economical reduction increments of from 5% to 20% decrease in area in each die and without intermediate recrystallizations or annealings. This method of heat treatment during successive drawing operations produces a special arrangement of crystalline structure in the wire wherein the randomly oriented hexagonal crystals, which normally can deform only to a very limited extent, become aligned more or less uniformly in a direction of preferred orientation so that the wire can withstand repeated reductions without breaking. Annealing to cause recrystallization at any point during or between the successive treatments would break up such alignment and return the metal to a random crystal orientation which would be much weaker and less ductile than the preferred orientation worked into the wire by the process of this invention. Accordingly, our heat treatment starts at a temperature lower than the customary annealing temperature for magnesium, and a still lower temperature is used as the preferred orientation becomes more and more developed in order to prevent any appreciable recrystallization.

Specifically, the present invention involves heat treating magnesium wire during a series of successive drawing reductions by heating the wire to a temperature of between 325° F. and 500° F. and holding the wire substantially at that temperature while carrying out the initial reductions, and then heating the wire to a temperature between 275° F. and 100° F. and holding the wire substantially at that temperature while carrying out at least

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the final reduction. Preferably, at least an initial 30% total reduction in area (i. e. amount of reduction in cross-sectional area in relation to the original area of the initial stock) should be done at the higher temperature.

In order to effect a preferred orientation, the heat treatment during the drawing operations is started at a temperature substantially lower than the customary annealing temperature for magnesium and does not last for a period of time sufficient to cause any appreciable degree of annealing or recrystallization at these temperatures. A period of at least several hours is necessary to cause annealing at these temperatures. The temperature is lowered as the reduction proceeds so that there will be no appreciable recrystallization. While heat-treatment temperatures between 100° F. and 275° F. can be used during the final drawing reductions, it is preferable while finish drawing wire having a diameter larger than 0.025 inch to hold the wire at a temperature above 175° F. Furthermore, where the reductions in area total 80% or greater or where the rod drawn has an initial diameter of 1/2 inch or larger, it is preferable to have a heat treatment at an intermediate temperature in the range between 200° F. and 350° F. while carrying out intermediate drawing reductions between the initial and final reductions. The initial and intermediate and final reductions must be carried out at successively lower temperatures.

It is advantageous to coordinate the temperature of each successive heating operation with the total reduction in cross-sectional area. At least the first 30% total reduction in cross-sectional area should be at a temperature within the highest temperature range and at least the final 2% total reduction in area should be done at a temperature within the lower temperature range. The remainder of the heat treatment may be carried out at one or more intermediate temperatures. The lower temperature for the final reductions imparts to the wire a better finish and more uniform diameter. Wire having a smoother surface finish has an appreciably greater tensile strength than does wire having surface defects, which may spread under tension. When the finished wire is of smaller size than 0.025 inch in diameter, temperatures lower than 175° F. are generally used with advantage for the finishing reductions.

We have found that, in order to prevent the magnesium metal from welding to the edge of the dies at elevated temperatures, it is desirable to artificially cool the wire drawing dies during each successive drawing reduction while the wire is being treated at a temperature within the initial temperature range. Accurate control of the contemplated treatment temperatures during drawing is assured by auxiliary thermal control of the temperature of the dies. This is facilitated by constructing the dies and their holders of materials having high heat conductivity and having interior passages for the flow of cooling fluid. Preferably, the successive dies each carry out a reduction in cross-sectional area of from 5 to 20% (relative to the cross-sectional area of the wire immediately preceding any particular drawing reduction).

The initial stock of the wire may be rolled or extruded magnesium rod or wire. In the interest of simplicity, "magnesium" is used herein to include commercially pure magnesium metals or magnesium base alloys such as those containing aluminum (e. g. 2.5 to 12%) with or without zinc (e. g. 0.2% to 3.5%), and with or without small percentages of other alloying elements, since we have found the improved heat treatment procedure of the invention equally applicable to both pure magnesium and such magnesium base alloys. The initial reductions of the stock must be made hot. The heat treatment temperature of the wire varies with the composition of the stock. For commercially pure magnesium initial heat treatment temperatures of from 325° F. to 350° F. are

satisfactory, but this temperature will vary with the grade and size of the stock. Magnesium base alloys require higher initial temperatures, up to as high as 500° F. Generally, the high temperatures are used for the heat treatment during the first two to five reductions depending on the size and quality of structure (i. e. absence of coring) of the stock, and at least the last reduction is carried out at the lower temperature of from 275° F. to 100° F. A primary purpose of having at least one finish draw at a lower temperature is to produce a good surface and maximum tensile strength.

Any conventional type of wire drawing apparatus is suitable for practicing this invention. The initial stock and the wire at various stages in its reduction can be immersed in heated baths of mineral oil or oil-like chemicals to regulate the temperature of the wire and to lubricate it prior to its passage through a reducing die. The heating period is only long enough to bring the wire to a uniform temperature, and usually this is effected in from one to five minutes. The wearing surfaces of the dies are preferably cooled or refrigerated by continuously circulating ice water or other suitable cooling medium through cases surrounding the dies. Various dies of known design are adapted to be cooled in this manner.

A principal advantage of the method of this invention, in addition to the increased tensile strength and ductility of the product, is the higher drawing speeds attainable in comparison with previous practice in the drawing of magnesium wire. Drawing speeds of 300 feet per minute and upwards can be employed while carrying out reduction increments of as high as 20%.

It is of primary importance in carrying out the present invention that any appreciable annealing of the magnesium wire be avoided in order to preserve the preferred crystalline orientation which is worked into the wire by the heat treatment method of this invention. Heating for too long a period has been found to reduce the toughness of the wire and to cause breaks during drawing. We have found that subjecting magnesium to temperatures below 600° F. for periods of say five to fifteen minutes will cause no substantial degree of annealing. The lack of any substantial recrystallization for short periods at such temperatures has been clearly demonstrated by the absence of the characteristic indicia of recrystallization in tensile strength and elongation tests carried out on magnesium wire which has been held at temperatures below 600° F. for brief periods of say five to fifteen minutes.

As an advantageous embodiment of this invention, we will describe the drawing of an extruded rod of commercially pure magnesium 0.240 inch in diameter to a wire 0.177 inch in diameter in several successive drawing reductions. Each reduction was about 10% in area. The first five reductions to 0.235, 0.224, 0.212, 0.202 and 0.192 inch in diameter were carried out with the wire stock at a temperature of 330° F. The final two reductions to 0.182 inch and 0.177 inch diameter were carried out at a temperature of 200° F. The wire was drawn at a speed of 375 feet per minute. After each drawing reduction, the wire was passed through a heated oil bath to appropriately adjust its temperature for the next succeeding drawing. Oil from the heating bath served as lubricant for the die. Ice water was continuously circulated through a cooling box about the die case in order to minimize the tendency of the metal to stick to the dies. The first 36% total reduction of area was carried out at the high temperature of 330° F. The final 9.7% total reduction in area was carried out at the lower temperature of 200° F. The finished wire has a tensile strength of 37,800 pounds per square inch and an elongation of 8.9% in ten inches.

In another example a magnesium base alloy containing 4% aluminum was satisfactorily drawn to a finished wire of 0.010 inch diameter, using a temperature of 500° F. for the initial or break-down reductions, a temperature

of 350° F. for several intermediate reductions, and room temperature for the last two finishing reductions.

At heat treatment temperatures above 500° F. during the initial reductions much trouble was experienced with the magnesium metal welding and sticking to the edges of the die opening. Furthermore, the tensile strength of the product was always below the standard (30,000 pounds per square inch) considered commercially practicable. Elongation tests gave values of only 7 to 9% in ten inches. To avoid these difficulties and in order to prevent any appreciable annealing effect, it was found that the temperature of the wire during the initial heat treatment should not exceed the upper limit of 500° F.

The criticality of the lower limit of 325° F. for the initial reductions was demonstrated by the fact that commercially pure extruded magnesium rod one-half inch in diameter held at a temperature of 300° F. for the initial draws could not be drawn satisfactorily. It broke repeatedly during the first draw. The same one-half inch diameter rod drew satisfactorily at initial drawing temperatures of 450° F. and 400° F., giving a product having a tensile strength of 31,000 to 32,000 p. s. i. and elongation test values of 11 to 17% in ten inches. The first five draws were at the high temperature and the last draw was at 200° F. About 10% reduction was carried out on each draw.

When commercially pure extruded magnesium rod one-fourth inch in diameter was drawn at an initial temperature of 300° F., fractures occurred intermittently with reductions of 10% or greater. Even a single initial or break-down draw could not be carried out consistently without breaks at temperatures of 300° F. or lower. However, when the initial three to five drawings of one-fourth inch diameter magnesium rod, with at least 10% reduction per drawing, were carried out at 350° F., and then one or two finishing draws to 0.177 inch diameter were carried out at a wire temperature of 270° F. or 200° F., excellent results were obtained. No fractures occurred and the product had a tensile strength of 32,000 to 38,000 p. s. i. with an elongation test value of 13% in ten inches.

The improvement in surface and strength obtained by having at least one finishing pass at a low temperature was demonstrated by the three to five thousand pounds per square inch increase in tensile strength produced by such a finishing pass. When an extruded magnesium rod of 0.240 inch diameter was hot drawn in six passes while the wire was held at 400° F. for each pass, the tensile strength of the final product (having a diameter of 0.162 inch) was only 27,600 pounds per square inch with an elongation in ten inches of 15%. However, when this same extruded rod was hot drawn at 400° F. and finished with one cold pass at 200° F. the tensile strength of the final product of 0.162 inch diameter was increased to between 31,000 and 33,000 pounds per square inch with an elongation in ten inches of 12%.

An advantageous wire drawing schedule for the present invention is summarized below in Table I:

Table I

Wire Temp., °F.	Reduction per die, percent	Total area Reduction, percent
350-400	10	up to 50.
200-250	10-15	50 to 95.
100-150	10-20	above 95.

A comparison of the tensile strength and elongation of magnesium wire drawn from an original diameter of 0.250 inch to a final diameter of 0.162 inch at a constant temperature of 500° F. and an identical wire drawn

according to the method discovered by us is summarized below in Table II:

Table II

Wire Temp., °F.	Stage of Reduction	Final Tensile Strength, p. s. i.	Final Elongation, per cent in 10"
500.....	throughout.....	27,650	15 to 19.
330.....	initial.....		
200.....	intermediate.....		
Room.....	final.....		
		37,800	8.9.

It will be seen from Table II that the magnesium wire drawn according to the method disclosed herein has substantially higher tensile strength than the wire continuously drawn at a temperature of 500° F. yet also has good ductility. Thus, it can be seen that not only are the temperature limits disclosed herein critical, but that the use of successively lower heat treatment temperatures as the drawing proceeds is also of critical importance.

Artificial cooling of the dies is an advantageous although not essential aspect of this process in order to minimize sticking of the magnesium metal to the die surfaces at temperatures above 325° F. It is possible, however, to employ uncooled dies with wire temperatures up to 500° F. and yet encounter no sticking difficulties during welding if sufficient care is taken in the lubrication of the dies. Artificial cooling of the dies avoids the necessity for particularly careful lubrication.

The function of the lubricant in a wire drawing operation is to prevent the wire from welding or sticking to the wearing surface of the die. In the preferred practice of the invention the contemplated heating of the wire preceding any drawing reduction is carried out in a heated bath of mineral oil or oil-like liquid suitable for die lubrication of the die by the residual film of oil remaining on the wire after its passage through the wiper, particularly in the case of high viscosity mineral oil which carbonizes during use and tends to coat the wearing surface of the die with a carbonaceous deposit. Artificial cooling of the dies minimizes this tendency of mineral oil to carbonize and coat the surface of the die.

We claim:

1. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out the initial reductions, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire substantially at such lower temperature while carrying out at least the final reduction, each of said heatings being of shorter duration than is required to effect substantial recrystallization of the metal at the heating temperature, whereby said heat treatment avoids causing any substantial degree of annealing of the metal.

2. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 175° F., and holding the wire substantially at such lower temperature while carrying out at least a final 2% total reduction in area, each of said heatings being of shorter duration than is required to effect substantial recrystallization of the metal at the heating temperature, whereby said heat treatment avoids causing any substantial degree of annealing of the metal.

3. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium

wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area, the reducing dies being artificially cooled during each drawing reduction, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 175° F., and holding the wire substantially at such lower temperature while carrying out at least a final 2% total reduction in area, each of said heatings being of shorter duration than is required to effect substantial recrystallization of the metal at the heating temperature, whereby said heat treatment avoids causing any substantial degree of annealing of the metal.

4. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out the initial reductions, the reducing dies being artificially cooled during each drawing reduction, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire substantially at such lower temperature while carrying out at least the final reduction, each of said heatings being of shorter duration than is required to effect substantial recrystallization of the metal at the heating temperature, whereby said heat treatment avoids causing any substantial degree of annealing of the metal.

5. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature of between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area, said reduction being made in increments of from 5% to 20% decrease in area each, the reducing dies being artificially cooled during each drawing reduction, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire substantially at such lower temperature while carrying out at least a final 2% total reduction in area, each of said heatings being of shorter duration than is required to effect substantial recrystallization of the metal at the heating temperature, whereby said heat treatment avoids causing any substantial degree of annealing of the metal.

6. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out the initial reductions within 15 minutes after the wire has reached such temperature, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire at such lower temperature while carrying out the final reductions.

7. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between 325° F. and 500° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area within fifteen minutes after the wire has reached such temperature, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire at such lower temperature while carrying out at least the final reduction.

8. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature between

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325° F. and 500° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area within fifteen minutes after the wire has reached such temperature, the reducing dies being artificially cooled during each drawing reduction, thereafter heating the resulting initially reduced wire to a lower temperature between 275° F. and 100° F., and holding the wire at such lower temperature while carrying out the final reductions.

9. The method of heat treatment to increase the ductility of magnesium during the drawing of magnesium wire in a series of successive drawing reductions which comprises heating the wire to a temperature of about 350° F., holding the wire substantially at such temperature while carrying out at least an initial 30% total reduction in area within fifteen minutes after the wire has reached such temperature, said reduction being made in increments of from 5% to 20% decrease in area each, thereafter heating the resulting initially reduced wire to a temperature of about 200° F., and holding the wire at such temperature while carrying out at least the final reduction.

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