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METHOD OF MAKING A MAGNESIUM-BASE ALLOY CONTAINING ALUMINUM AND ZINC

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The invention relates to an improved method of making a magnesium-base alloy containing aluminum and zinc. It more particularly concerns an improved method of making an alloy of the foregoing character which on being cast, as in a continuous casting machine, exhibits a homogeneous fine grain structure.

Heretofore in making a commercial magnesium-base alloy containing aluminum and zinc, e. g. that having the ASTM designation AZ31, the difficulty arises that on being cast into ingot, as by the continuous casting method, the metal exhibits a coarse columnar grain structure. This grain structure renders the metal unsuitable for many uses. For example in rolling the ingot into sheet excessive scrap losses result from edge cracking during rolling. Sheet rolled directly from the ingot metal is not generally suitable for use in the graphic arts, for example, in which a relief image is developed by etching in a nitric acid etching bath as in photoengraving work. In the photoengraving art, it is desirable, if not essential, for the metal of the photoengraving sheet or plate to exhibit a fine homogeneous grain structure, that is, a structure in which all the grains are generally substantially the same size and small. The application of conventional alloying methods and attempts to refine the grain prior to continuous casting into ingot of the AZ type of magnesium-base alloy by conventional methods have not been effective to produce ingot having a fine homogeneous grain structure. Die expressing (extruding) the continuous cast ingot yields an extrude having a fine grain structure suitable for rolling but the extrusion operation is costly and time consuming. Insofar as we are aware, there is no commercially available method by which the AZ type of magnesium-base alloy containing from 2.5 to 3.5 percent of aluminum and 0.75 to 1.5 percent of zinc can be formulated so that on being cast continuously into ingot a fine homogeneous grain structure is obtained suitable for rolling directly, without excessive edge cracking, into sheet acceptable for use in photoengraving. Accordingly, it is the principal object of the invention to provide an improved method of making the AZ type of magnesium-base alloy which on being cast continuously into ingot exhibits a fine homogeneous grain structure and can be rolled directly without the need for first forming the metal into an extrude. Other objects and advantages will appear as the description of the invention proceeds.

The invention then contemplates the preparation of AZ type magnesium-base alloy in which the aluminum content (A) ranges from 2.5 to 3.5 percent and the zinc content (Z) ranges from 0.75 to 1.5. Small amounts of calcium may be present if desired such as amounts of up to 0.04 percent, and manganese up to 0.25 percent.

In carrying out the invention two melts, designated 1 and 2 herein for convenience, are prepared separately and subsequently mixed together in about equal proportions to form the desired alloy. Melt 1 is commercial magnesium to which a small amount of zirconium is added, the amount used depending upon the amount of

iron impurity in the magnesium. Generally from 0.3 to 0.6 percent by weight of zirconium is sufficient to bring about the reduction or removal of iron. In preparing melt 1, commercial magnesium may be melted in an open melting pot under the protection of a conventional saline flux, such as is used in conventional magnesium melting practice. The melt is brought to a temperature preferably in the range of 1300° to 1400° F. While heated to this temperature, the zirconium is added in any convenient manner. Although metallic zirconium may be used, it is preferable to introduce the zirconium into the melt as a magnesium-zirconium alloy, e. g. an alloy composed of 30 percent zirconium, the balance being magnesium. Another method of introducing the zirconium into the melt is by reacting with it a fused mixture of equal parts of zirconium tetrachloride and potassium chloride. The amount of the mixture to use may be calculated from stoichiometrical considerations. In general, about 2½ pounds may be used of the 50:50 mixture of zirconium tetrachloride and potassium chloride per 100 pounds of the melt.

After dispersing the zirconium in the melt, a small amount of aluminum is added, the amount used being from about 3 to 5 percent of the weight of the zirconium added. The so-treated melt is then allowed to remain in a quiescent state for a time sufficient to bring about settling of impurities in suspension particularly those containing iron. The settling time may range from about 15 to 40 minutes, 30 minutes being generally sufficient. After allowing the so-prepared melt to settle, the supernatant portion is ready for use in the method.

Melt 2 is also prepared from commercial magnesium by melting the metal preferably in an open melting pot using conventional magnesium foundry flux for protection against atmospheric attack. To the molten magnesium is added about twice as much aluminum and zinc as that desired in the final alloy, such as an amount by weight between 5 and 7 percent in the case of the aluminum constituent, and 1.5 to 3 percent in the case of the zinc constituent for the preparation of an AZ31 alloy. The alloying of these metals is effected in conventional manner. If desired, the resulting alloy may be treated with a halogenated carbonaceous material or other carbonaceous grain refining material such as lamp black as an aid to grain refinement. The melt so-prepared is then ready for use in the method.

Equal weights, or substantially equal weights of melts 1 and 2, as above prepared, are blended together. The blended melt is preferably held in the molten state at about 1230° to 1260° F. for 60 to 100 minutes for example. This settling period permits undesirable suspended particles, if present, to settle out of the blended melt before casting.

After settling the blended melt and before casting into ingot, it is desirable to add a small amount of calcium, e. g. an amount sufficient to produce a calcium concentration in the alloy of up to 0.04 percent.

Example

The following example is illustrative of the practice of the invention.

A melt of 4000 pounds of commercial magnesium was made in an open top melting pot (#1) and the temperature of the melt raised to between 1300° and 1400° F. Between 80 to 100 pounds of a fused mixture of 50 parts of $ZrCl_4$ and 50 parts of KCl was added and stirred into the melt. The reaction which ensued liberated metallic zirconium in the melt. One pound of aluminum was then added to the melt which was then puddled with saline flux so as to flux refine the melt. The so-fluxed melt was allowed to settle for 30 minutes. In a second melting pot (#2) 3670 pounds of commercial magnesium was melted

together with 240 pounds of aluminum, 80 pounds of zinc, and 7 pounds of manganese, thereby forming a magnesium-base alloy containing about 6 percent of aluminum, 2 percent of zinc, and 0.25 percent of manganese. The melt obtained was heated to between 1300° and 1400° F., treated with about 40 pounds of saline magnesium foundry flux and allowed to settle.

About 2000 pounds of the supernatant portion of each of the two melts so-prepared were decanted into a separate melting pot #3, thereby forming a blend of the two melts in which the concentration of aluminum and zinc was thereby reduced to about one-half that of the concentration of the melt of pot #2. The resulting blended melt was treated with 12 ounces of polytetrafluoroethylene (Teflon) and then allowed to settle at a temperature between 1230° and 1260° F. for 60 minutes. After settling the blend 4 pounds of a calcium alloy consisting of 80 parts of calcium and 20 parts of magnesium was added, thereby introducing a small amount of calcium into the blend. The melt thus obtained was pumped to a continuous casting machine thereby forming a rectangular section ingot 11" x 27" at the rate of 2.5 inches per minute. Samples of the so-cast ingot revealed a fine homogeneous grain structure.

We claim:

1. The method of making a fine homogeneous grain magnesium-base alloy ingot containing aluminum in amount between 2.5 and 3.5 percent and zinc between 0.75 and 1.5 percent by weight which comprises dispersing in the first batch of molten magnesium an amount of zirconium equal to between 0.3 and 0.6 percent of the weight of the magnesium followed by dispersing in the resulting melt an amount of aluminum equal to from about 3 to 5 percent of the weight of the zirconium added to the melt, settling the so-treated melt, alloying from 5 to 7 percent of aluminum and 0.75 to 1.5 percent of zinc by weight with a second batch of molten magnesium, blending together substantially equal weights of supernatant portions of batches 1 and 2, and thereafter casting the so-treated blend into ingot.

2. The method of making a fine homogeneous grain magnesium-base alloy ingot containing aluminum in amount between 2.5 and 3.5 percent and zinc between 0.75 and 1.5 percent by weight which comprises dispersing in a first batch of molten magnesium an amount of zirconium equal to between 0.3 and 0.6 percent of the weight of the magnesium followed by dispersing in the resulting melt an amount of aluminum equal to from about 3 to 5 percent of the weight of the zirconium added to the melt, settling the so-treated melt, alloying from 5 to 7 percent of aluminum and 0.75 to 1.5 percent of zinc by weight with a second batch of molten magnesium, blending together substantially equal weights of supernatant portions of batches 1 and 2, treating the resulting blend with a small amount of halogenated carbonaceous material, and thereafter casting the so-treated blend into ingot.

3. The method of making a fine homogeneous grain magnesium-base alloy ingot containing aluminum in amount between 2.5 and 3.5 percent and zinc between 0.75 and 1.5 percent by weight which comprises dispersing in a first batch of molten magnesium an amount of zirconium equal to between 0.3 and 0.6 percent of the weight of the magnesium followed by dispersing in the resulting melt an amount of aluminum equal to from about 3 to 5 percent of the weight of the zirconium added to the melt, settling the so-treated melt, alloying from 5 to 7 percent of aluminum and 0.75 to 1.5 percent of zinc by weight with a second batch of molten magnesium, treating the resulting batch of alloy with a carbonaceous grain refining material, blending together substantially equal weights of supernatant portions of batches 1 and 2, treating the resulting blend with a small amount of halogenated carbonaceous material, and thereafter casting the so-treated blend into ingot.

4. The method of making a fine homogeneous grain magnesium-base alloy ingot containing aluminum in amount between 2.5 and 3.5 percent and zinc between 0.75 and 1.5 percent by weight which comprises dispersing in a first batch of molten magnesium an amount of zirconium equal to between 0.3 and 0.6 percent of the weight of the magnesium followed by dispersing in the resulting melt an amount of aluminum equal to from about 3 to 5 percent of the weight of the zirconium added to the melt, settling the so-treated melt, alloying from 5 to 7 percent of aluminum and 0.75 to 1.5 percent of zinc by weight with a second batch of molten magnesium, treating the resulting batch of alloy with a carbonaceous grain refining material, blending together substantially equal weights of supernatant portions of batches 1 and 2, treating the resulting blend with a small amount of halogenated carbonaceous material, adding up to 0.04 percent of calcium to the blend, and thereafter casting the so-treated blend into ingot.

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