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METHOD OF UNIFORMLY REDUCING THE DIMENSIONS OF MAGNESIUM SHAPES

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The invention relates to a method of uniformly reducing the dimensions of magnesium shapes and more particularly to reducing their cross-sectional area by submerging them in a bath of specified composition accompanied by movement of the submerged shape or of the bath relative to each other.

By the term magnesium, as used herein, is meant magnesium metal and magnesium alloys wherein magnesium comprises at least 85 percent of the alloy by weight.

In the production of extruded, rolled, molded, or cast shapes, or articles fabricated from such shapes as by welding, it is frequently not feasible to produce the shape in the final desired dimensions in the fundamental forming process. For example, in the extrusion of magnesium tubing and sheet which are of thin gauge, the metal tends to buckle, stretch, or collapse at the extrusion temperature if the gauge is too thin to resist fully the external forces accompanying the forming process. A further example wherein the production of a magnesium article of the final desired dimensions is not feasible is the production of such articles as templates and the like which are unsymmetrical or those which are of a more or less intricate or complex geometric design. It is therefore highly desirable that there be a method of uniformly reducing the cross-sectional area of such shapes to achieve the final desired dimensions subsequent to the fundamental forming process.

Incidental to the treatment of magnesium shapes in pickling baths and cleaning baths there has been some reduction in the cross-sectional areas and dimensions of the treated shapes. Such reduction has been unsatisfactory and usually has been an erratic uncontrollable effect due to the corrosive nature of such baths. At least one of the following undesirable surface conditions has accompanied reduction in size of magnesium shapes treated in such baths: Depressions, dimples, or pits are formed. Certain areas are relatively unaffected, leaving pimples and raised areas or other irregularities resulting in a general surface roughness. Gas bubbles form causing flow lines and channels. The acid ingredient of the bath, if sufficiently high to impart a reasonably long life and reasonably fast action to the bath, results in a bath which is too vigorous, generates high heat, and results in a mottled and roughened surface. If the acid ingredient in the bath is maintained sufficiently low to avoid the mottled or roughened effect described above, an excessively long and impracticable treating time is required. The baths in use, to avoid mottled or roughened surfaces, are of relatively low activity and therefore become rapidly depleted in effective ingredients. As a result, the recesses and other less readily accessible areas of the treated magnesium shapes are not reduced in size as much as are the more exposed areas.

We have discovered a highly efficient and economical method of producing uniformly sized magnesium shapes without the undesirable concomitant formation of pits, ridges, striations, raised areas, rounded corners, and the like by submerging magnesium shapes for a measured period of time in a bath of specified composition, the bath and the submerged shape being in motion relative to each other.

The method is efficient because it produces a smooth finished magnesium article uniformly reduced to desired

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dimensions with a relatively small expenditure of time and effort. It is economical, both because the initial cost of the bath is low and because the bath can be revived repeatedly during use merely by adding additional nitric acid to the bath and by separating excess nitrate salt from the bath as by decantation or filtration.

The method of our invention of uniformly reducing the dimensions of magnesium shapes is not to be confused with current practices of pickling to etch and chromate coat magnesium surfaces, or with cleaning to remove mill scale, graphite, etc. or with methods to brighten the magnesium surface.

Briefly the invention contemplates reducing the dimensions of magnesium shapes, particularly the thickness of such shapes and thereby to attain accurately the desired size subsequent to the fundamental forming process.

In practicing the invention, a clean magnesium shape is submerged in an aqueous bath comprising from about 0.70 to 25.0 percent by weight of nitric acid and from about 3 percent by weight to the saturation point of a nitrate selected from the nitrates of ammonium, alkali, and alkaline earth nitrates. The usual amount of nitric acid employed is between about 5.0 and 18.0 percent and that of the nitrate is between about 10 and 30 percent.

The temperature of the bath, though not critical, is usually from room temperature to about 110° F. The time of submergence depends upon the concentration and temperature of the bath and the thickness of magnesium sought to be removed, a longer submergence being required at lower concentrations and temperatures and when more magnesium is sought to be removed. The time will vary anywhere from 2 or 3 minutes to 20 or more minutes. It is necessary that there be a relative motion between the bath and the submerged magnesium shape. This may be accomplished by a manual or mechanical means, as by slowly oscillating or rotating the submerged shape. The complexity of the path of the moving shape will be determined by the design or intricacy of the shape. The movement need be only sufficiently complex to bring fresh solution in contact with the surface of the submerged shape. A regular solid, for example a prism, parallelepiped or cylinder, may be slowly moved about its own axis and inverted once or twice during the period of submergence. The recommended procedure for sizing elongated shapes such as tubes, rods, and the like is to submerge the shape in a position which is about 3° to 30° from the horizontal and rotate it slowly, e.g., 1 r.p.m., and also turn it completely around, i.e., end for end, slowly about once every 3 to 4 minutes of submergence. On the other hand, a shape having deep recesses or narrow channels therein, wherein air pockets may form, is to be moved in such a manner as to bring all surfaces into contact with the solution at almost the same time, as by rapidly changing the orientation of the shape in the bath. After the dimensions have been reduced in size to that desired, the submerged magnesium shape is removed from the bath and rinsed thoroughly with water.

It is often desirable to reduce only the cross-sectional area or to reduce only certain selected dimensions of a magnesium shape. This may readily be achieved by protecting those portions of the shape to be submerged which are not to be reduced in size. One method of attaining this protection is by masking or stopping off the areas where reduction in thickness is not desired, i.e., where action by the bath is to be prevented, by applying a coating of a material impervious to the bath formulation such as a resin, e.g., polyvinyl chloride, to such areas. An example of an area where action of a bath is undesirable is a small area where the shapes will be welded since a better weld is obtained between relatively thick magnesium pieces than between thin ones.

In some instances it is desirable to reduce the thickness of but one or a few small areas on a magnesium alloy shape or article. In other instances it is desirable to enlarge one or more openings that are so located or shaped that they do not lend themselves readily to conventional means of enlargement such as reaming, drilling, or cutting. Such reduction in thickness or enlargement of an opening may be accomplished by first masking with a protective resin film all areas which are not to be subjected to the action of the novel solution of the invention and thereafter submerging the shape or article in the solution according to the method of the invention.

The following example is illustrative of a mode of carrying out the invention:

An article of magnesium alloy composed of 96 percent magnesium, 3 percent aluminum, and 1 percent zinc, constructed of four 0.09 inch thick rectangular panels and welded into a rectangular instrument housing in the shape of hollow tube open on each end and of about 36" x 12" x 12" outside dimensions, was fabricated. A margin was defined at the ends of each of the four panels comprising the housing tube by applying 1 inch wide strips of polyvinyl chloride resin transversely about the inside and outside of the tube so that they extended about 1 inch inwardly from the open ends of the tube. The margin thus defined was thereby prevented from direct contact with the bath during subsequent immersion therein and were not reduced in thickness. A margin of original thickness was therefore preserved for the purpose of obtaining a better weld joint when the housing tube was installed following the reduction in size by immersion in the bath.

A bath was prepared according to the invention by dissolving 180 ml. (about 24.5 percent by weight) of 70 percent aqueous nitric acid and 300 g. (about 30 percent by weight) of sodium nitrate in a liter of water. The housing was first degreased by immersing it in an aqueous alkaline bath, rinsed with water, and dried according to the conventional manner of preparing magnesium for subsequent surface treatment. The cleaned housing was then submerged in the bath in a substantially horizontal position for 9 minutes while it was slowly turned both about its own axis and about a horizontal axis. It was thereafter rinsed well with water and dried.

The panels composing the housing were found to be uniformly reduced from a thickness of 0.09" to one of 0.06". The corners were not rounded. The interior and exterior surfaces of the housing were smooth and of uniform thickness without any irregularities. The individual panels of the housing were sized precisely as desired. The polyvinyl chloride resin was stripped off leaving an inch border at each end of the housing, which had not been reduced in thickness.

The bath of the example may be continued in use for an indefinite period. As the bath is continued in use, there is formed magnesium nitrate by the action of the bath on the magnesium metal so that the magnesium nitrate concentration in solution in the bath increases until the bath is saturated. Also, as the magnesium shapes are removed, some of the bath adheres to them. This adhering bath, known as drag-out, contains nitric acid

and sodium nitrate of the original bath. Since the magnesium nitrate is being produced in the process and the sodium nitrate is not, the sodium nitrate is therefore gradually replaced by magnesium nitrate as the active nitrate of the bath. At intervals, as determined by control tests, it is necessary to add nitric acid to the bath to maintain the bath within the desired concentration range; no other additive is necessary. In time the magnesium nitrate produced is in excess of the amount soluble in the bath and it settles out. This accumulated magnesium nitrate may be separated at intervals by decantation, siphoning, or otherwise drawing off the saturated aqueous bath leaving the excess magnesium nitrate, or the excess magnesium nitrate may be removed by filtration.

Having thus described our invention, what we claim and desire to protect by Letters Patent is:

1. The method of uniformly reducing the dimensions of a metal shape composed of at least 85 percent by weight of magnesium which consists of submerging said shape in an aqueous bath consisting of from about 0.70 to 25.0 percent of nitric acid, from about 3.0 percent to the point of saturation of a nitrate selected from the class consisting of ammonium nitrate and alkali metal and alkaline earth metal nitrates, and maintaining a relative motion between said shape and said bath during at least a portion of the duration of the submergence, and thereafter removing said article from the bath and rinsing it with water.

2. The method of claim 1 wherein the nitrate is sodium nitrate.

3. The method of claim 1 wherein the nitrate is magnesium nitrate.

4. The method of claim 1 wherein the bath is circulated while said metal shape is submerged therein.

5. The method of uniformly reducing selected cross-sections of the members of an article of at least 85 percent by weight of magnesium metal which consists of masking the surface of those portions through which the cross-section is to remain unreduced with a coating material which is impervious to aqueous nitric acid solution, submerging the article in a solution prepared by admixing from about 10 to 350 ml. of from 50 to 70 percent aqueous nitric acid, from about 30 ml. to the point of saturation of a nitrate selected from the class consisting of ammonium, alkali metal and alkaline earth metal nitrates, and enough water to make 1 liter of solution at room temperature, and maintaining a state of relative motion between said shape and said solution for at least a portion of the time of submergence until the selected cross-section of the members of said article are reduced to the desired dimensions, removing the submerged article from said bath, and rinsing the article with water.

6. The method of claim 5 wherein said coating is polyvinyl chloride.

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