The invention relates to an improved method of removing contaminants adhering to the surfaces of articles of magnesium and its alloys. It more particularly concerns an improved method of pickling articles of magnesium and its alloys, which have become dirty especially as a result of hot mechanical working especially between lubricated forming devices such as mill rolls, forging, and drawing dies.

In my prior patent No. 2,602,839, I have disclosed a method of removing mill scale and the like from rolled articles of magnesium and magnesium-base alloys based upon pickling the articles in an aqueous solution of certain carboxylic acids, particularly acetic acid. One of the disadvantages of pickling articles of magnesium and the magnesium-base alloys in an aqueous acetic acid solution is that there results a considerable metal loss, and, in the case of the magnesium-base alloys containing aluminum, a black smut is formed over the surface of the articles giving it a black speckled appearance. A further disadvantage of the acetic acid pickle is that graphite, oftentimes present in the lubricants used on the metal when hot working it with dies and the like, is difficult to remove especially when deeply embedded.

I have now discovered that these disadvantages are overcome and a better pickling action is obtained with a reduced loss of metal by including in the acetic acid pickling solution the nitrate of an alkali metal, alkali earth metal, or ammonium. The invention then consists of the improved acetic acid pickling method hereinafter fully described and particularly pointed out in the claims.

In carrying out the invention, acetic acid is used in water solution in a concentration generally of about 5 to 45 per cent by weight. For special purposes as in pickling magnesium or magnesium-base alloys in sheet form a concentration of about 15 to 25 per cent is to be preferred. A generally effective concentration is about 20 per cent. In cases where the surface to be pickled is heavily contaminated with graphite, an acetic acid concentration of about 30 to 40 per cent is preferred.

The alkali metal, alkali earth metal or ammonium nitrate is added to the acid solution in an amount affording a nitrate (NO₃⁻) concentration of 2 to 6 per cent. A generally useful concentration is about 2 to 6 per cent, the preferred concentration being about 5 per cent.

It is desirable first to degrease the article to be pickled so as to remove excessive oil or grease contamination. The articles are then immersed or otherwise subjected to the action of the acetic acid-metal nitrate solution for a time sufficient to remove the surface contaminants but insufficient to permit any substantial attack upon the underlying metal. The duration of the treatment is easily gauged by visual examination during the operation. The work usually becomes clean and bright within about 1 minute. After cleaning with the acetic acid-metal nitrate solution, the article is rinsed with water to remove the solution and dried.

The following examples are illustrative of the invention:

**Example 1**
A piece of rolled sheet 3" x 6" x 0.040" of a magnesium-base alloy having a nominal composition of 3 per cent aluminum, 1 per cent zinc, 0.3 per cent manganese, the balance being magnesium, is degreased and immersed at room temperature for 1 minute in a solution composed of 400 grams of acetic acid and 400 grams of sodium nitrate dissolved in enough water to make 1 liter of solution. The temperature of the solution is 76°F., the pH 2.3. The piece before immersion has the usual mill scale embedded in the surface. After the immersion, the piece is clean and bright. The thickness is reduced 0.0004 inch. In comparison, acetic acid alone in water in a concentration of 400 grams per liter leaves the surface of a similar as-rolled and degreased piece of sheet speckled with black smut when pickled therein for 1 minute. The metal thickness is reduced 0.0014 inch.

**Example 2**
A similar piece of as-rolled sheet to that of Example 1 is degreased and immersed at room temperature for 1 minute in a solution composed of 400 grams of acetic acid and 50 grams of magnesium nitrate (Mg(NO₃)₂) and enough water to make 1 liter of solution. The pH of the solution is 1.45. After the immersion, the piece is clean and bright and has lost 0.0004 inch of thickness.

Of the various nitrates which may be used according to the invention, e. g. NaNO₃, KNO₃, LiNO₃, NH₄NO₃, Ca(NO₃)₂, Mg(NO₃)₂, I prefer to use Mg(NO₃)₂.

Among the advantages of the invention are that a more complete cleaning is had without formation of any smut produced when acetic acid alone is used, especially on magnesium-base alloys containing aluminum. There is a greatly reduced attack on the metal underlying the surface.
contaminants. The pickling operation is rapid. I claim:

1. A method of removing surface contaminants from articles of magnesium and its alloys which comprises subjecting the article to the action of a solution consisting of acetic acid and a water-soluble nitrate selected from the group consisting of the alkali metal nitrates, alkali earth metal nitrates, and ammonium nitrate dissolved in water, the concentration of the acetic acid being 5 to 45 per cent by weight and the water-soluble nitrate producing an NO₃ concentration of 1.5 to 12 per cent, at room temperature for a time sufficient to remove the surface contaminants.

2. The method according to claim 1 in which the acetic acid concentration is 15 to 25 per cent and the water-soluble nitrate produces an NO₃ concentration of 2 to 6 per cent.

3. The method according to claim 1 in which the water-soluble nitrate is sodium nitrate.

4. The method according to claim 1 in which the water-soluble nitrate is magnesium nitrate.

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