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NICKEL DEPOSITING BATH AND METHOD

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This invention relates to the electrodeposition of nickel.

Among the objects of the invention are the provision of a bath and operating conditions which will enable the deposition of the nickel at a high current density and consequent rapidity, to obtain a fine grain structure of good color and to produce a deposit of a high degree of ductility.

The electrolyte, according to my invention, consists essentially of an aqueous solution of a nickel salt of a strong acid (hydrochloric acid), a nickel salt of a weak acid (acetic acid), and a sufficient quantity of the strong acid (hydrochloric acid) to adjust the pH value of the solution to the desired range. In general, the total nickel concentration may vary from about 4.5 to about 17.0 oz./gal., and the ratio of nickel from chloride to nickel from acetate may vary from 4.0 to 12.0 oz./gal. nickel from chloride to 0.5 to 5.0 oz./gal. nickel from acetate. A particularly favorable bath concentration is about 7.5 to 10.0 oz./gal. of total nickel, of which from 4.0 to 5.0 oz./gal. is supplied by nickel chloride and from 3.5 to 5.0 oz./gal. is supplied by nickel acetate. Experiments conducted by me have shown an important cooperative effect of the particular bath ingredients employed. Aqueous solutions of nickel chloride alone offer good electrical conductivity, but usually produce hard, brittle deposits. Aqueous solutions of nickel acetate show a relatively poor conductivity and are easily decomposed by hydrochloric acid in quantities necessary to adjust the pH value of the bath. The performance of a bath in which nickel chloride, nickel acetate and hydrochloric acid are all present in the proper proportions, is such as to strongly indicate that an important and favorable interaction occurs between the bath components, as a result of which the bath is remarkably free from hydrogen evolution at the cathode, and highly ductile nickel deposits of fine-grained, semi-bright character and light in color can be obtained over a current density range extending from around 15 amps./sq. ft. to far beyond 400 amps./sq. ft. of cathode surface. Upon a basis metal of cold rolled steel, properly prepared, the deposit shows an almost complete freedom from porosity. These observations have been made in a bath containing 8.5 oz./gal. total nickel, supplied in equal proportions by nickel chloride and nickel acetate, with sufficient concentrated hydrochloric acid to adjust the pH value of the bath to the range of 4.0 to 5.5 electrometric and operated at a temperature

of about 130° F. The resistivity of such a bath at 130° F. was found to be about 8.75 ohms/c. c. at the pH value of 4.5. The resistivity tends to decrease at higher temperatures and lower pH values. The anode efficiency and the cathode efficiency were found to be 90% or better, and the bath composition and pH value were observed to remain approximately constant, with rolled nickel anodes of about twice the area of the cathode, over a long period of use. This particular bath composition is preferred over other concentrations and ratios of components, because of the particularly favorable operating conditions and the wide range of current densities over which the deposit remains of good color, semi-bright and ductile.

The operating temperature for any of the bath compositions according to my invention may be anywhere from room temperature to below the boiling point, but the temperature range of 130 to 150 is considered most practical for commercial use.

Light, semi-bright, ductile deposits have been obtained from baths containing 12.0 oz./gal. nickel from chloride and about 5.0 oz./gal. nickel from acetate, with sufficient hydrochloric acid to adjust the pH value to 4.5, when operated above 350 amps./sq. ft. of cathode surface. Similar deposits have been obtained at 15 to 200 amps./sq. ft. of cathode surface from baths containing 4.0 oz./gal. nickel from chloride and about 5.0 oz./gal. nickel from acetate, with sufficient hydrochloric acid to adjust the pH value of the bath to 4.65 to 5.00. Likewise, such deposits have been obtained at 10 to 35 amps./sq. ft. of cathode surface in baths containing 4.2 oz./gal. nickel from chloride and 0.5 oz./gal. nickel from acetate, with sufficient hydrochloric acid to adjust the pH value to 4.55. At a pH value from 1.0 to 4.0, this bath gave a light matte, ductile deposit over a somewhat wider range of current densities, but the cathode efficiency is generally low and the resistivity high. These observations were obtained at a temperature in the neighborhood of 130° F. At pH values below 2.0 there is a marked tendency in many of the baths for instability and decomposition of the acetate to occur, and I consider the useful range of pH to be between 2.0 and 5.5 in most respects, such as the character of the deposit and the stability and operating conditions of the bath.

While I have mentioned bath compositions with as low as 0.5 oz./gal. of nickel from nickel acetate, the operating conditions with baths hav-

ing so small a quantity of nickel acetate are not nearly so favorable as those in which greater quantities of nickel acetate are present. On the other hand, it has also been observed that the operating conditions are not quite so favorable when the quantity of nickel from acetate is in substantial excess of the nickel from chloride. In general, a bath in which the nickel is supplied in about equal proportions from the chloride and the acetate appears to be more suitable for general use. It should be pointed out that the function of the nickel acetate is that of an electrolyte, to furnish nickel and acetate ions for conductivity and nickel ions for deposition, in addition to the function of the nickel acetate as a buffer under the conditions of use. For this reason, the nickel acetate should constitute a substantial proportion of the electrolyte. While some other acetate salt might serve the function of a buffer, and salts of other metals than nickel and acids other than those employed according to my invention might serve to increase the conductivity of the bath, it is believed to be important for the proper action of my bath to avoid such substitutes for any of the components of the bath specified by me. Alkali metals and their salts are particularly objectionable as giving rise to decomposition of water and secondary deposition at the cathode, with consequent impairment of the physical character of the nickel deposit. In other words, the invention contemplates a bath in which there is a common ion effect due to the nickel from the two sources (nickel chloride and nickel acetate) and a common ion effect from the chloride radical of the two sources (nickel chloride and hydrochloric acid), so that the ionization of the nickel chloride is somewhat depressed by the chloride ion of the hydrochloric acid, with consequent increase in the ionization of the nickel acetate.

I claim:

1. A nickel depositing bath consisting essentially of an aqueous solution of nickel chloride and nickel acetate adjusted by hydrochloric acid to a pH value within the range of 2.0 to 5.5 electrometric, the total nickel content of the bath being from 4.5 to 17.0 oz./gal., of which 4.0 to 12.0 oz./gal. are supplied by the nickel chloride and from 0.5 to 5.0 oz./gal are supplied by the nickel acetate.

2. A nickel depositing bath consisting essentially of an aqueous solution of nickel chloride

and nickel acetate adjusted by hydrochloric acid to a pH value within the range of 4.0 to 5.5 electrometric, the total nickel content of the bath being within the range of about 7.5 to 10.0 oz./gal., of which from about 4.0 to 5.0 oz./gal are supplied by the nickel chloride and from about 3.5 to about 5.0 oz./gal. are supplied by the nickel acetate.

3. A nickel depositing bath consisting essentially of an aqueous solution of nickel chloride and nickel acetate adjusted by hydrochloric acid to a pH value in the neighborhood of 4.5 electrometric, the total nickel content of the bath being approximately 8.5 oz./gal., of which approximately equal proportions are supplied by the nickel chloride and the nickel acetate.

4. A method of electrodepositing nickel, which comprises passing an electrolyzing current from an anode to a cathode through an electrolyte consisting essentially of an aqueous solution of nickel chloride and nickel acetate, with sufficient hydrochloric acid to adjust the acidity of the bath to a pH value within the range of 2.0 to 5.5, the total nickel content of the bath being within the range of 4.5 to 17.0 oz./gal., of which at least 4.0 oz./gal. are supplied by nickel chloride and at least 0.5 oz./gal. are supplied by nickel acetate.

5. A method of electrodepositing nickel, which comprises passing an electrolyzing current from an anode to a cathode through an electrolyte consisting essentially of an aqueous solution of nickel chloride and nickel acetate, with sufficient hydrochloric acid to adjust the acidity of the bath to a pH value within the range of 4.0 to 5.5, the total nickel content of the bath being from 4.5 to 17.0 oz./gal., of which at least 4.0 oz./gal. are supplied by the nickel chloride and at least 3.5 oz./gal. are supplied by the nickel acetate.

6. The method of electrodepositing nickel, which comprises passing an electrolyzing current from an anode to a cathode through an electrolyte consisting essentially of an aqueous solution of nickel chloride and nickel acetate, with sufficient hydrochloric acid to adjust the acidity of the bath to a pH value in the neighborhood of 4.5, the total nickel content of the bath being about 8.5 oz./gal. supplied in approximately equal proportions by the nickel chloride and the nickel acetate.

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