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PLATING SURGICAL NEEDLES

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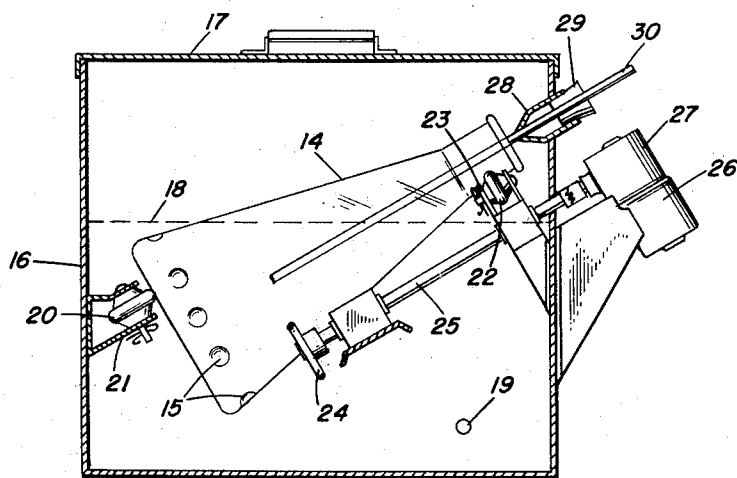
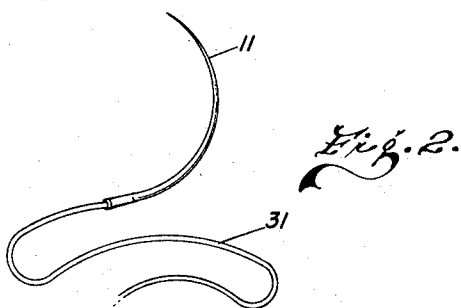
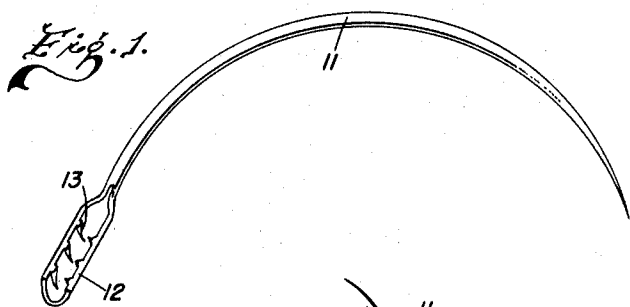


Fig. 3.

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PLATING SURGICAL NEEDLES

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3 Claims. (Cl. 128—339)

This invention relates to improvements in nickel-nickel phosphide plated needles for surgical sutures, the sutured surgical needle and methods and apparatus for producing them.

At present surgical needles are produced from a fairly high carbon steel so as to give a tough sharp needle point for use in suture work. Many of the needles are of the eyeless type in which various means are used for connecting a single strand of a suture into a needle. Such needled sutures are then stored until time for use. Certain sutures, such as gut sutures, must be stored in conditioning fluid such that the suture is in proper condition for use when removed from the storing container. Others for use with silk sutures, or other types of sutures may be stored dry. Under either condition it is necessary that the needles remain bright and shiny and appear free from contamination for esthetic reasons, as well as practical utility. In many instances the point of the needle is of a much harder metal than the shank or butt end of the needle.

In the past it has frequently been customary to store the needled gut sutures in a conditioning fluid to which a rust inhibitor had been added. The use of such a rust inhibitor induces additional problems as it may deleteriously effect the gut or the label which is present to identify the needled suture. Among other materials sodium nitrite has been used, which is apt to discolor paper.

Obviously, it would be advantageous if a needle were available which did not require special handling to prevent corrosion. The requirements are very rigorous because the cracks and crevices present in the flange portion of the eyeless needle, which are necessary so that the needle may be clamped around the suture, are such that it is difficult to clean the needle, and there are many corners in which corrosion may be initiated. It would seem obvious to use a plated needle to prevent corrosion. However, conventional plates such as nickel plate are not satisfactory because if a plate on the needle is sufficiently thick to prevent corrosion, the plate tends to become brittle, and flakes off when the flange of the needle is closed about the suture. It would thus seem that there are conflicting requirements, which have in the past presented insuperable difficulties.

It is necessary therefore to secure a plate which is smooth, uniform and sufficiently ductile to remain on the needle during subsequent operations, which is bright and shiny in order to be acceptable, and insures corrosion-resistance. The nickel-nickel phosphide plate is superior in corrosion-resistance to electrolytic nickel plate. Surprisingly it is found that an electrodeless plate of nickel-nickel phosphide is of such characteristics that a suitable plated needle is obtained. It may be that the nickel phosphide in the nickel gives sufficient ductility and resilience so that the plate will remain on the surface of the needle without rupture even during closing operations, and the superior throwing power of an electrodeless plating solution insures that the cracks and crevices in the flange of

the needle are adequately plated and that the corners and re-entrant edges are adequately plated before the plate on any portion becomes so thick as to be brittle. Additionally, the nickel-nickel phosphide plate is thin enough so that sharp points and edges of the needles remain sharp.

Electrodeless plating solutions and processes have been disclosed from time to time. One which is particularly advantageous in conjunction with the present manufacture of surgical needles is that described in a U. S. patent to Brenner, 2,532,283, Nickel Plating by Chemical Reduction. The compositions and methods there described when applied to a mass of surgical needles gives amazingly satisfactory results. It is desirable that to insure a smooth and uniform plate over all surfaces of the needles, that the needles be agitated during the plating operations, and by using an agitating device so that the needles are tumbled. Continuous agitation is convenient but not necessary. The nickel-nickel phosphide coating is smoothly and uniformly adherent over the entire surface of the needles.

It is preferred that the nickel be furnished by soluble nickel salts such that not substantially in excess of four parts by weight to 100 parts by weight of solution of nickel ion is present and the hypophosphite radicals be present not substantially in excess of three parts by weight per 100 parts by solution. It is preferred that the solutions be hot during use and whereas the nickel of the solutions can be exhausted during the plating process, normally the amount of nickel-nickel phosphide required for the plate is so small that it is more convenient to only partly utilize the nickel present in the solution and replace the solutions, because the plating solutions will not remain stable over an extended period of time.

The needles must be clean prior to plating, and conventional cleaning processes may be used, such as either a caustic or alkaline phosphate, or a solvent degreasing, or both followed by acid dip which activates the surface of the needles.

It is very difficult to measure the exact thickness of the coating on the surface of the needles either directly with a micrometer, or indirectly by area and weight relationships. As nearly as can be determined by measurements, a plate which is uniform and from about 0.05 to 0.35 thousandth of an inch gives a satisfactory plate which is sufficiently thick to be corrosion-resistant, and sufficiently thin to be ductile enough to permit closing the flange of the needle about the suture without rupture. The nickel-nickel phosphide coating contains more than one phase, and on analysis of the plate obtained by prolonged plating, had a phosphorus content of 2.17%, although this may vary with thickness and plating conditions. Some publications report a higher phosphorus content.

The accompanying drawings show:

Figure 1 a curved eyeless needle,

Figure 2 a needled suture,

Figure 3 the plating apparatus.

As shown by Figure 1 conventional eyeless needles have a flange 12 which consists of an opened portion of the needle in which there are serrations 13. The needles in this form are plated. When being attached to the suture itself, the suture is placed in the flange, adjacent to the serrations, and the flange is closed about the suture so that it clamps and holds the suture 31, as shown in Figure 2. The drawing down of the flange about the suture is a swaging operation which is frequently accomplished by drawing the needle forceably through a closing die which gives both a bending and a shearing action, which tends to scrape off the plate from the flange portion of the needle. The interior portions of the flange are particularly difficult to plate smoothly and uniformly and are areas in which corrosion most easily occurs. It

is impractical to attempt to electroplate the flange of a needle so that the interior portions are corrosion-protected by the plate, and the external portions are not so heavily plated as to become brittle.

As shown in Figure 3 the needles are conveniently plated in a rotating container. Because surgical needles are rather small and are conveniently plated in fairly small batches, a glass flask is convenient as a plating container. As shown in Figure 3 a conventional Erlenmeyer flask 14 has agitating dents 15 formed in the side near the bottom thereof. For example a 4-liter Erlenmeyer flask may have about a dozen dents formed equally spaced around the lower periphery by heating the glass in spots and using the blunt end of a file to force the glass wall interiorly of the flask to form a dent. In rotating, these dents pick up the needles and cause the mass of needles to move with the flask and be tumbled rather than sliding around the interior surface of the flask. The number or shape of the dents is not critical.

The flask is supported in a hot water bath 16. The hot water bath may be of stainless steel. It is preferably provided with a cover 17. The bath water 18 may be heated by heating means 19, which may either be an electrical heater or a steam injector to maintain the bath at a desired temperature usually near boiling. Inside the hot water bath is a group of support rollers. A bottom support roller supports the flask axially, and consists of a non-corrosive roller 20 rotatably mounted in a support bracket 21. The roller is preferably of a non-corrosive material which is self-lubricating, such as "Teflon" or "Nylon." Such plastic self-lubricating materials do not require lubrication and resist the effects of the boiling water for long periods. The neck of the flask is supported on neck support rollers 22 which are journaled in the neck support roller bracket 23. Near the lower part of the periphery of the flask are the rotating rollers 24 which are mounted on shafts 25, which are in turn rotated by the stirring motor 26 through a gear reducer 27. As so journaled the flask can easily be placed in and removed from the hot water bath with a minimum of effort. Opposite the neck of the flask, when in position on the rollers, is a thermometer support 28, which is a tubular bracket, having a hole at the bottom thereof for the insertion of a thermometer. A thermometer 30 is positioned in the thermometer support by a stopper 29. By inserting the stopper down into the interior flask the temperature of the plating solution during use is ascertained.

Obviously, the apparatus may be modified somewhat from the details shown for purposes of convenience depending upon the quantity of material to be plated in one batch, and the materials which are available for construction. The apparatus as shown, using a 4-liter flask, is convenient for plating a batch of several thousand surgical needles at one time, depending somewhat on the size of the needles being plated.

For small size batches, particularly for experimental or laboratory use, an ordinary 250 milliliter Erlenmeyer flask which is agitated by hand is conveniently used. An external magnet suitably moved may be used to agitate the needles. For plating on a larger commercial scale a glass lined barrel may be used.

As illustrative but not in limitation of the invention the following examples are given, in which parts are by weight unless otherwise specified.

Example 1

100 eyeless needles, designed for a number 00 size suture, are cleaned by washing twice in a mixture consisting of 40 volumes xylene, 40 volumes naphtha, and 20 volumes of methyl ethyl ketone, rinsed in acetone, and dried on tissue paper. The needles are immersed in a 5% hydrochloric acid solution for one minute, washed,

and immersed in 100 milliliters of a solution consisting of:

	Parts
Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	10
Sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$)	1
Ammonium chloride (NH_4Cl)	5
Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$) $\cdot 11\text{H}_2\text{O}$	10
Sufficient water to make up to 100 parts.	

The pH is adjusted to pH 8 using ammonium hydroxide. The needles are plated for 15 minutes with agitation, then removed from the solution, washed with water, and permitted to dry. A bright plating results which does not crack on sharp bending. The plate on the flange is so adherent that the flange may be bent until the flange itself breaks before the plate is detached from the needle.

Example 2

The above run is repeated leaving the needles in the solution for 30 minutes. The resulting needles show a bright plate which is sufficiently adherent that the flange may be sharply bent without cracking, or the needles may be attached to sutures using standard flanging dies without damage to the plate. The plate has the brightness characteristic of a bright nickel plate, even though the plate itself is a nickel-nickel phosphide.

Example 3

Example 1 is repeated using a plating solution the pH of which is not adjusted, but which measures between pH 8 and pH 9 during the plating operations. The needles are permitted to remain in the plating solution for 30 minutes, then removed and washed. On testing the thickness of the plate it is found to be approximately 0.0002 inch.

Example 4

A group of 100 larger needles, designed for a number 2 size suture are washed twice with a solution consisting of 40 volumes of xylene, 40 volumes of naphtha, and 20 volumes of methyl ethyl ketone, then rinsed in acetone and dried on tissue paper. The needles are rinsed in 5% hydrochloric acid for one minute and then immersed for 10 minutes in the plating solution of Example 1 at a pH of 8. The needles are then removed, washed with water, and permitted to dry. A bright corrosion-resistant ductile adherent plate on the needles results.

Example 5

100 very small needles, designed for a 6/0 suture are treated by following the method of Example 4 except that the needles are plated for 45 minutes. A smooth bright uniform plate results. The needles may be attached to gut sutures without the plate on the flange portion cracking.

Example 6

100 needles, designed for a 6/0 suture, are washed in 100 milliliters of a washing solution consisting of 40 volumes of xylene, 40 volumes of ligroin, and 20 volumes of methyl ethyl ketone. The needles are washed twice, are then rinsed in acetone and dried on paper. The needles are immersed in a 2% hydrochloric acid solution for 2 minutes, washed and are then placed in the plating solution containing:

	Parts
Nickel chloride	20
Sodium hypophosphite	1
Ammonium chloride	5
Sodium citrate	10
Sufficient water to make up 100 parts.	

The needles are plated for 20 minutes with a solution at a pH of 8, and then washed and dried. A bright good plate is obtained, and which adheres to the needles during the flanging operation.

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Example 7

80 medium size, curved eyeless needles are washed twice in 200 milliliters of xylene, and then washed with 100 milliliters of acetone. The needles are air-dried and then pickled in 150 milliliters of 5% hydrochloric acid for 30 seconds and washed with water until the wash is neutral to litmus. A plating solution is prepared from:

	Grams
Nickel chloride -----	135.5
Sodium hypophosphite -----	13.5
Ammonium chloride -----	67.9
Sodium citrate -----	135.3
Water -----	1000

To this mixture is added 175 milliliters of concentrated ammonium hydroxide, thereby giving a solution with a pH of 8.4. 200 milliliters of this plating solution is heated to 80° C. and the needles are added thereto. The needles are agitated for 5 minutes, and then the solution is washed out with running water, the needles rinsed with water, and allowed to air-dry. A bright finish is obtained on the needles. After flanging on the gut sutures, and shaping of the needles no damage to the plate is observable under a microscope. The needles are corrosion-resistant when allowed to stand in air for a period of at least several months.

Example 8

5000 electropolished needles for a number 3/0 suture are allowed to stand in 500 milliliters of xylol for 20 minutes at room temperature. The needles are then rinsed with water and placed into a solution containing 200 milliliters of water, and 113.4 grams of "Metex T-103" at 86° C. The needles are allowed to stand for 3 minutes, and then rinsed. They are then placed in 500 milliliters of a solution containing 60 grams of NH_4HF_2 and 360 grams "Troxide" per liter for 30 seconds at room temperature. Fluorides are particularly useful in conditioning electropolished needles for plating. The needles are again rinsed with water, placed in a "Metex T-103" cleaning solution at 77° C. for 3 minutes, rinsed, and placed in 2000 milliliters of solution containing 120 grams of "Troxide" per liter for 1 minute at 56° C. and then rinsed with distilled water. "Metex T-103" is a sodium hydroxide, sodium metaphosphate, sodium metasilicate, plus wetting agent cleaner, and "Troxide" is a sodium bisulfate and wetting agent pickling agent prepared for the electroplating industry by MacDermid, Inc. of Waterbury, Connecticut.

In large size batches it is generally more convenient to use alkaline degreasers in aqueous solutions rather than solvents because of the fire hazard of using inflammable solvents. The needles may alternatively be cleaned by using a wash composed of 40 volumes of xylene, 40 volumes of ligroin, and 20 volumes of methyl ethyl ketone, and after washing the needles twice, rinsing with acetone, then air-drying on tissue paper.

After being cleaned 5000 needles are placed in 2500 milliliters of a plating solution, prepared as in Example 7, for 10 minutes at 81° C. in the apparatus described above and shown in the drawings. The needles are washed in distilled water, then air-dried. The needles then have a bright finish which does not flake, or peel when the needles are flanged on the gut and shaped. The needles are corrosion-resistant, and may be left exposed to air for periods of some several months without exhibiting corrosion or darkening of the plate. The needles are resistant to the normal conditioning fluids which may be used in storing of surgical gut sutures. A corrosion-in-

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hibitor is not required. The needles may be sterilized by heat, wet or dry, or given other conditioning treatment for the suture without damage or corrosion to the needles.

Example 9

50 eyeless needles are placed in a 250 milliliter Erlenmeyer flask in which they are kept for the entire plating process. The needles are washed twice with xylene, then with acetone. The acetone is poured off, and 5% aqueous hydrochloric acid is added. The needles are pickled for one minute, the acid poured off, and the needles are washed with water. The water is drained off and 100 milliliters of cold plating solution is immediately added. The plating solution contains:

	Percent
Nickel chloride -----	10
Sodium hypophosphite -----	1
Ammonium chloride -----	5
Sodium citrate -----	10
Water -----	74
Ammonium hydroxide—sufficient to adjust pH to 8.	

The flask is heated in a steam bath to 90° C., and the plating time measured from the time at which the temperature reaches 80° C. After plating for 8 minutes, the plating solution is poured off, and the needles are washed with water, then with acetone, and air-dried.

The plate is adherent, corrosion-resistant, and sufficiently ductile to permit attaching to sutures.

Example 10

Example 9 is repeated, the plating time being 15 minutes.

Corrosion-resistant needles with a ductile plate are obtained.

We claim:

1. A needled surgical suture comprising: an eyeless needle, a shiny, corrosion-resistant, flexible nickel-nickel phosphide plate on the surface thereof, and a flexible surgical suture swaged to said needle.

2. A needle for a surgical suture comprising: an eyeless needle, and on the surface thereof, a shiny, corrosion-resistant, flexible nickel-nickel phosphide plate.

3. A process for preparing shiny, corrosion-resistant nickel-nickel phosphide plated needled surgical sutures which comprises: cleaning steel eyeless needles, immersing in an aqueous solution of a nickel salt and an alkaline hypophosphite, the nickel ion being present in an amount not substantially in excess of about 4 parts by weight to about 100 parts by weight of said solution, the hypophosphite radical being present in an amount not substantially in excess of about 3 parts by weight to about 100 parts by weight of said solution, plating said needles by autocatalytic reaction of said nickel salt and said hypophosphite, at a temperature between about 70° C. and 100° C., until a ductile corrosion-resistant nickel-nickel phosphide plate is obtained, washing and drying the needles, and swaging said needles onto suture strands.

References Cited in the file of this patent

UNITED STATES PATENTS

145,217	Leiter -----	Dec. 2, 1873
230,857	Briggs -----	Aug. 10, 1880
1,960,117	Lydeard -----	May 22, 1934
2,532,283	Brenner -----	Dec. 5, 1950
2,620,028	Kohut -----	Dec. 2, 1952
2,694,019	Gutzeit -----	Nov. 9, 1954
2,816,846	Talmey -----	Dec. 17, 1957