United States Patent [19]

Economopoulos

[11] **3,785,878**

[45] **Jan. 15, 1974**

[54] **TREATMENT OF METAL ROD OR WIRE**

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[73] Assignee: **Centre National De Recherches Metallurgiques,** Brussels, Belgium

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July 10, 1970	Belgium	42902
July 10, 1970	Belgium	42903
July 10, 1970	Belgium	42905
July 14, 1970	Belgium	42910
July 14, 1970	Belgium	42911

[52] **U.S. Cl.** **148/18,** 148/143, 148/156, 148/157

[51] **Int. Cl.** **C21d 1/60,** C21d 9/52

[58] **Field of Search** ..... 148/18, 15, 14, 28, 148/20.6, 143, 155, 156, 157, 12, 12.3, 12.4; 134/9, 14, 15, 64, 122; 266/3, 4 R, 4 A, 4 S, 6 R, 6 S

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Primary Examiner—Charles N. Lovell
Attorney—Holman & Stern

[57] **ABSTRACT**

The process is particularly applicable to steel rod or wire. Metal rod or wire is cooled from a temperature at which the metal is in a first stable phase to a temperature at which the metal is in a second stable phase. Part of the cooling is by means of a liquid consisting of an aqueous fluid containing, in suspension and/or solution, one or more substances which modify the heat transfer conditions of the aqueous fluid. The liquid is at a temperature greater than 80° C and above 75 percent of its boiling temperature expressed in degrees centigrade. The heat transfer conditions of the liquid and the duration of cooling by means of the liquid are such that all, or the major part, of the optimum allotropic transformation of the metal takes place during cooling by means of the liquid.

19 Claims, 7 Drawing Figures

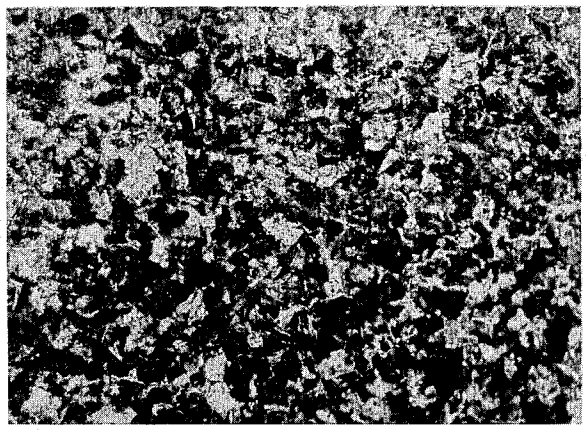


FIG. 1

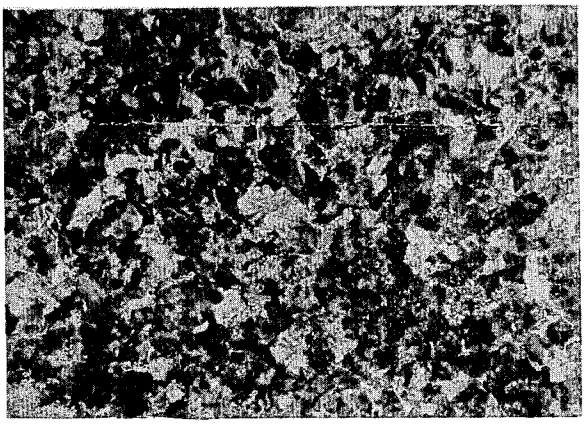


FIG. 2

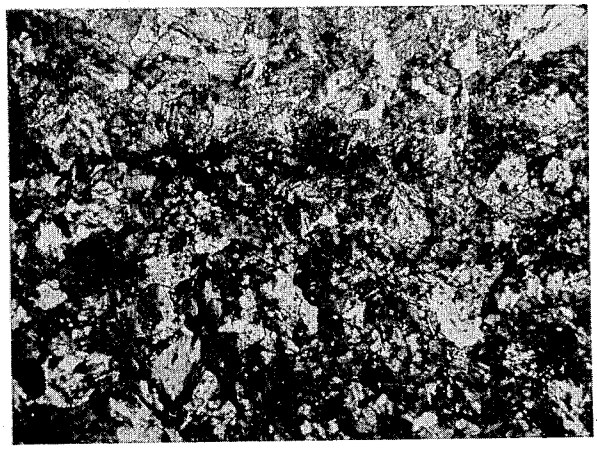


FIG.3



FIG.4

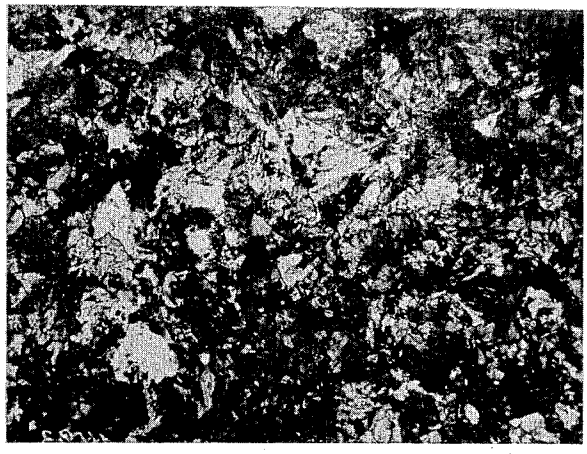


FIG. 5

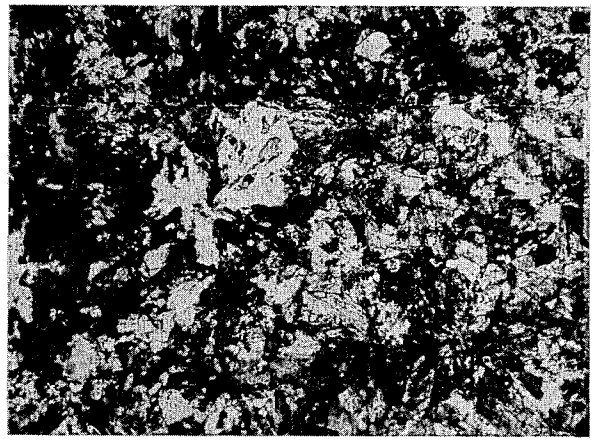


FIG. 6

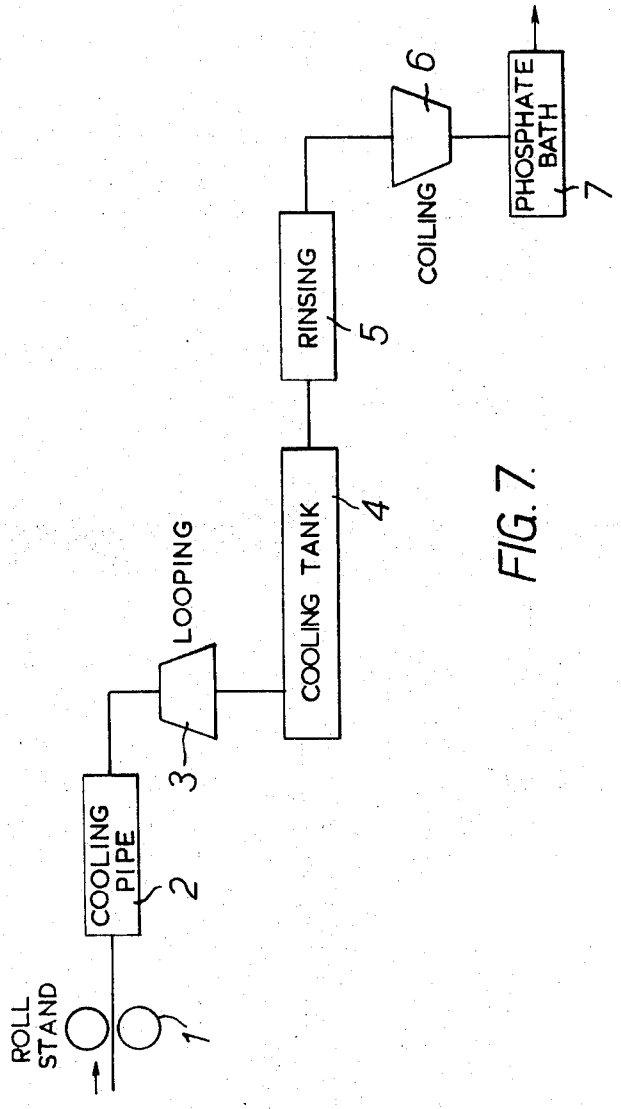


FIG. 7.

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TREATMENT OF METAL ROD OR WIRE

THE PRESENT INVENTION relates to a process for the treatment of metal rod or wire, produced by rolling, drawing, or otherwise. Such a process is particularly applicable to the different stages of the manufacture of steel wire from wire rod.

As is known the manufacture of steel wire usually comprises two types of operation. The first is the hot rolling of steel to form rod, generally termed wire rod, having a diameter which is usually greater than 5mm. The wire rod is usually wound into reels or flat coils. The second type of operation is the drawing of this wire rod to produce a wire, an operation effected in one or more passes.

If we examine first of all the problem of the treatment of hot-rolled wire rod, it will be recalled that the quality of the wire rod is the result of the combination of various characteristcs of this wire rod, among which there may be mentioned, for example: the dimensions, and in particular the area of cross-section, of the filament; the purity of the steel, and in particular the presence of any possible inclusions; the mechanical properties of the wire rod, and in particular its tensile strength, which depends on the microstructure of the steel and the transverse and longitudinal homogeneity of this microstructure; and finally the superficial appearance of the wire rod, and in particular the possible degree of oxidation.

Among these conditions which determine the final quality of wire rod, the microstructure of the wire rod and the homogeneity of the microstructure, and the quantity and nature of the scale on the surface of the wire rod, depend almost exclusively on the treatment undergone by the wire rod in the last stages of the rolling operation and on the speed of cooling or the cooling regime to which the wire rod is subjected on issuing from the last roll stand.

As is well known moreover, up to about 10 years ago wire rod intended for wire drawing was almost always submitted to three treatments, that is to say: de-scaling, patenting (generally termed prepatenting in order to distinguish it from patenting during the wire drawing operation), and coating with lime. De-scaling is for the purpose of removing the scale from the wire rod after rolling or after patenting; the prepatenting is intended to provide the wire rod with an adequate structure prior to wire drawing, either to ensure adequate drawability or to ensure that the final product possesses satisfactory properties; the lime treatment finally is a surface treatment improving the adhesion of the lubricant utilized during wire drawing.

These operations, which are usually carried out during wire drawing, are all the more costly when applied to the production of small quantities of wire or rod, a fact which explains the desirability of carrying out all or a part of these operations at the exit of the rolling mill itself.

To this end different processes have already been proposed for the controlled cooling of the wire rod, generally carried out with the wire rod in the form of loose turns, the most interesting of which at present known only allow one to dispense with prepatenting (patenting before the first wire drawing operation) and in certain less frequent cases patenting in air before the last wire drawing operation. In a number of cases, prepatenting with lead is indispensable in order to ensure with certainty a suitable structure before or after wire drawing.

These processes, applied at the exit of the last roll stand, reduce the thickness of the scale formed on the wire without however eliminating the need for descaling during wire drawing. Finally, they ensure an acceptable longitudinal homogeneity in the wire, which is less homogeneous, however, than that which can be obtained by patenting and especially lead patenting.

Apart from these methods, several of which have been applied on the industrial scale, a technique has been proposed based on the cooling of wire rod by fluid bed techniques. The results obtainable with this new technique are satisfactory as far as concerns the structure of the product. These techniques, however, have disadvantages, among which might be mentioned the complexity of the technology and the high capital cost, as well as maintenance costs by reason of the movement of relatively abrasive materials.

What is desired is a process for the treatment of wire rod, applicable to wire rod leaving the last roll stand of the rolling mill, for the purpose of conferring on the wire rod properties, especially microstructure and a tensile strength, which are better suited to the requirements of users and in particular of wire drawing.

It would also be desirable for such a process to be applicable to the treatment of wire in wire drawing installations prior to or between the various wire drawing passes.

It is known in fact that at present it is impossible in practice to manufacture by hot rolling methods metal rod having a diameter less than 5mm. The manufacture of rod or wire having a smaller diameter requires one or more wire drawing operations in the course of which the microstructure of the rod or wire and also its mechanical properties are undergoing modification. This is the reason why in wire drawing installations one is compelled, in addition to any desired prepatenting operation, to carry out, depending on the final diameter of the rod and the properties or qualities thereof, one or more intermediate patenting operations which are intended to restore the microstructure of the rod. These pantenting operations prior to or during the course of drawing are carried out according to requirements by means of a heat treatment either in a lead bath or in a fused salt bath or simply in air. These different methods are however attended with well known drawbacks from the point of view of their being carried into effect.

The present invention provides a process for the treatment of metal rod or wire, comprising cooling the rod or wire from a first temperature at which the metal is in a stable phase to a second temperature at which the metal is in a second phase, the process including cooling the rod or wire by means of a liquid consisting of an aqueous fluid containing dispersed therethrough at least one substance which modifies the heat transfer conditons of the aqueous fluid, the heat transfer conditions of the liquid and the duration of cooling by means of the liquid being such that at least the major part of the optimum allotropic transformation of the metal takes place during cooling by means of the liquid, the liquid being at a temperature greater than 80° C and above 75 percent of its boiling temperature expressed in degrees centigrade.

In the case of steel the optimum transformation is the transformation which when terminated yields, in place

of the austenitic structure which is the initial phase, a structure which is practically free of ferrite on the one hand and of compounds resulting from quenching on the other hand such as bainite and/or martensite. Of course, the initial temperature of the steel must be in the austenite range. In most cases the temperature of the intiation of the optimum transformation of the steel corresponds substantially to the nose of the CCT or TTT curve.

The rod or wire which is subjected to the above process is initially at a temperature higher than the temperature of the initiation of the allotropic transformation; it may be that the treatment is applied to rod leaving a hot rolling mill while it is still at a high temperature, or starting with rod or wire at ambient temperature, that this rod or wire has been brought to a temperature higher than the temperature of the initiation of the allotropic transformation, by a known heating means, to produce the phase which is to be transformed.

According to an advantageous method of carrying out the process according to the invention, the liquid utilised is an aqueous saline solution, preferably saturated or supersaturated.

The aqueous saline solution is preferably brought to its boiling temperature in order to ensure optimum transfer of heat from the rod or wire and to ensure the maintenance of uniformity of the temperature and consequently of the conditions of treatment of the wire or rod. The aqueous solution can contain one or more other salts.

In a particularly advantageous embodiment of the invention, the liquid composition is selected such that its coefficient of thermal conductivity lies between 500 and 2,000 kcal/m²/hour/° C at a surface temperature of about 500° C of the wire or rod. For example, a solution containing (by weight) 80 percent $CaCl_2$ and 20 percent water at a temperature of 160° to 165° C has been successfully used.

The substance or substances in suspension and/or in solution in the aqueous fluid may include substances the presence of which increases the de-scaling of the wire or rod by the liquid or enables electrolytic descaling to be carried out in a bath of the liquid.

It is possible that the materials in suspension and/or in solution in the aqueous fluid which is substantially at boiling temperature ensure that there is a removal of heat from the wire or rod in such manner that the allotropic transformation is brought about at least to a partial extent in a substantially isothermal manner, owing to the well-known phenomenon of recalescence.

In accordance with another advantageous variation of the method, the nature of the substance or substances in suspension and/or solution in the aqueous liquid is such that the substance or at least one of the substances causes passivation of the rod or wire, for example by phosphatation.

In another embodiment of the process which forms the subject of the invention, which is particularly economical and very easy to carry out in the manufacture of wire rod, the substance or at least one of the substances in solution in the aqueous liquid is a surface-active substance. This surface-active substance may be of natural origin or obtained synthetically.

When using surface-active substances, the aqueous liquid is preferably used at boiling temperature.

Suitable surface-active substances are palmitates, stearates, or oleates, of sodium or potassium. Use may also be made of detergent substances of the sodium alkyl sulphonate type.

It is not going beyond the scope of the invention to use a mixture of several types of surface-active substances.

This embodiment of the process which is the subject of the invention has the advantage of not necessitating a subsequent rinsing treatment when the aqueous cooling fluid is comprised only of surface-active substances in solution; this constitutes a considerable economy in capital cost and operational cost. Furthermore, the quantities of substance to be used need only be very small — generally of the order of a thousandth in volume — and the cost of the substance is very low. Finally it should also be noted that the cooling conditions of the wire or rod in the aqueous liquid — especially the heat transfer — vary only slightly in the case of quite considerable modifications in the concentration of surface-active substances in the liquid; for instance, if the quantity of materials present in the aqueous liquid varies from two or three thousandths to 1 percent in volume.

Some particularly advantageous results have been obtained using as surface-active material a non-ionic surface-active substance obtained by the condensation of ethylene oxide with alkyl phenols. The rod obtained by this process has a quite remarkable microstructure which is substantially homogeneous both in the transversal and longitudinal direction.

In order to increase still further the efficiency of the process it has been found of advantage to add to the foregoing surface-active substances, in appropriate proportions, another substance or substances intended to facilitate heat transfer from the metal wire or rod to the liquid and more particularly to reach during the allotropic transformation a cooling rate bordering on that obtained in known treatments with molten salts. Among these substances there may be mentioned in particular iron sulphate and also copper sulphate either used individually or in a mixture.

Heat transfer from the rod or wire to the liquid containing surface-active substances, when the rod or wire is immersed in the liquid, can also be modified by setting up an electric field around the rod or wire, for example by applying a potential difference between the rod or wire and one or more electrodes in contact with the liquid. The advantage of this is that it permits easy regulation of the cooling capacity of the liquid, particularly by changing the sense or intensity of the electric field.

It is also possible, without departing from the scope of the present invention, to combine the actual advantages of the two main embodiments described above by cooling the wire or rod by an aqueous solution of a salt (such as $CaCl_2$) followed by water containing a surface-active substance (for example soap). The combined times of treatment of the wire or rod by the two liquids is substantially the same as that which would be necessary using one of the above-mentioned liquids alone. Moreover, apart from its specific action as concerns the properties and microstructure of the wire or rod, the aqueous fluid containing surface-active agents serves at the same time as a rinsing fluid after the first stage of treatment in the salt solution.

According to one mode of operation of the invention, in which the process is applied to steel wire rod issuing from the last stand of a hot rolling mill, the wire rod is

first subjected to cooling (in a conducting pipe for example) down to a skin temperature of 500° to 900° C, then to air cooling in order to bring about a given degree of homogenisation of temperature, and is then cooled by means of the liquid.

The first cooling to which the wire rod issuing from the rolling mill is subjected, may be rapid cooling with the aid of a fluid such as water and/or air, during which the austenitic grain size may be controlled. This control of the austenitic grain size can be made during the first stages of cooling.

In such cases, where recourse is had to a preliminary cooling of the wire or rod prior to submitting it to the action of the liquid, the preliminary cooling may preferably be conducted in such a manner that the optimum allotropic transformation of the metal cannot commence before the wire or rod has been submitted to the action of the liquid. The conditions of transport (speed, time, path length, etc.) of the wire or rod before it is submitted to the action of the liquid should therefore be determined in such a manner that the temperature conditions mentioned above in respect of the wire or rod are adhered to.

If necessary one may also use known means, such as radiation devices, in order to control the loss of heat from the wire or rod, when the latter leaves the conducting pipe, in such a manner as to adhere to the conditions set forth above in respect of the temperature of the wire or rod.

In one possible method of carrying out the process according to the invention the liquid is contained in a tank and the rod or wire is immersed in the liquid. Water is preferably supplied to the tank in proportion to the evaporation which takes place, this supply of water being controlled by means of an automatic system for detecting the temperature rise of the aqueous solution or suspension. The regulation of the temperature of the wire or rod is preferably such that the allotropic transformation is complete when the wire or rod leaves the liquid; the temperature of the wire or rod on leaving the liquid is preferably between 500° and 550° C in the case of steel, for example. It is also possible to allow the major part of the optimum transformation to occur in the liquid and to allow the transformation to be completed outside the liquid, for example by air cooling.

According to another mode of carrying out the invention the liquid may be sprayed on the wire or rod. The spraying of this liquid on the wire or rod can be carried out, depending on the conditions, for example through one or more concentric jets in succession or individually, or by combination of these means. In all cases the mutual direction of the jets may be different especially in view of achieving uniformity in the degree of cooling of the wire or rod.

It should be noted that the process according to the present invention may be carried out in at least two stages; these stages may, as required, constitute different operations, for example the allotropic transformation partially or totally during the course of one stage, the treatment in an aqueous de-scaling solution and/or the phosphatation of the wire or rod at least during another stage.

It has also been found advantageous to complete the surface treatment of the wire while accelerating the process of cooling after the completion of the allotropic transformation. For this purpose, on issuing from the aqueous fluid in which the controlled cooling is carried out, it is advantageous to subject the rod or wire after it has been rinsed to a treatment for the purpose of preventing surface oxidation. This treatment of the rod or wire may be carried out by immersion in a solution ensuring for instance the passivation of the rod or wire, notably phosphatation, or the coating of this rod or wire with a protective material, which may be a metal, for instance copper. In the particular case of steel rod or wire, it may be dipped in a solution of copper sulphate.

To accelerate the process of cooling the rod or wire on issuing from the bath in which the transformation of the metal of the rod or wire has taken place, the rinsing bath or at least one of the rinsing baths for the protection of the rod or wire may be at a temperature lower than 75 percent of its boiling temperature expressed in degrees centigrade.

The process forming the subject of the invention can be carried into effect no matter what may be the configuration of the wire or rod and especially in a particularly advantageous embodiment when the wire is looped in loose turns; the turns may for example be spread out non-concentrically on a conveyor, or may be wound around any axis of orientation, or may be moved along a given trajectory, the turns remaining or not remaining parallel to one another, as desired.

The process according to the invention enables the operations of patenting in lead or in fused salts, which are conventionally applied between the different stages of the wire drawing, to be replaced by a more simple treatment.

By virtue of the process described above, metal wire or rod may be manufactured the microstructure and the mechanical properties of which are comparable to those of rod or wire which has been subjected to treatment consisting of patenting in lead.

The present process is applicable to wire rod either after issuing from the last roll stand of the rolling mill (with or without guide tube), or prior to wire drawing; it is also applicable to wire which has been subjected to one or more passes through a wire drawing die.

Moreover the process is applicable to steel rod irrespective of its carbon content. Particularly advantageous results have, however, been obtained in the treatment of hard-steel rod, that is to say rod manufactured from a steel the carbon content of which lies between 0.4 and 0.85 percent.

The invention will be described further, by way of example only, with reference to the accompanying drawings, in which:

FIG. 1 is a micrograph of a steel wire rod in a region near the surface;

FIG. 2 is a micrograph of the core region of the same wire rod;

FIG.S 3 to 6 are micrographs of wire rod of another steel; and

FIG. 7 schematically shows apparatus for treating rod.

Looking at FIG. 7 first wire rod 5.5 mm in diameter of steel containing 0.8 percent carbon, issuing from the last roll stand 1 of the rolling mill at a speed of 50 m/s, passes through a cooling water pipe 9.5 m long 2, where its skin temperature falls from 850° to 450° C, whereas the temperature at the core falls from 1,010° to 975° C. The wire is looped at 3 in the open air in non-concentric overlapping turns on a conveyor which

passes through a tank 15 m long 4 containing an aqueous solution of $CaCl_2$ (300 g $CaCl_2$ per 100 g water) at a temperature of 160° C, the conveying speed being 1 m/s. On issuing from the tank the wire rod, which has a skin temperature of 525° C and a core temperature of 535° C, is immersed in a rinsing bath 5 which is 10 m long where it is cleaned and considerably cooled. After the turns have been tightened and aligned to form a coil at 6, the rod is subjected to suitable surface preparation treatment and is then immersed in a tank 7 containing zinc phosphate.

The wire rod obtained is very homogeneous, suitably descaled, and adequately protected against atmospheric oxidation. Its microstructure is substantially identical to that of wire rod patented with lead.

In another example, steel rod 5.5 mm in diameter, containing 0.61 percent carbon, issuing from the last roll stand of a rolling mill at a speed of 50 m/s, passes through a guide pipe where its skin temperature falls to 825° C, whereas the core temperature falls to 980° C. The wire is looped in loose turns in the air and is placed in non-concentric overlapping turns on a conveyor belt which passes through a tank 50 m long containing a saturated aqueous solution of NaCl and KCl at a temperature of 112° C, at a conveying speed of 1 m/s. On issuing from the tank the wire, which has a skin temperature of 500° C and a core temperature of 510° C, is passed through a rinsing tank 10 m long, where it is cleaned and considerably cooled. After tightening of the loose turns into a coil, the rod is subjected to suitable surface preparation treatment and is then immersed in a bath containing zinc phosphate.

The rod obtained has a mean tensile strength of 109.1 kg per mm^2 and a mean reduction in area of 47.1 percent; it is very homogeneous, and is adequately descaled and protected against atmospheric oxidation. Its microstructure is substantially identical to that of rod patented in lead as is shown by the micrographs, FIGS. 1 and 2 (magnified 500 times), of two test specimens one taken from near the skin and the other from the core of the wire.

Results are given below of tests carried out on hot rolled rod and on drawn wire, subjected on the one hand to patenting in lead in the conventional manner and on the other hand subjected to treatment according to the invention. These tests in confirmation of results previously obtained have been carried out by workers of the Societe Anonyme Cockerill.

EXAMPLE 1

An aluminium-killed LD steel was made containing as tapped: 0.80 to 0.85 percent carbon; 0.10 to 0.80 percent manganese; 0.15 to 0.25 percent silicon; less than 0.040 percent phosphorus; less than 0.020 percent sulphur; and less than 0.040 percent aluminium. Wire rod was made from this steel, and contained 0.845 percent carbon, 0.709 manganese, 0.183 percent silicon, 0.030 percent phosphorus, 0.015 percent sulphur and 0.030 percent aluminium. The wire rod leaving the last roll stand of the rolling mill with a diameter of 5.5 mm had a temperature between 800° and 1,050° C. It was made up into two reels each of 60 to 70 kgs which were subjected to a treatment according to the invention, in aqueous baths containing surface-active substances. Sixteen tensile tests were carried out on the first and last turns of each of these reels. Table I below gives the mean values of the ultimate tensile strength and of the reduction in area which have been obtained, and also the corresponding values of these parameters for a wire of the same batch subjected on the one hand to patenting in lead and on the other hand to air cooling in accordance with a modern industrial cooling procedure, on leaving the roll stand of the rolling mill.

TABLE I

	Ultimate tensile strength (kg./mm.²)			Reduction in area		
	First turn	Last turn	Mean	First turn	Last turn	Mean
First reel, treated in accordance with the invention	127	131.5	129.1	40.2	49.2	45
Second reel, treated in accordance with the invention	124.5	129	126.8	39	44.4	42.6
Rod patented by conventional lead treatment			133			40–45
Rod cooled in air			110–117			40–45

FIGS. 3 to 6 are micrographs of sections of rod taken respectively from the first and last turns of the first reel and from the first and last turns of the second reel. They show that the microstructure of the wire exhibits neither free ferrite nor free cementite, but that is is composed of very fine pearlite and sorbite, which ensure a very high strength.

EXAMPLE 2

A quantity of rod 5.5 mm in diameter was prepared from the same steel as in the preceding example, which was then made up into two reels of about 600 kgs and 3 reels of about 60 kgs. These reels were passed through a wire drawing mill where the large reels were subjected to patenting in lead in the conventional manner and the smaller reels were subjected to treatment in accordance with the invention in an aqueous bath containing surface-active substances. The reels were then butt welded and the resulting rod was then drawn down to a diameter of 2.40 mm by the conventional industrial procedure. Table II gives the extreme values and the mean value of the ultimate tensile strength R_r, the number of flexings or torsions leading to rupture and the reduction of cross-sectional area, of the drawn wire.

TABLE II

	Diameter d(mm.)	R_r (kg./mm.²)	Flexing ($r=5d$)	Torsion ($100 \times d$)	Reduction of area, percent
Patented in lead	2.41	231–235	6–7	26	41–47
		Vm=233	Vm=7		Vm=46
Do	2.42	215.6–217.8	7–8	25–28	46–51
		Vm=216.7	Vm=7	Vm=26	Vm=48
Treated in accordance with the invention	2.41	211.3–213.7	8	19–23	45–47
		Vm=213.5		Vm=20	Vm=46
Do	2.42	196.7–207.6	6–8	28–31	48–51
		Vm=200	Vm=6	Vm=29	Vm=50
Do	2.41	202.6–215.2	7–8	20–32	46–50
		Vm=207.4	Vm=8	Vm=29	Vm=49

NOTE.—Vm=Mean Value, r=radius of curvature.

I claim:

1. A process for the treatment of steel rod or wire, comprising cooling the rod or wire from a temperature in the austenitic range to a temperature below the austenitic range, the process comprising the steps of cooling the rod or wire by means of a solution of suspension in water of at least one alkali or alkaline earth inorganic salt which modifies the heat transfer conditions of the water, and the heat transfer conditions of the solution or suspended phase, the duration of cooling by means of the solution or suspension being such that at least the major part of the transformation of the steel from austenite to pearlite takes place during cooling by means of the solution or suspension, which is at a temperature higher than 80° C and above 75 percent of its boiling temperature expressed in degrees centigrade.

2. A process as claimed in claim 1, wherein the solution or suspension is boiling.

3. A process as claimed in claim 1 further comprising in connection with said salt a non-ionic surface-active agent obtained by condensation of ethylene oxide with an alkylphenol.

4. A process as claimed in claim 1, wherein the water contains at least one substance facilitating heat transfer from the rod or wire through the water.

5. A process as claimed in claim 1, wherein the water contains at least one substance which causes de-scaling of the rod or wire.

6. A process as claimed in claim 1, wherein the water contains at least one substance which causes passivation of the rod or wire.

7. A process as claimed in claim 1, wherein the step of cooling the rod or wire by means of a solution or suspension comprises the steps in sequence of cooling the rod or wire by means of an aqueous solution of at least one salt, and cooling the rod or wire by an aqueous fluid containing at least one surface active agent.

8. A process as claimed in claim 1, wherein the rod or wire is steel rod leaving the last stand of a hot-rolling mill, the process further comprising, prior to cooling by mans of the solution or suspension, the steps of cooling the rod to a skin temperature of 500° to 900° C, and air cooling the rod, whereby the temperature of the rod is homogenized.

9. A process as claimed in claim 8, wherein the temperature of the rod immediately after cooling by means of the solution or suspension, is between 500° and 550° C.

10. A process as claimed in claim 1, wherein the step of cooling by means of the solution or suspension includes immersing the rod or wire in the solution or suspension.

11. A process as claimed in claim 10, including the step of setting up an electric field around the rod or wire to control the cooling capacity of the solution or suspension.

12. A process as claimed in claim 10, wherein the water contains at least one substance permitting electrolytic de-scaling of the rod or wire.

13. A process as claimed in claim 1, wherein the step of cooling by means of the solution or suspension includes spraying it on to the rod or wire.

14. A process as claimed in claim 1, further comprising treating the rod or wire to inhibit surface oxidation.

15. A process as claimed in claim 1, wherein the rod or wire is in loose turns throughout the step of cooling by means of the solution or suspension.

16. The process of claim 1 wherein the salt is calcium chloride.

17. The process of claim 16 wherein the solution or suspension contains 80 weight percent of calcium chloride and 20 weight percent of water at a temperature of 160° to 165° C.

18. The process of claim 8 wherein the solution or suspension contains 80 weight percent of calcium chloride and 20 weight percent of water at a temperature of 160° to 165° C.

19. A process as claimed in claim 1 wherein the solution or suspension is a saturated aqueous solution of NaCl and KCl at a temperature of about 112°C.

* * * * *

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,785,878 Dated January 15, 1974

Inventor(s) Mario Economopoulos

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Insert Figures 1 through 6, as part of Latters Patent.

Signed and sealed this 8th day of October 1974.

(SEAL)
Attest:

McCOY M. GIBSON JR.
Attesting Officer

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SHEET 1 OF 4

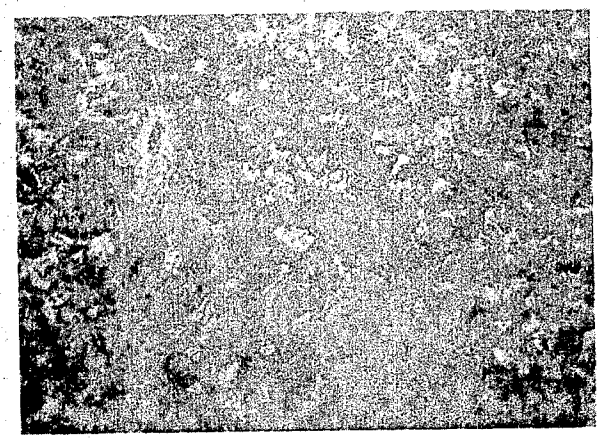


FIG. 1

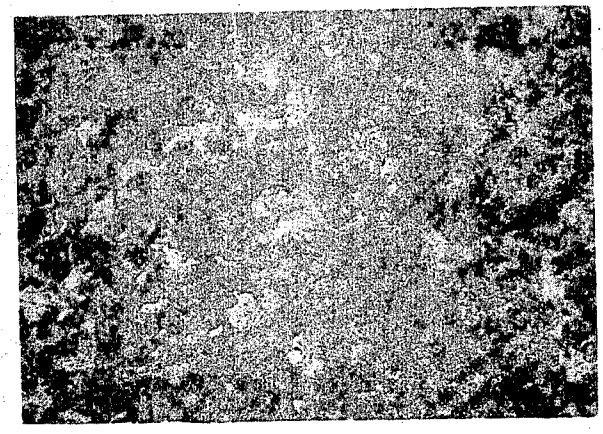


FIG. 2

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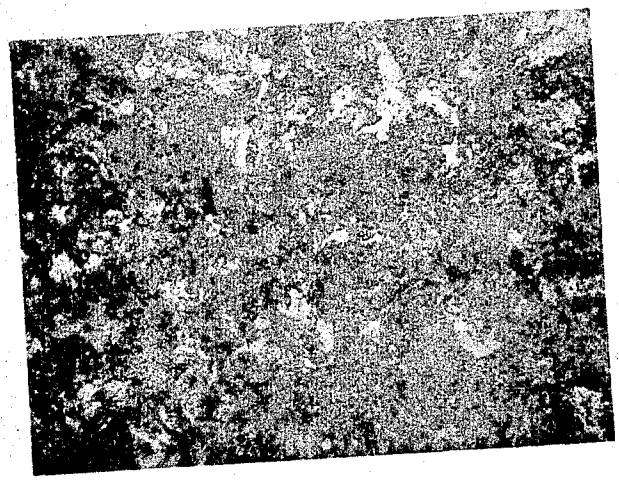


FIG.3

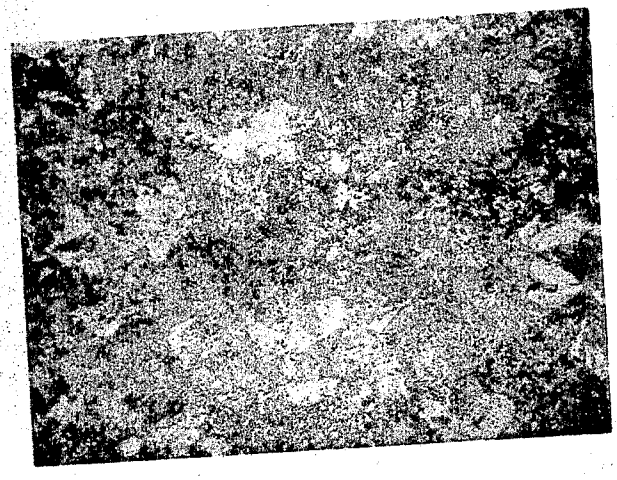


FIG.4