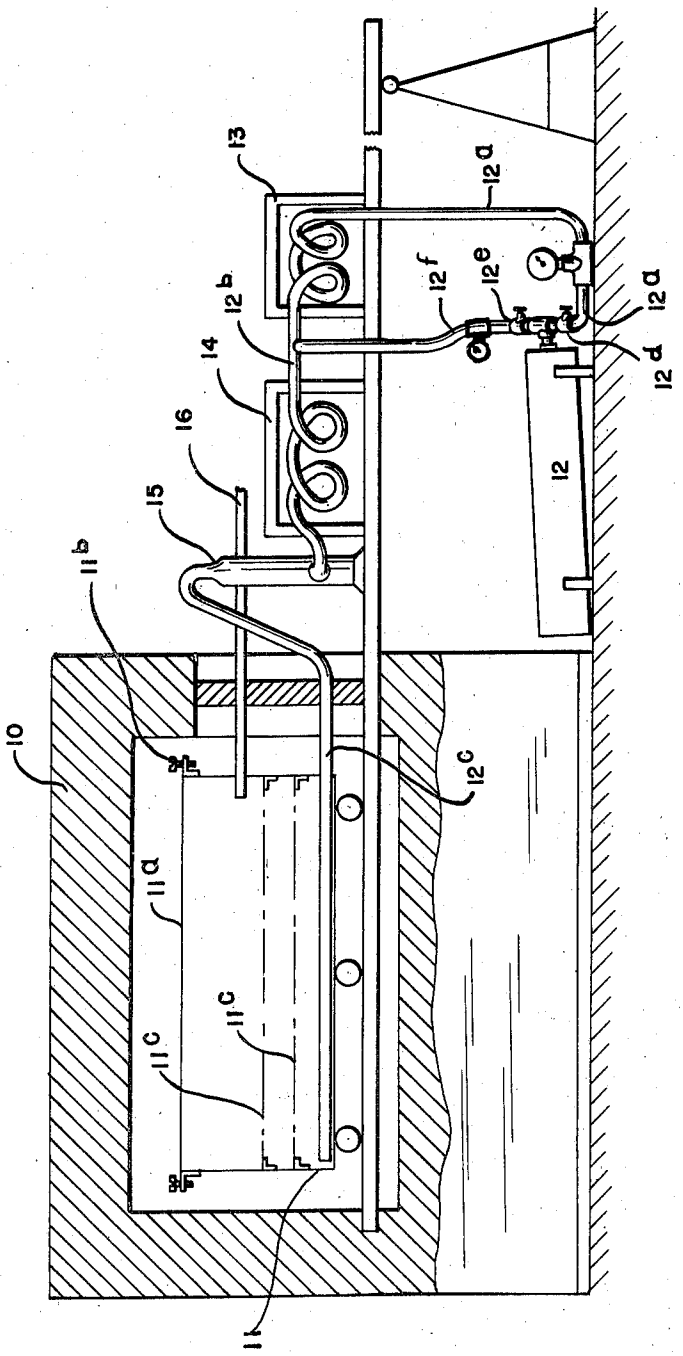
Nov. 2, 1948. A. L. FEILD 2,452,915

NITRIDING PROCESS

Filed June 27, 1947



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Patented Nov. 2, 1948

2,452,915

UNITED STATES PATENT OFFICE

2,452,915

NITRIDING PROCESS

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Application June 27, 1947, Serial No. 757,426

4 Claims. (Cl. 148—16.6)

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This application is a continuation-in-part of my copending application, Serial No. 550,644, filed August 22, 1944, now abandoned, and entitled Nitriding process and the invention relates to a hardening treatment for alloy steel, and more particularly to a method of nitriding stainless steel.

An object of my invention is the provision of a method of nitriding alloy steel, especially stainless steel, which as a procedure is characterized by directness and simplicity, and which ensures the obtainment of uniformly nitrided steel surfaces.

A further object is the provision of a thoroughly practical and highly effective method of rapidly and efficiently case-hardening stainless steel in a nitrogenous atmosphere and at the same time removing oxide film from the metal directly as a part of the nitriding procedure.

A still further object of my invention is the provision of durable, wear-resistant case-hardened stainless steel products which individually possess high surface hardness, which hardness moreover is uniform along the surface.

Other objects of my invention in part will be obvious and in part will be pointed out hereinafter.

The invention accordingly consists in the several operational steps, and in the relation of each of the same to one or more of the others as described herein, the scope of the application of which is indicated in the following claims.

In the single figure of the accompanying drawing there is schematically shown apparatus illustratively employed in the practice of my invention.

As conducive to a clearer understanding of certain features of my invention it may be noted at this point that stainless steel is defined as a low-carbon alloy metal which comprises from about 10% to approximately 35% chromium, with or without nickel, and with or without supplemental additions of manganese, silicon, cobalt, copper, molybdenum, tungsten, vanadium, columbium, titanium, sulphur, and the like, for special purposes, and a balance which is substantially all iron.

There are methods of nitriding stainless steel for the purpose of case-hardening the same which form a part of commercial hardening practices. The heretofore known methods have been employed seeking hard, wear-resistant surfaces to extend the range of utility of the stainless steels and to render the steels more satisfactory as in

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uses previously established for a softer quality of corrosion-resistant surface.

An impediment to the formation of nitrided layers in stainless steel arises due to a characteristic quality of surface oxide film which has a tendency to insulate the metal from the effects of conventional nitriding treatment. It has been usual practice to case-harden the stainless steels by nitriding heat-treatment as in a current of ammonia gas of commercial grade. To ensure satisfactory formation of nitrides in this practice, however, it is necessary first to clean up the metal as by sand-blasting to remove the oxide film. Unless the initial cleaning procedure is followed with reasonable promptness by the nitriding operation and oxide film forms anew on the metal.

At times the very shape or size of a stainless steel object makes it difficult or impossible to subject the metal with satisfaction to the essential cleaning operation. Where sand-blasting is employed, a certain roughness which as such is frequently undesirable, is imparted to the metal. This remains to spoil appearance or even limit the utility of the finished hardened product. The sand-blasting or like treatment when brought to bear upon small or delicate objects, such as instrument dials and pointers, often results in deformation and distortion.

Even with this cleaning discoloration during the nitriding operation occasionally is encountered, this I attribute not to the fact that previous nitriding gases are inadequately dried, but that some residual oxygen must be present with the ammonia which combines with hydrogen subsequently available to form water when the gas passes over the hot steel being nitrided. This water causes discoloration in the prolonged heating.

A further and quite important objection to conventional nitriding procedures resides in the frequent obtainment of nitrided surfaces which lack uniformity of hardness and wear-resistance. This non-uniformity apparently is due to inefficient removal of passive oxide film initially or it is due to reformation of oxide after the cleaning operation and prior to nitriding.

An outstanding object of my invention, accordingly, is the provision of an economical and industrially practical process of nitriding stainless steel products wherein cleaning and surface hardening are achieved in a thoroughly satisfactory manner with a minimum of operational steps.

Referring now more particularly to the practice of my invention, I nitride stainless steel, more particularly stainless steel products as for

example tools, cutlery, machine parts, dental and surgical instruments, drafting equipment, valve elements including balls and seats, needles and other pointed objects, plumbing fixtures, trim, or ornamental pieces and the like by subjecting the same at nitriding temperature to a nitrogenous atmosphere which as initially introduced to the steel comprises an abundance both of ammonia gas and nascent hydrogen with or without nascent nitrogen. This atmosphere contains from about 15% to 85% ammonia by volume and the remainder hydrogen with or without nitrogen, the hydrogen amounting to at least 11% by volume. And, in general, I prefer to first establish the ammonia-hydrogen atmosphere and then introduce the steel to be nitrided into this atmosphere. The nitriding treatment by way of this mixed atmosphere I find serves effectively to remove the oxide film from the metal and ensures the formation of a uniformly hard nitrided case which is resistant to wear and corrosion.

A nitrogenous atmosphere of the character described into which I prefer to introduce the stainless steel comprises such substantial amounts of composed ammonia gas, at least 15% by volume, and the gaseous products of cracked ammonia as to make available promptly an abundance of free hydrogen, at least about 11% by volume, in the vicinity of the steel for oxide film removal and nitriding purposes. I find that it is the cracked ammonia which effects film removal and that it is the composed ammonia which largely is responsible for the nitriding.

As illustrative of the practice of my invention, I nitride a batch of stainless steel instrument pointers in a suitable electric, gas or oil fired batch-type metallurgical furnace **10** (see the accompanying drawing) in the heating chamber of which is housed a nitriding box **11** made as of 12% chromium–80% nickel alloy metal and having a lid or removable top *11a* sealed in substantially gas-tight relation to the box sidewalls as with an asbestos gasket *11b* covered with aluminum foil. The instrument pointers are supported for treatment in the sealed nitriding box, preferably on horizontal screens *11c* of nickel or nickel alloy metal, and along with the box are furnace heated at a holding temperature suitable for nitriding which I find to be in the range of 1100° F. to 1400° F. To remove oxide film from the heating knife blades and for case-hardening these stainless steel products, I supply to the nitriding box a gaseous mixture in a current, which as initially introduced contains an abundance both of uncracked ammonia and the gaseous products of cracked ammonia, for example, about 70% ammonia by volume and 30% nitrogen and hydrogen taken together, the hydrogen amounting to about 22.5%.

For continuously supplying the treating gas to the nitriding box I prefer to employ a drum or tank **12** of commercial anhydrous ammonia from which gaseous ammonia is conducted as under low positive pressure as controlled by valve *12d* through tube *12a* to a cracking unit **13**. A tube *12b* leads the gas from the cracking unit to a suitable preheating furnace **14**. A tube *12f* and associated valve *12e* bleeds into tube *12b* from tube **12** a controlled quantity of uncracked ammonia gas. At this point the gas is composed of a mixture of cracked and uncracked ammonia. Within the heating furnace I find that any small amount of oxygen absorbed with the uncracked ammonia is given the opportunity of combining with the hydrogen present. The resulting moisture then is removed in a drying operation as by passing the cracked and uncracked ammonia mixture through the drying tower **15** containing lime for example. Following this the dried cracked and uncracked ammonia mixture is conducted to within nitriding box **11** by means of tube *12c* which like all other tubes of the supply system preferably is of nickel. The gas circulates beneath and up over the stainless steel articles on supporting screens *11c*, and thence from the nitriding box through gas outlet tube **16** to suitable reclamation or disposal means (not shown). A control is maintained over the gas composition and flow through the system just described as by means of pressure regulating valves *12d* and *12e* on the ammonia supply tank.

As treatment of the instrument pointers continues at nitriding temperature in the presence of the nitrogenous gas, which as initially introduced contains both cracked and uncracked ammonia in substantial amounts, this being from about 15%-85% uncracked ammonia and the remainder cracked ammonia, the oxide film is effectively removed from the surface of the articles and a hard nitrided case begins to form. Over an extended period of continued treatment the nitrided layer gradually increases to a useful thickness; usually a treatment period ranging from approximately 30 hours to 90 hours gives a thickness which is satisfactory for most articles, though I do not wish to be bound by any such period.

Following the nitriding treatment, I shut off the furnace and the source of ammonia supply, and subsequently remove the nitrided stainless steel articles from the nitriding chamber. They are stored for future use or assembled and put into service as desired, either with or without further treatment such as polishing.

My method of nitriding stainless steel articles is successfully practiced on articles and products of widely differing quality, whether the products be of a form which is rugged or delicate. Due to the combined cleaning effect and strong nitriding action which prevail in the treating operation, the removal of surface oxide film from the metal is ensured and uniformly hardened surfaces are obtained on the products treated.

In the practice of my method it is possible to largely avoid preliminary treatments such as sand-blasting to remove passive oxide film, or other separate cleaning steps for the same purpose, and to avoid the many objections which frequently accompany the employment of the separate cleaning operations. Savings in time are directly effected. The products hardened as a result of my treatment are substantially free of soft spots at the surface, are uniformly resistant to corrosion, abrasion and wear, and uniformly retain surface hardness at high temperatures. In cross-section the products have a nitrided case in which the nitrides are in a fine state of dispersion and impart an extreme hardness at the metal surface. The hardness gradually decreases in intensity inwardly from the surface and at innermost points corresponds to that of the product core.

The method of nitriding which I provide is economical and requires operational steps which are easy to perform and conditions of operation which are simple to control in the mass production of nitrided stainless steel articles of outstanding quality. The equipment and materials

needed are readily available and require little space for giving effective treatment.

Thus it will be seen that there is provided in this invention a method of case-hardening stainless steels in which the various objects hereinbefore noted together with many thoroughly practical advantages are successfully achieved. It will be noted that the method is particularly suited for rendering the stainless steels clean and responsive to nitriding action in the same atmosphere which is employed for nitriding, and that the resulting nitrided products are of outstanding quality. It will also be noted that the method cleans up the steels without necessary use of harsh abradents and thus is highly satisfactory for producing nitrided articles, even those which are delicate in construction, with a minimum of spoilage.

As many possible embodiments may be made of my invention and as many changes may be made in the embodiments hereinbefore set forth, it is to be understood that all matter described or illustrated herein is to be interpreted as illustrative and not as a limitation.

I claim:

1. In a method of nitriding stainless steel, the art which includes cracking ammonia gas, mixing uncracked ammonia gas therewith to give a mixture consisting of about 15% to 85% by volume of cracked ammonia and about 85% to 15% by volume of uncracked ammonia, heating the mixture of cracked ammonia and uncracked ammonia so as to form moisture from any available oxygen present with the ammonia, removing this moisture by a drying operation, then passing the preheated and predried mixture of gases over the stainless steel while held at nitriding temperature for some 30 to 90 hours to nitride the same.

2. In a method of nitriding stainless steel, the art which includes, mixing cracked ammonia gas with uncracked ammonia gas to form a mixture consisting by volume of about 70% uncracked ammonia and about 30% cracked ammonia with the hydrogen content of the cracked ammonia amounting to about 22.5%, heating the mixture so as to form moisture from any available oxygen present with the ammonia, drying the gas mixture, and then passing the heated and dried mixture over the stainless steel to be nitrided for some 30 hours or more.

3. In a method of nitriding stainless steel, the art which includes heating a gas mixture consisting of about 15% to 85% by volume uncracked ammonia and the remainder cracked ammonia, so as to form moisture from any oxygen present with the uncracked ammonia, drying such mixture, introducing to such gas mixture said stainless steel, and heating the same to nitriding temperature for a sufficient length of time to remove passive oxide film from the metal surface and nitride the steel.

4. In a method of nitriding stainless steel, the art which includes heating a gas mixture consisting of about 15% to 85% by volume of cracked ammonia and 85% to 15% by volume of uncracked ammonia so as to form moisture from any available oxygen present, removing this moisture by a drying operation, then passing the preheated and predried mixture of gases consisting of cracked ammonia and uncracked ammonia over the stainless steel while held at a temperature range sufficiently high and for a period of time sufficiently long for nitriding the same.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,300,274	Dahl	Oct. 27, 1942