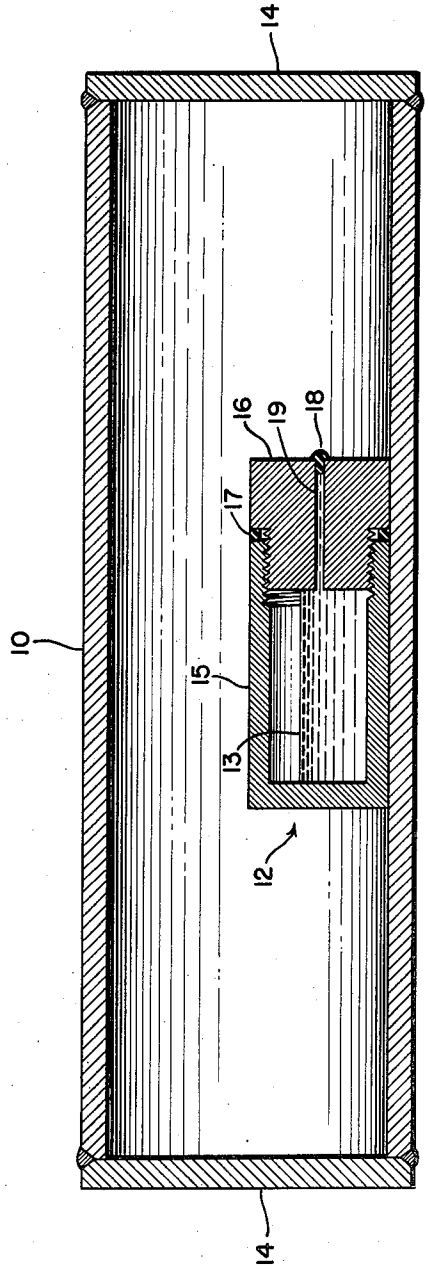
May 20, 1952 R. L. CHENAULT ET AL 2,596,981

METHOD FOR NITRIDING METALLIC SURFACES

Filed Oct. 5, 1949



INVENTORS
ROY L. CHENAULT &
BY GERALD E. MOHNKERN
Donald G. Dalton
ATTORNEY

Patented May 20, 1952 2,596,981

UNITED STATES PATENT OFFICE

2,596,981

METHOD FOR NITRIDING METALLIC SURFACES

Roy L. Chenault and Gerald E. Mohnkern, Oil City, Pa., assignors to United States Steel Company, a corporation of New Jersey

Application October 5, 1949, Serial No. 119,728

8 Claims. (Cl. 148—16.6)

1

This invention relates to methods for nitriding metallic surfaces, particularly interior surfaces of steel tubing.

Nitriding is a common method of case hardening steel surfaces. Previous nitriding methods with which we are familiar involve heating the steel to approximately 800° to 1200° F. in a special furnace which is sealed to exclude the atmosphere. Ammonia gas is circulated through the furnace at about atmospheric pressure or slightly above to assist in excluding air. Ammonia dissociates at this temperature into nascent nitrogen and hydrogen. Nascent nitrogen combines with the steel surface to produce the desired nitriding effect, but unless it combines immediately, it forms molecular nitrogen which is inert. Such methods have the disadvantages that they require special furnace equipment, they are undesirably slow and they are wasteful of ammonia. Furthermore the sealed construction of the furnace makes it difficult to rotate the steel articles, and thus in the case of long slender articles there is likelihood of warpage.

An object of the present invention is to provide improved nitriding methods which are faster and which conserve ammonia over previous methods with which we are familiar, and which can be performed in ordinary heating furnaces.

A further object of the invention is to provide improved methods of nitriding interior surfaces of steel tubing in which a liquid ammonia capsule is sealed in the tubing and the tubing is heated to nitriding temperatures, thereby releasing the ammonia under high pressure in the tubing.

In accomplishing these and other objects of the invention, we have provided improved details of structure, a preferred form of which is shown in the accompanying drawing, in which:

The single figure is a vertical sectional view of a metallic tubing containing an ammonia capsule for nitriding in accordance with the present invention. The capsule is shown of greatly exaggerated size compared with the tubing to furnish a better showing of its construction.

In the drawing there is shown a length of steel tubing 10, the interior surface of which is to be nitrided. In accordance with the present invention a capsule 12, which contains a body of liquid ammonia 13, is sealed within the tubing. Preferably the sealing is accomplished by welding steel plates 14 to the ends of the tubing.

Capsule 12 is of such construction that it releases its contents when it is heated. The capsule illustrated comprises a cylindrical body 15, a cap 16 which is threadedly engaged in the open

2

end of said body, and a gasket 17 which seals the cap to the body. Cap 16 has a longitudinal passage 19, the end of which has a plug 18 of material which melts at a temperature lower than the nitriding temperature, for example solder. However it is apparent that other capsule constructions could be substituted which would furnish the same effect. For example, the capsule could have a diaphragm that bursts under the pressure which generates when liquid ammonia is heated and vaporizes. Therefore, I do not wish to limit the present invention to any particular construction of capsule, as long as it is capable of confining liquid ammonia at ordinary temperatures, but releases its contents when it is heated to somewhat below nitriding temperatures.

After the capsule has been inserted in the tubing and the ends of the tubing have been sealed, the tubing is placed in any suitable heating furnace and is heated to a nitriding temperature, which is approximately 800° to 1200° F. The tubing can be supported on level bearings in the furnace and continuously rotated to prevent its warping. The capsule releases the ammonia at a temperature somewhat lower than the nitriding temperature. When the tubing reaches its nitriding temperature, the pressure within the tubing is of the order of 100 atmospheres, but varies somewhat with the volume of the tubing, the initial quantity of liquid ammonia in the capsule, and the exact temperature. The maximum pressure is limited only by the tensile strength of the tubing and the welds, but we find 100 atmospheres is a desirable operating maximum. Ammonia dissociates at the temperature to which the tubing is heated and thus furnishes nascent nitrogen for nitriding the interior surface of the tubing, and as usual the excess nitrogen which does not combine immediately with the tubing surface forms molecular nitrogen.

At the pressures and temperatures encountered within the tubing, the dissociation reaction of ammonia is reversible, thus:

$$2NH_3 \rightleftarrows N_2 + 3H_2$$

This reversibility enables molecular nitrogen to recombine with molecular hydrogen to replenish continuously the ammonia supply, the dissociation of which again produces active nascent nitrogen. This procedure can continue until practically all of the nitrogen which was originally present in the liquid ammonia has combined with the steel. The time required varies with the ammonia density, or pressure, and with the temperature. For example, with the temperature at approximately 1000° F. we have obtained the following results:

Pressure (p. s. i.)	Time (hrs.)	Depth of Nitriding (inches)	Rate of Nitriding (thousandths inch per hr.)
343	15	.004	.27
652	15	.009	.60
1,176	14½	.012	.83

Ordinarily removal of the air from the tubing is unnecessary since the hydrogen released by the ammonia dissociation effectively scavenges the oxygen, and the nitrogen merely contributes to the nitrogen supply. Also the hydrogen furnishes a reducing atmosphere that prevents oxidation of the tubing. Nevertheless, when desired, the air can be flushed from the tubing, preferably with nitrogen gas.

The initial quantity of liquid ammonia varies with the total interior surface of the tubing and the degree of nitriding which is desired. We have found that approximately 5 to 12 grams of ammonia per square foot of tubing surface furnishes satisfactory nitriding, with the lower regions of this range of course furnishing lesser degrees of nitriding.

We have described the nitriding method of the present invention as applied to interior surfaces of tubing. This method is particularly suited to nitriding tubing which forms such articles as subsurface pump barrels, the interiors of which customarily are case hardened to furnish wear resistance. Nevertheless it is apparent that the same method can be employed for nitriding metallic articles of other shapes. The only variation necessary is to seal such articles in a confined space which also contains the ammonia capsule. The articles then are heated the same as already described.

While we have described certain preferred procedures for practicing the present invention, it is apparent modifications may arise. Therefore we do not wish to be limited by the disclosure, but only by the scope of the appended claims.

We claim:

1. A method of nitriding surfaces of metallic articles which comprises confining the surface of a metallic article and a capsule containing liquid ammonia in approximately the amount of 5 to 12 grams per square foot of said surface in a sealed space, and heating said surface and said capsule to a temperature approximately between 800° and 1200° F. and thereby releasing ammonia from the capsule as a vapor, but confining this vapor within said sealed space.

2. A method of nitriding interior surfaces of steel tubing which comprises placing in the tubing a capsule of liquid ammonia, sealing the ends of said tubing, and heating said tubing with said capsule therein to a temperature approximately between 800° and 1200° F., thereby releasing the ammonia from said capsule as a vapor which is confined to the interior of the sealed tubing.

3. A method of nitriding interior surfaces of steel tubing for a considerable case depth which comprises placing in the tubing a capsule containing liquid ammonia in approximately the amount 5 to 12 grams per square foot of interior tubing surface, sealing the ends of said tubing, and heating said tubing with said capsule therein and thereby releasing the ammonia from said capsule as a vapor which is confined to the interior of said tubing, and continuing the heating until the tubing reaches a temperature approximately between 800° and 1200° F. for approximately 15 hours.

4. A method of nitriding interior surfaces of steel tubing for a considerable case depth which comprises placing in the tubing a capsule containing liquid ammonia in approximately the amount 5 to 12 grams per square foot of interior tubing surface, welding plates over the ends of the tubing and thereby sealing these ends, and heating said tubing with said capsule therein and thereby releasing the ammonia from said capsule as a vapor which is confined to the interior of said tubing, and continuing the heating until the tubing reaches a temperature approximately between 800° and 1200° F. for approximately 15 hours.

5. A method of nitriding interior surfaces of steel tubing for a considerable case depth which comprises placing in the tubing a capsule containing liquid ammonia in approximately the amount 5 to 12 grams per square foot of interior tubing surface, welding plates over the ends of the tubing and thereby sealing these ends, and heating said tubing with said capsule therein and thereby releasing the ammonia from said capsule as a vapor which is confined to the interior of said tubing and continuing the heating until the tubing reaches a temperature approximately between 800° and 1200° F. for approximately 15 hours, and continuously rotating said tubing while it is at said temperature.

6. A method of nitriding metallic surfaces comprising contacting the surface with ammonia gas at a temperature approximately between 800° and 1200° F., at which the gas dissociates to nascent nitrogen and hydrogen, while confining said gas under a pressure of the order of 100 atmospheres against the surface in order that the unreacted nitrogen and hydrogen can recombine readily.

7. A method of nitriding a surface of a steel body comprising confining said surface within a sealed space, introducing an ammonia charge to said sealed space in an amount equivalent to 5 to 12 grams of ammonia per square foot of said surface, and heating said surface to a temperature approximately between 800° and 1200° F. while preventing escape of gases from said sealed space and utilizing only the original ammonia charge as a source of nitrogen.

8. A method of nitriding a metallic surface comprising confining said surface within a sealed space, introducing ammonia in an amount equivalent to at least 5 grams per square foot of said surface and heating said surface to a temperature approximately between 800° and 1200° F. while preventing escape of ammonia from said sealed space.

ROY L. CHENAULT.
GERALD E. MOHNKERN.

REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
2,039,487	Lindemuth	May 5, 1936
2,395,329	Holden	Feb. 19, 1946

FOREIGN PATENTS

Number	Country	Date
314,219	Great Britain	Jan. 27, 1929
345,233	Great Britain	Mar. 16, 1931

OTHER REFERENCES

"Transactions of the American Society for Steel Treating," volume 15, pages 544, 545, 1929.