

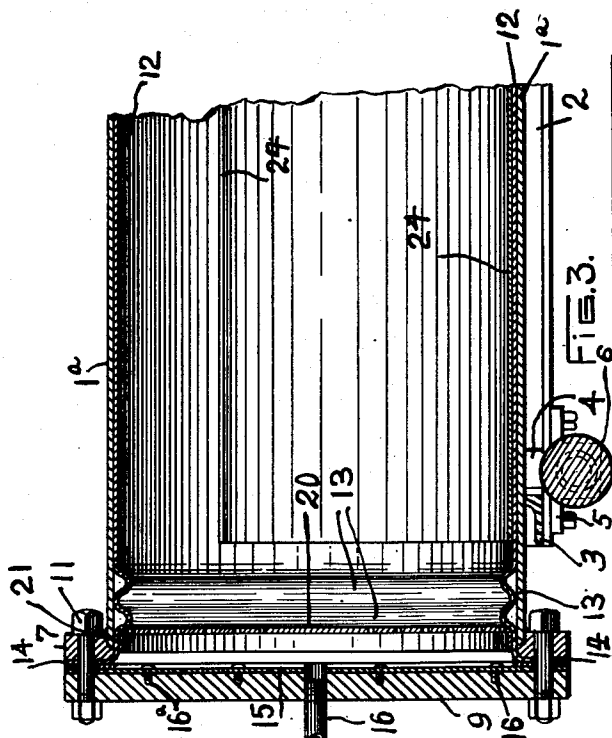
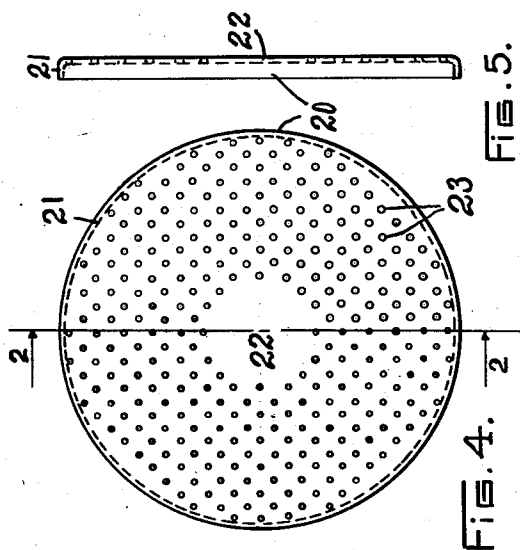
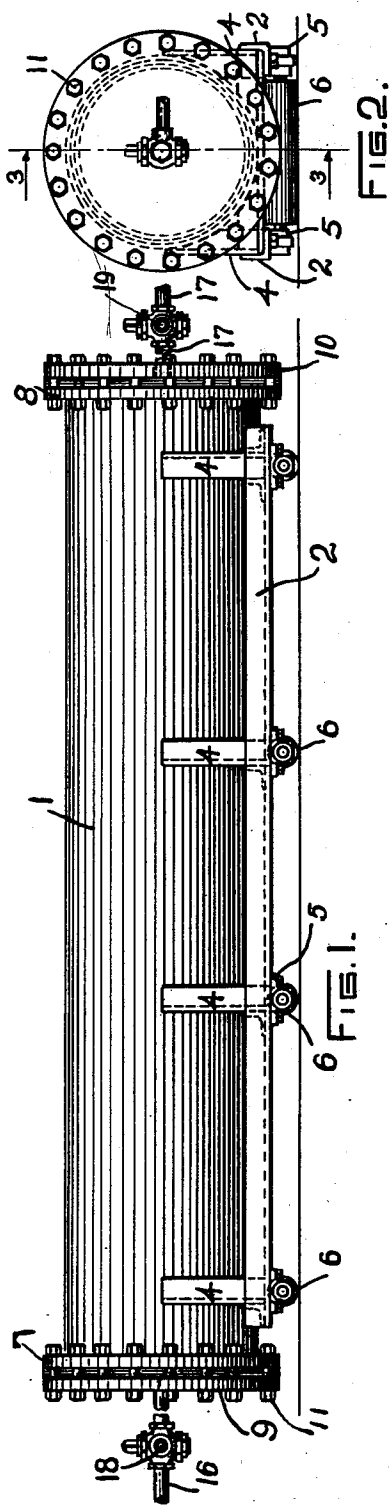
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N. M. SALKOVER

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NITRIDING APPARATUS

Filed March 11, 1943



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NITRIDING APPARATUS

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The operation of nitriding to produce a case-hardening, and which in general consists in subjecting steel parts, (usually machined and heat treated articles) to the action of a nitrogenous medium at an elevated temperature is well known. My invention relates to apparatus therefor.

A principal object of my invention is the provision of an environment and procedural steps whereby not only can a more uniform nitrogenous atmosphere be maintained about all of the articles being treated in a given batch, but also whereby a more accurate control of the nature and composition of the atmosphere itself is rendered possible.

Another principal object of my invention in the apparatus aspect is the provision of an improved form of container in which the articles are held during nitriding, the construction and advantages of which will hereinafter be more fully set forth.

These and other objects of my invention, which will be mentioned in these specifications or will be apparent to one skilled in the art upon reading them, I accomplish by the use of apparatus which I shall now describe exemplary embodiments. Reference is made to the accompanying drawing wherein:

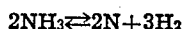
Figure 1 is a side elevation of my improved container.

Figure 2 is an end elevation thereof.

Figure 3 is a partial sectional view on an enlarged scale of the container.

Figures 4 and 5 are respectively elevational and sectional views of a perforated gas flow control plate.

The operation of nitriding generally comprises subjecting the articles to be hardened to the action of ammonia gas for specific periods of time while the articles are maintained at temperatures ranging from about 900° F. to 1200° F. The articles are usually placed in a container to which gas connections are made, and this container in turn is placed in an insulated muffle or furnace in which the required temperatures can be maintained. Sometimes the furnace or furnace lining itself is the container. During the treatment the ammonia decomposes in some measure into nitrogen and hydrogen according to the following reaction:



At the moment of dissociation, the nitrogen is in a nascent condition and combines with the steel or with alloy elements in it to form nitrides. These nitrides, which are in a fine state of dispersion in the outer layer or layers of the article, impart extreme hardness to the surface of the

steel, forming a "case" thereon. If such a treated article is examined, the case will be found to have a hardness which gradually decreases inwardly until it corresponds to the hardness of the untreated portion or "core" of the article.

In the operation of nitriding, a control of the degree of ammonia dissociation has been understood to be vital. The gas mixture leaving the furnace consists of hydrogen, nitrogen, and undissociated ammonia. It is usual to run frequent tests on the issuing gas in order to determine the degree of association. This may be readily done in known ways. If the percentage of dissociation is found to be too great, the flow of ammonia introduced into the container is increased, while if too little dissociation is found to occur, the flow of ammonia is decreased. It is usually considered necessary to control the ammonia flow so that the percentage of dissociation, particularly for a number of hours after the start of the heat, will not exceed 35%. In some operations, a percentage of dissociation up to 50% or higher can be tolerated without injurious effects, according to some workers. The exact percentages of dissociation to be sought for by the particular worker in the use of my apparatus are not themselves limitations upon my invention. The important point is the necessity of controlling the degree of dissociation throughout the heat and maintaining it at a level which experience has demonstrated produces the results desired.

The available control for the degree of association has hitherto been found only in a control of the rate of introduction of the ammonia into the container. One of the most difficult problems involved in the nitriding operation has in the past been the design and construction of efficient containers. It is desirable to obtain a constant flow of the ammonia and its decomposition products through the container and in contact with all of the articles within the container. Dead pockets, through which the gases do not circulate constantly and uniformly, result in improperly nitrided work. Containers in the past have been so designed that in practice an efficient circulation of gas about all of the articles being treated could frequently not be obtained unless the rate of gas introduction was maintained above a certain level; yet, this is obviously inconsistent with proper control of the degree of dissociation, since such control involves variation of the rate of flow.

Furthermore, the material of which the container is made has a most profound effect upon the general efficiency of the nitriding operation. As will presently be explained, the containers

themselves, after a period of use, acquire the property of promoting the dissociation of ammonia. This further requires control of ammonia flow substantially without regard to the necessity of bathing each individual article in a suitable circulating supply of the gas.

The original and commonest material for the containers was ordinary carbon steel. Satisfactory results were obtained for one or two heats. Thereafter the container, which had itself been nitrided by the nascent nitrogen to which it was exposed, not only became extremely brittle and liable to failure or accident, but also acquired the property of exerting a catalyzing effect on the ammonia. This made it extremely difficult, if not impossible, to secure satisfactory nitriding results.

The difficulty is not confined to steel. All metals, which are capable of forming nitrides readily when subjected to the action of nascent nitrogen after they are once nitrided, become catalysts, effectively increasing the dissociation of ammonia during future nitriding operations. Attempts have been made in the past to use containers of stainless steel, pure nickel, alloys high in nickel, copper, and other materials; but it has been found necessary after a relatively brief period of service to discard such containers. The extra cost of them has not been justified by the results obtained. Vitreous enameled steel has been suggested and used, but has not proved a solution for the problem. While the vitreous enamel itself is inert, containers made with vitreous enamel coatings, when subjected to repeated heatings and coolings, develop innumerable minute cracks which permit the penetration of the ammonia to the underlying metal surface, and thus defeat the purpose of enamel coating. Moreover, such coatings are delicate in their nature and are subject to cracking and spalling not alone from repeated temperature extremes, but also from mechanical injury.

I have found, however, that these difficulties can be overcome through the use of a material and a construction of which I am about to describe an exemplary embodiment. I have found that aluminum is not affected by the nascent nitrogen or the decomposition products of ammonia, under nitriding conditions. By "aluminum," I mean to include not only pure aluminum, but the range of commercial aluminums including those which contain relatively small percentages of alloying elements. My researches have indicated that so long as the percentage of alloying elements in the aluminum is small, say, not greater than say two or three percent, the alloy is suitable for my purpose. By way of example, aluminum containing silicon, manganese, magnesium, or combination of these or other alloying elements within the percentage set forth, is not affected by the atmosphere during nitriding.

The distinctive feature of aluminum which makes it superior to any other material is its apparent complete lack of nitriding ability. Despite many months of continuous service, an aluminum container retains all of its original ductility. It exerts no observable catalytic action upon the dissociation of ammonia.

It is known that hydrogen is capable of embrittling many metals with which it comes in contact. The dissociation of ammonia produces nascent hydrogen as well as nascent nitrogen, yet aluminum is for my purpose completely immune from embrittling by hydrogen under the operating conditions of nitriding. So far as I have

been able to determine, aluminum acquires with service no catalytic action, and the results obtained with aluminum containers after many months of service are the same as and require no control steps different from those characteristic of their use at the commencement of such service.

In the containers in accordance with the present invention, the aluminum is employed primarily as a lining for the sake both of strength and of economy. The aluminum is obtained in the form of relatively heavy sheets, say, about $\frac{3}{8}$ of an inch thick, and is formed into the shape of the container desired. Joints are welded to give gas-tight results. Since aluminum is inert under nitriding conditions, the specific thickness of the sheet used to make the container is influenced solely by the mechanical considerations involved, and is of little importance where the required strength is attained from some other material, as hereinafter described. In the broadest aspect of my invention, however, I do not exclude the construction and use of containers made wholly of aluminum or aluminum alloys, inasmuch as containers of sufficient weight and strength may be so made, either from sheets or plates, or as castings, forgings, or the like.

Considering the exemplary form of my container, I have shown it in Figure 1, as a long, tubular body 1, of which the dimensions may be such as to comport with the type of articles being treated. The container is held, preferably removably, on a cradle or truck formed of side stringers 2, cross piece 3, and uprights 4. The parts may be made of angle irons welded together in a relationship such as that shown. As will be apparent from Figure 2, the cross piece 3 and uprights 4 may be cut within on arcuate lines to form a sort of cradle in which the tubular body 1 may rest. Since it is desirable to have some means whereby the container may readily be moved into and out of a muffle or furnace, I prefer to provide the cradle stringers 2 with bearing blocks 5 at intervals, in which I mount the reduced necks of rollers 6 which act as wheels for the device.

The container proper comprises a suitably strong tubular shell, indicated at 1a in Figure 3. This shell may be and preferably is of steel. At each end, it will be provided with flanges 7 or 8, and these likewise may be made of steel and welded to the tube 1a. The container is closed at both ends by discs 9 or 10, of substantially the same diameter as the flanges, and to which they may be bolted as by means of bolts 11. Between the discs and flanges, any suitable seal may be employed as desired.

I provide the tubular body 1a with an aluminum lining 12. This may be formed, as hereinabove described, of bent sheet aluminum with welded joints, but it may otherwise be formed, as will be clear. Since the coefficient of expansion of aluminum and steel differs markedly, I may form my lining with one or more corrugations 13 to take up expansion. These corrugations will not interfere with the loading or unloading or with the support of articles within the containers, providing they are kept close to the end or ends of the lining 12. I prefer, after the lining has been placed inside the body 1a, to turn its ends over the flanges 7 and 8, as shown at 14 in Figure 3. To protect the inner faces of the discs 9 and 10, as well as to prevent their inner surfaces from becoming nitrided and acting as catalysts, I coat them with aluminum in some

suitable way. This may be conveniently done by providing them with an aluminum sheet surfacing member 15, which may be held in place by aluminum bolts 16a or otherwise as desired. The sheet 15 is preferably coextensive with the disc to which it is attached, so that seal members may be placed between its periphery and the flanged portion 14 of the liner 12. For best results the joint between the incoming gas conduit and the aluminum covering should be gas tight.

At least one of the discs 9 and 10 will be perforated for the admission and/or egress of gas. In the containers hitherto employed in the art, it has been usual to introduce and withdraw the gas at the same end, if desired enforcing some sort of circulation within the container by a conduit or other means extending from the inlet or outlet at one end of the container to a point adjacent the other end. This, I find, is cumbersome and inefficient. Instead, I prefer to provide but one perforation in each of the discs 9 and 10. At each of these perforations, I attach a conduit, as at 16 or 17, and attach a three-way valve, 18 or 19, to each of the conduits. The remainder of the conduit connections are not illustrated; but it will be understood that the structure just described is one which enables me to introduce the ammonia at either end of the container and withdraw it at the other end to reverse the flow of ammonia at any time during the course of any given nitriding treatment. My standard practice is to run ammonia in one direction through the container for a period of two hours, then reverse it, and run in the opposite direction for an equal length of time. These reversals are repeated throughout the duration of the heat. In this way, any possible dead pockets in the container resulting from packing methods are eliminated. I have found that the results obtained through the use of my container in accordance with this process aspect are remarkably uniform. The articles treated are free from soft spots and exhibit a uniform case. The procedure works equally well with large and small articles.

The aluminum sheet 15 is, of course, perforated, as shown in Figure 3, at the point where the conduit 16 or 17 is attached. As is the practice in nitriding operations, I prefer to use as conduit material either nickel or Inconel; but the material for the conduits is not a limitation of my invention.

I prefer also to provide additional means for insuring uniform flow of the gases through the container. These means comprise plates 20, which are preferably in the form shown in Figures 4 and 5. The plates are turned over at their edges, as at 21, to form a cylindrical flange; and after filling the container, they may be installed in the liner 12 as shown in Figure 3 merely by being thrust therein until they abut one of the corrugations 13. Over an area opposite the perforation in the disc 9 or 10, the plate is left unperforated as at 22. This interposes a jet of gas issuing through the orifice in the disc and prevents the gas from passing on into the container in jet form. Elsewhere, the plate is perforated, as at 23, to permit entry of the gas into the liner 12 in a uniform manner. By these means, movement of the gas through the container is induced along parallel lines, and all articles within the container tend to be bathed in uniformly moving gas.

The container, where desired, may have a pro-

tective inner liner, as at 24; and this may be made also of aluminum.

Modifications may be made in my invention without departing from the spirit of it. While I have spoken of aluminum containers and aluminum linings for containers, I do not mean to exclude from the broadest aspects of my invention containers which are made of other materials, but which have imposed upon their inner surfaces at least a coating of aluminum of sufficient thickness, integrity, and durability to serve my purpose. Conceivably, this might be done by hot dipping or by the careful fabrication of containers from sheet steel or other sheet metal which has been coated with aluminum.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. A nitriding container comprising a body and a closure together with gas connections, substantially all interior parts of said body and closure presenting continuous aluminum surfaces inert to nascent hydrogen and nitrogen in a nitriding process.

2. A nitriding container comprising a body of substantially tubular form having end flanges, closure discs for attachment to said flanges, and gas connection means enabling the passage of gas through said container in either direction, substantially all interior portions of said container presenting an aluminum surface inert to nascent hydrogen and nitrogen in a nitriding process.

3. A nitriding container comprising a metallic shell and closure means therefor, said shell and closure means each having a lining of aluminum.

4. A nitriding container comprising a metallic shell and closure means therefor, said shell and closure means each having a lining of aluminum, the lining of said body being separate therefrom so as to be capable of expanding at its own rate under the influence of temperature, said lining having at least one corrugation therein whereby independent expansion and contraction is permitted, and means whereby the lining of said body may be sealed in a substantially gas-tight manner to the lining of said closure.

5. A nitriding container comprising a substantially tubular ferrous body having end flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and a tubular aluminum lining for the inner surface of said body.

6. A nitriding container comprising a substantially tubular ferrous body having end flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and a tubular aluminum lining for the inner surface of said body, said tubular lining being separate from said body, and having flanges at its ends lying along the flanges of said body.

7. A nitriding container comprising a substantially tubular ferrous body having end flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and a tubular aluminum lining for the inner surface of said body, said tubular lining being separate from said body, and having flanges at its ends lying along the flanges of said body, said tubular lining being further characterized by a corrugation permitting it to follow its own rate of expansion under temperature change.

8. A nitriding container comprising a substantially tubular ferrous body having end flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and

a tubular aluminum lining for the inner surface of said body, said tubular lining being separate from said body, and having flanges at its ends lying along the flanges of said body, said tubular lining being further characterized by a corrugation permitting it to follow its own rate of expansion under temperature change, said plates having gas connections therethrough, and gas distribution plates of aluminum located adjacent the ends of said tubular lining, having imperforate parts opposite said gas connections and being perforated elsewhere whereby to induce a flow of gas through said container along substantially parallel lines.

9. A nitriding container comprising a substantially tubular ferrous body having end flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and a tubular aluminum lining for the inner surface of said body, said tubular lining being separate from said body, and having flanges at its ends lying along the flanges of said body, said tubular lining being further characterized by a corrugation permitting it to follow its own rate of expansion under temperature change, said plates having gas connections therethrough, and gas distribution plates of aluminum located adjacent the ends of said tubular lining, having imperforate parts opposite said gas connections and being perforated elsewhere whereby to induce a flow of gas through said container along substantially parallel lines, said gas connection means each including a three-way valve whereby gas may be sent through said container in either direction.

10. A nitriding container comprising a substantially tubular ferrous body having end

flanges, ferrous plates for closing said flanges, an aluminum covering for one face of said plates, and a tubular aluminum lining for the inner surface of said body, said tubular lining being separate from said body, and having flanges at its ends lying along the flanges of said body, said tubular lining being further characterized by a corrugation permitting it to follow its own rate of expansion under temperature change, said plates having gas connections therethrough, and gas distribution plates of aluminum located adjacent the ends of said tubular lining, having imperforate parts opposite said gas connections and being perforated elsewhere whereby to induce a flow of gas through said container along substantially parallel lines, said gas connection means each including a three-way valve whereby gas may be sent through said container in either direction, and a protective inner liner of aluminum located within said tubular lining and being of lesser length.

11. A nitriding container comprising a body and a closure together with gas connections, substantially all interior parts of said body and closure which are subject to the nitriding temperature presenting continuous aluminum surfaces inert to nascent hydrogen and nitrogen in a nitriding process.

12. A nitriding container comprising a body and a closure together with gas connections, substantially all interior parts of said body presenting continuous aluminum surfaces inert to nascent hydrogen and nitrogen in a nitriding process.

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