CONTROLLED NITRIDING USING A ZEROFLOW PROCESS*

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Abstract

The present concept assumes carrying out the process of controlled nitriding with NH3 alone. This is a simpler process than that using two-component mixes of ammonia diluted with N2 or NH3diss. The regulation of the chemical composition of the atmosphere in the retort, and therefore the regulation of the nitriding potential KN, is obtained through the variation of the inflow rate of NH3 into the retort. What is a completely new things in a relevant process, the inflow of NH3 into the furnace is at times reduced to zero, which widens the range of the gas composition in the retort (and by the same token, of KN). The amount of NH3 introduced into the furnace is regulated (controlled) with the aid of a gas analyzer (for example an infrared analyzer). The gas analyzer opens and closes periodically the NH3 inlet valve with a frequency maintaining a desired gas composition (a desired KN) in the retort. The concept was based on experimental and theoretical investigations in term of thermodynamic and kinetic of gas nitriding. Investigations were carried out in a laboratory furnace with a quartz tube and also in an industrial furnace with a steel retort and a circulating fan. Nitriding was performed using NH3, NH3+H2, NH3+NH3diss. mixtures. In an industrial furnace the supply of NH3 into the retort was periodically closed, though with an operating circulation fan. The studies demonstrated that the growth rate of nitrided layer depends only on the composition of the atmosphere, (or K_N), in the retort. It depends neither on a type of atmosphere introduced into furnace (NH_3, NH_3+NH_3diss. or NH_3+H_2) nor a type of furnace (laboratory with a quartz tube or industrial with steel retort and fan). The results proved also that entirely closing of NH_3 supply to the retort does not affect the growth rate of nitrided layer. A detailed analyses of the results was performed in a paper.

Keywords: Nitriding; Layer; Kinetic growth; Gas consumption.

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1 INTRODUCTION

Traditional nitriding, which has limited control of the growth of the nitrided layer,is still very much in use worldwide[1]. The process is carried out using only ammonia, and the resulting layer is composed of superimposed ε + γ' +α (epsilon plus gamma prime plus alpha) phases. In industrial practice, the superficial zone of iron nitrides ε + γ' (as a rule exceedingly thick and brittle) is usually removed by grinding[2,3], which substantially adds to the cost of the manufacturing process. This process involves a high consumption of ammonia (NH3).Controlled gas nitriding, performed for more than 60 year represents an advancement over the (but still used today) traditional method using 100% ammonia atmospheres. Two-component atmospheres consisting of ammonia and dissociated ammonia (NH3 and dissociated (NH3)[4,5], as well as ammonia and molecular nitrogen (NH3 and N2) came into use about 50 to 60 years ago[6-8]. Through proper selection of atmosphere composition and adjustment of ts flow rate through the retort, it is possible to form layers of the required phase composition (consisting of ε + γ' +α, , γ' +α or α zones) having the required thickness of individual zones, together with a limited nitrogen concentration at the surface. These processes allow the elimination of final grinding, thus reducing the cost of the entire manufacturing process. However, nitriding using two-component atmospheres involves considerable consumption of gases as well. In addition, both processes have other disadvantages. Because of the nonequilibrium characteristic of nitriding in a NH3 + N2 mixture, control of kinetic growth of the nitrided layer is less accurate than in a NH3 and NH3 + dissociated NH3 mixture (nitriding in these two atmospheres exhibits equilibrium characteristics). In addition, the need for an ammonia dissociator when using the NH3 + dissociated NH3 mixture adds to the cost of the installation. This concept assumes carrying out the process of controlled nitriding using NH3 alone. This is a simpler process than that using two-component mixes of ammonia diluted with N2 or dissociated NH3. The regulation of the chemical composition of the atmosphere in the retort, and, therefore, the regulation of the nitriding potential KN, is obtained through the variation of the flow rate of NH3 into the retort. What makes this process unique is that the flow of NH3 into the furnace is at times reduced to zero, which widens the range of the gas composition in the retort (and KN as well). The amount of NH3 introduced into the furnace is regulated (controlled) with the aid of a gas analyzer (for example an infrared analyzer). The gas analyzer opens and closes the NH3 inlet valve periodically at a frequency required to maintain the desired gas composition (as well as the desired KN) in the retort. The concept is based on experimental and theoretical investigations in terms of the thermodynamics and kinetics of gas nitriding.

2 MATERIAL AND METHODS

Investigations were carried out in a laboratory furnace using a quartz tube and also in an industrial furnace using a steel retort and a circulating fan. Nitriding was performed using NH3, NH3+ H2, NH3+ dissociated NH3 mixtures. In the industrial furnace, the supply of NH3 into the retort was periodically stopped though with an operating circulation fan. The studies demonstrated that the growth rate of the nitrided layer depends only on the composition of the atmosphere or KN in the retort. It does not depend on either the type of atmosphere introduced into furnace (NH3, NH3 + dissociated NH3 or NH3 + H2) or the type of furnace (e.g., laboratory with a quartz tube or industrial with steel retort and fan). The results also prove that stopping the supply of NH3 to the retort does not affect the growth rate of nitride layer. Adetailed analysis of the results is given in Ref.[9]. Aspecial horizontal furnace suitable for nitriding was used to investigate the ZeroFlow process. The furnace was fitted with an NH3 inlet valve and an H2 gas analyzer. The regulation of the chemical composition of the atmosphere in the retort, and, therefore, the regulation of KN, was obtained as mentioned above by means of occasionally stopping the flow of NH3 into the furnace. The process was a two-stage nitriding process similar to that used in industrial practice.

3 RESULTS AND DISCUSSION

Figure 1a shows the variations of temperature, ammonia in flow rate, and NH3 content in the retort during two-stage nitriding. Operating parameters were: Stage I - 490°C (915°F) and 85% NH3 content in retort, Stage II - 530°C (985°F) and 40% NH3. The flow rate of NH3 into the retort was periodically closed both at the beginning of first and second stage of nitriding, which caused a relatively fast drop of NH3 content in the retort atmosphere up to the fixed values. Reaching a fixed gas composition takes up around 1 hour during the first stage and 46 minutes during the second stage (Fig. 1b). By comparison, in other processes, depending on the area of the charge, NH3 content reached a fixed value from 20 to 50 minutes. The rapid drop in NH3 is a favorable phenomenon from the standpoint of regulating the kinetics nitride layer growth. During the drop of KN, the possibilities of regulation of the growth of the nitrides are suspended until the gas composition reaches a level programmed for a given process stage. Beyond this point, gas composition is regulated through an intermittent flow of NH3 into the retort. The drop of KN depends on surface area and catalytic properties of retort and charge. Simulations of the growth of nitrides on 4140 were performed to estimate the influence of the rate of stopping the flow of NH3 (Fig. 2). The variation in changing rate of NH3 content (from 1 and 2.5 h) exhibits a small effect on the growth rate of nitrides (ε + γ'), and can practically be neglected. However, it must be noted that the dropping rate does not influence a growth of nitrided layer. Further, similar phenomenon also occurs during nitriding using two-component atmospheres. The investigation included a comparison between the nitrided layer obtained in a controlled standard process (using an NH3 + dissociated NH3 mixture) and that using ZeroFlow process. Figure 3 shows an example of a nirtrided layer and iron nitrides obtained.

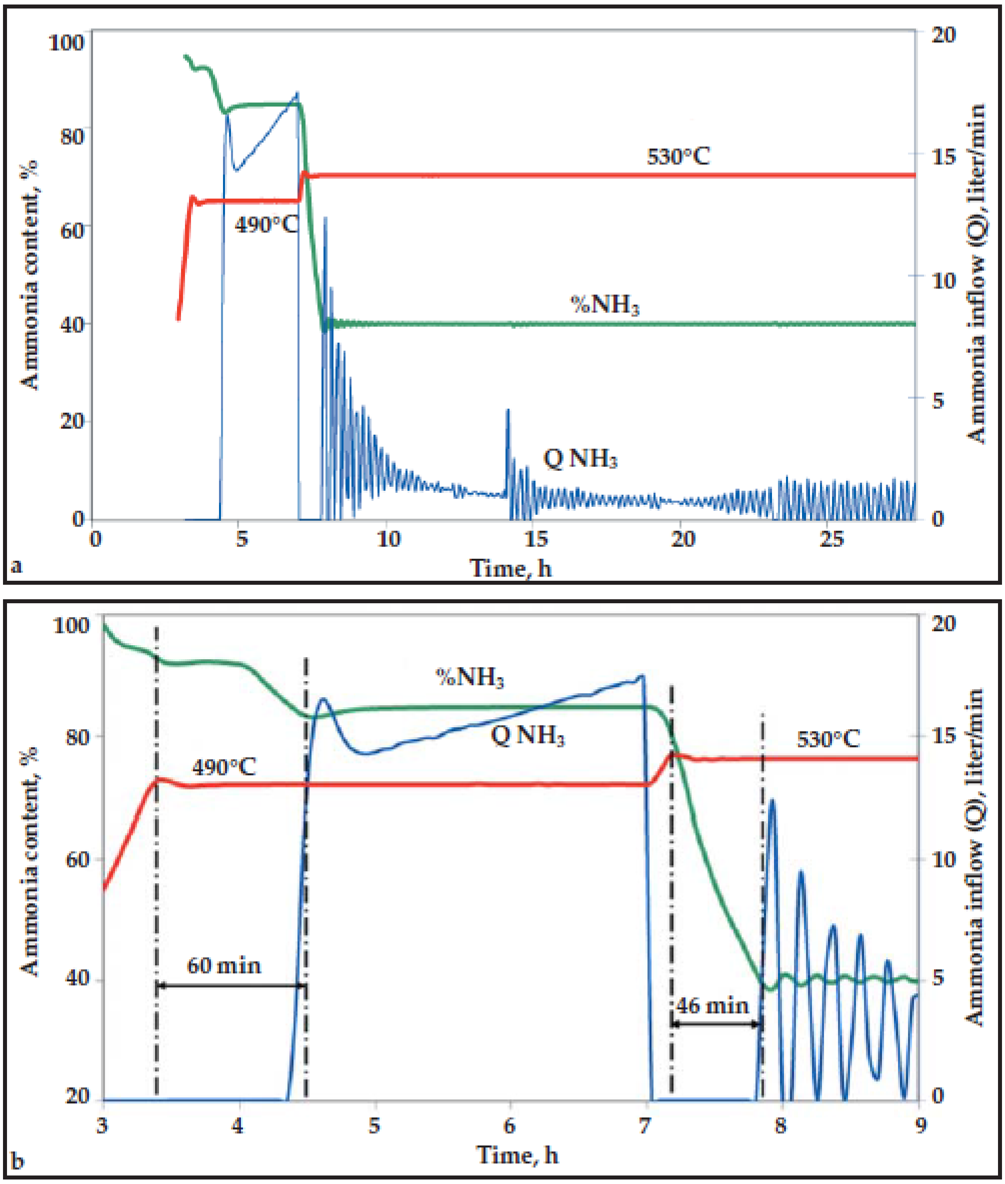

Fig. 1 — Variations of temperature, flow rate of NH3 into the retort, and NH3 content in a retort during two-stage nitriding (a); enlargement of left side of Fig. 2a between 0 and 9 hours (b).

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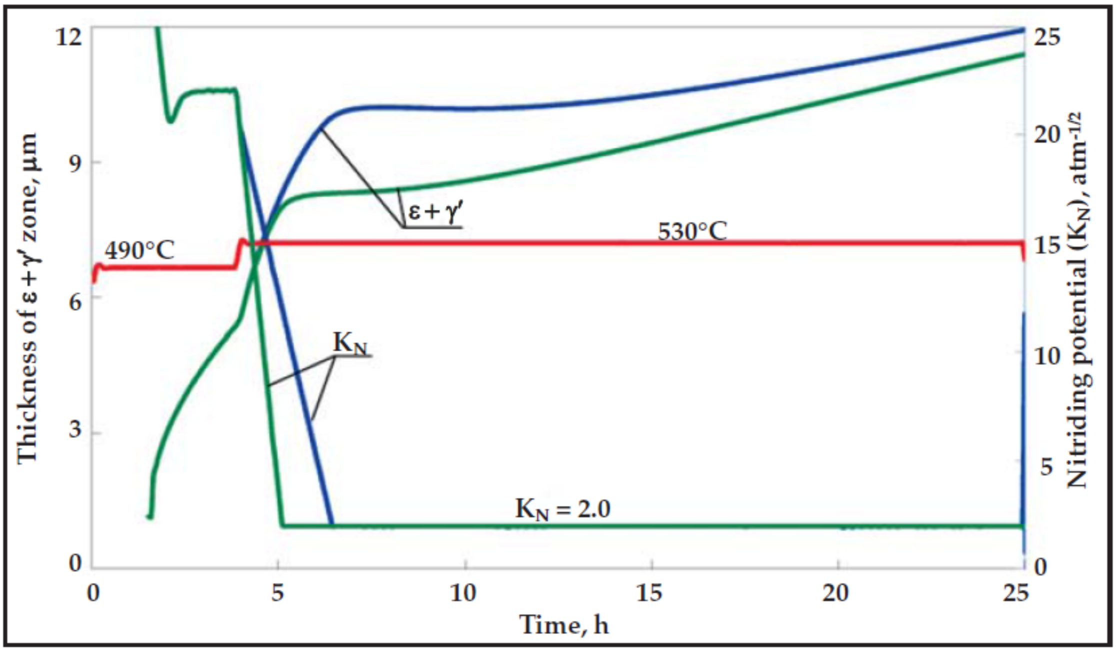

Fig. 2 — Comparison of growth rate of nitrides (ε + γ') on 4140 steel at a fast drop of KN (green) and slow drop of KN (blue).

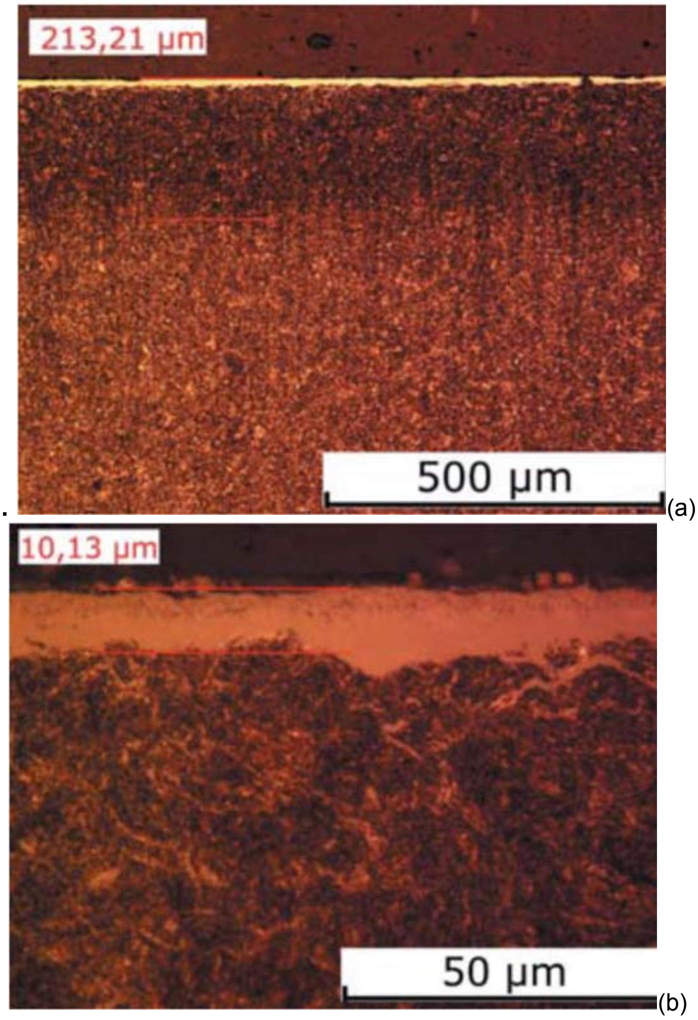

Fig. 3 — Photomicrographs of the nitrided layer (a) and iron nitrides (b) on 4140 steel nitrided using the ZeroFlow process at stage I (490°C, KN = 22 atm-1/2, and 3 h) and stage II (530°C, KN = 1.3 atm-1/2, and 22 hours).

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4 CONCLUSION

The results of the investigation indicate that by performing the Zero-Flow process using NH3 alone, it is possible to produce nitrided layers identical to those obtained using standard processes in two-component atmospheres. In such a process, the control of composition of the atmosphere in the retort is carried out by occasionally stopping NH3 flow into the furnace, thereby obtaining precise control of the kinetic growth of the nitrided layer similar to that available using NH3 + dissociated NH3 atmospheres. The ZeroFlow process offers practical, economical, and environmental benefits over processes using twocomponent atmospheres including:

- Low consumption of gas (up to 8 times less than in processes using NH3 + dissociated NH3 and NH3 + N2 atmospheres.
- Easier, less expensive nitriding intallation for the zero-flow process. Only one simple gas inlet valve and a gas analyzer is required to precisely regulate and control the chemical composition of the atmosphere obtained from NH3 compared with two inlet valves, a high quality gas flow meter, and a gas analyzer required for processes using NH3 + dissociated NH3 and NH3 + N2 atmospheres. In addition, a dissociater is required for the dissociated ammonia.
- The ZeroFlow process can be carried out in furnace fitted with a steel retort, which is significantly cheaper than that made of a Ni-base heat resistant alloy required for processes using a two-component atmospheres.

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