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FACULTY OF MECHANICAL ENGINEERING AND INFORMATICS



THE INFLUENCE OF BIAS VOLTAGE ON THE LAYER STRUCTURE IN ACTIVE SCREEN PLASMA NITRIDING

Booklet of PHD Theses

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The development of the technology meant the understanding of the process and layer formation (Bell, 1991), and improvement of different techniques. In industry there are three different methods to create nitrided layers. The gas nitriding has the lowest investment costs, the salt bath nitriding has the shortest process time, and the plasma nitriding means the lowest pollution. Every type has such kind of advantages, why it can be regarded suitable for certain applications; none of them is best for all cases.

1.2. Objectives

The active screen plasma nitriding method as the newest process has been chosen as the main topic of my theses.. The plasma nitriding technology is a complex process, so the researchers have to face a lot of unanswered questions. Besides understanding the kinetic of plasma nitriding there are new and more rigorous requirements against the products regarding the uniqueness and reproducibility, therefore, the technology should be continuously further developed.

The science of heat treatment was always an important research and education field at the Institute of Materials Science and Technology (formerly Department of Mechanical Technology). In the education, the fundamental knowledge of heat treating is considered as the main topic, but in some special lectures and project work, the most modern and newest technologies are also part of the curriculum. The heat treatment of steels – in particular the tools steels – has also important role in the research and development work of the Institute.

In many cases the ductile core and hard – a therefore wear resistant – surface is the requirement for mechanical components. The application of surface technologies can be regarded as a solution for these problems. Nitriding is one of these technologies . The plasma nitriding means low pollution (Georges et al, 2010), which makes this technology really important in the 20th century. Even the basic literatures (Totten, 1997) (Pye, 2003) (Krauss, 2005) (Mittemeijer, 2013) acknowledge the difficulties of plasma nitriding technology. Most of these problems (edge effect, non-uniform temperature in the furnace, arcing) can be neglected by applying the active screen plasma nitriding (ASPN) (Zhao, 1996). The total or partial removing of discharge from the workload to a metallic screen not just solve many of the problems (Li-Bell, 2003) (Hubbard, 2007) (Gallo-Dong, 2010) (Li, 2010), but also expand the application field of nitriding. This is the main reason why the research of the ASPN is a central topic and this is also the reason that I selected as the main theme of my theses, too. The research of this technology is at the beginning (Venturini, 2011) (Somers, 2013), and offer a lot of challenges for engineers (Doyle- Hubbard, 2009) (Nishimoto et al, 2009) (Nishimoto et al, 2010).

There is a lot of technology parameter of ASPN (Li-Bell, 2003) (Ahangarani, 2006) (Hubbard, 2007) (Gallo-Dong, 2010) (Li, 2010), but the most important is the BIAS voltage (Li et al, 2010). I decided to perform a systematic research to study the nature of the influence of this secondary voltage applied on the components to be treated.

2. METHODOLOGY

In the first part of the literature summary I described the metallography of nitrided layer. The iron-nitrogen equilibrium phase diagram is similar to the iron-carbon system due to the similarity of the effects of nitrogen and carbon. Nevertheless, the solubility of the nitrogen is higher than the carbon, and only ordered solution can be formed. The layer consists of a very hard white layer, and a moderated hard diffusion zone. The white layer ensures for the components wear and corrosion protection, diffusion zone has good fatigue resistance, and provides a support for the hard upper layer.

In the second part of the literature summary I introduced the technology of plasma nitriding. Direct current plasma nitriding (DCPN) generates glow discharge on the surface of the cathodic components, while the wall of the chamber is the anode. Although this is a reliable and modern technology, there are many difficulties which limit the application potential. These problems are: arcing effect, edge effect, hollow cathode effect, non-uniform temperature in the chamber. All of the application difficulties can be neglected or at least prevented, if the discharge partially or totally removed from the surface: this is the substance of active screen plasma nitriding (ASPN) technology. ASPN means the application of a metal screen (perforated sheet, foil or mesh) as a cathode, so the discharge happens on it. The components are on relatively low (BIAS) or floating potential. In case of DCPN the heating occurs by the bombardment of the ionised nitrogen, in case of ASPN, the hollow cathode effect occurring on the screen heats the parts by radiation: this kind of heating is much more uniform. The relatively low BIAS voltage does not cause edge effect or arcing. ASPN is the newest method for nitriding and it offers a lot of open questions for the researchers – that is why I have chosen this topic for my thesis.

Some researchers investigated the influence of the BIAS voltage on the depth of layers, but they are not compatible, so to compare these is not possible without limitations. In my research I have chosen 7 different steel grades: low and high alloy types, respectively. During the heat treatment processes only the value of the BIAS voltage was changed.

Hardness measurements and optical microscope investigations are the most commonly applied methods to characterize the nitrided layers. I measured the micro-hardness (HV0,1) of the layers as a function of the distance from the surface, so I could determine the diffusion depth at point, where the hardness is higher with 50HV0.1 than the base material hardness. The depth of the white layer can be measured and the case structure can be observed on optical microscope.

The white layer has an important role in wear resistance of nitrided components, especially in case of low alloy steels. The measurement of this part of nitrided layer is possible on the cross section of the components, using optical microscope. This method requires time and some device, which is not available for all of the industrial companies. I successfully applied the ball-cratering (i.e. Calotest) method combined with Nital etching to measure the depth of white layer. It is low-destructing and fast way to measure the depth of white layer.

During ASPN the growing of the nitrided layer is not linear: if the BIAS decreases, the amount of the nitrogen elements, which reach the surface, decreases. The lower nitrogen content of the surface delays the formation of white layer, in which the diffusion rate of the nitrogen is lower than in the diffusion zone. Due to this later formation, the maximum hardness of the diffusion layer occurs; when the BIAS voltage is lower (17%).

To compare the wear resistance of the produced layer I applied ball-on-disc wear test, using ZrO₂ ball with 3 mm diameter. The low oxidation wear is the typical mechanism for the

nitrided layers, which causes a lot of brown, lamellar debris on the surface. In case of high oxidation wear mechanism, the debris is black; the quantity of it is lower. Compared to the high oxidation wear in case of low oxidation the coefficient of friction is higher, but the area of the wear track is lower.

After comparing the layer structure I found that the layer depth in case of ASPN is higher than in DCPN: thanks to the applied screen, which is closer to the surface of the components, than the wall of the furnace, the path of the nitrogen ions is shorter: it means intensifying effect, and this is valid until the efficiency limit of the ASPN.

Although, it often can be read in the literature that edge effect cannot be seen in case of ASPN, only some researchers made measurements to characterize this phenomenon. I made some hardness measurements as a function of the distance from the edge, and I found followings: the edge effect occurs at higher hardness and inhomogeneity in the nitrided layer, the scatter of the measured data's is higher in case of DCPN compared to the ASPN.

3. NEW SCIENTIFIC RESULTS – THESES

- T1. In case of active screen plasma nitriding of BIAS on the surface structure (depth of white layer and diffusion zone) is strongly dependent on the base material composition, especially on the nitrogen-forming element content:
- if the chromium content is very high (13%), the depth of white layer does not change significantly, but the depth of the white layer decreases, if the BIAS decreases;
 - if the chromium content is high (5%) and the BIAS is lower than 17% of the main voltage, only diffusion layer (no white layer) is formed (11);
 - for low chromium content the maximum hardness of the diffusion layer can be achieved at 17% BIAS(10)(15),
 - nitriding behaviour of nitridable steels is similar to the high chromium content steels (22).
- T2. During active screen plasma nitriding because of the limited nitrogen absorption the white layer forms later; this is beneficial for nitrogen diffusion rate. Because of the white layer forming delay not the highest BIAS voltage results the deepest diffusion zone. In terms of layer depth and layer hardness 17% BIAS is the optimum (15).
- T3. The ball-cratering (Calotest) method combined with Nital etching is a new, low-destructive, quick method to measure the depth of white layer, without making cross-section specimen.
- T4. During ball-on-disc wear test, using ZrO₂ ball, near the wear track producing large amount of rust coloured, lamellar debris is typical. In some cases heavy oxidation wear can be observed, when the amount of debris is lower, its colour is black, and its shape is granular. In case of low oxidation wear the steady value of friction coefficient is higher, although the cross section of the wear track is smaller.
- T5. Comparing nitrided layer produced by conventional and active screen plasma nitriding we can conclude that applying the same process parameters (temperature, process time, gas consumption, pressure) the layer depth is higher for active screen nitriding. The active screen – if the BIAS voltage value is satisfactory for case producing – increase the intensity of nitriding.
- T6. In case of conventional plasma nitriding compared with active screen plasma nitrided layer, the results of the edge effect are (34):
- the visually observed discolouration around the edges means higher hardness compared to the inner part of the component,
 - the relative deviation of the measured hardness, and the uniformity of structure properties is less favourable in conventional nitriding compared to the layer produced by active screen plasma nitriding,
 - the probable cause of the difference is the higher nitrogen content.

These accepted by Judging committee:

- T1. During active screen plasma nitriding due to the limited nitrogen absorption the white layer formation occurs later; this is beneficial for the diffusion rate of nitrogen. For higher value of secondary voltage the higher amount of absorbed nitrogen results in earlier appearance of the white layer, leading to the decrease of diffusion rate. Based on experimental results for hardened and tempered steels, the value of secondary voltage resulting in the hardest diffusion zone can be assigned at given process parameters (15).
- T2. The ball-cratering (Calotest) method combined with Nital etching is a new, low-destructive, quick method to measure the depth of white layer, without making cross-section specimen.
- T3. During ball-on-disc wear test, using ZrO₂ ball, near the wear track producing large amount of rust coloured, lamellar debris is typical. In some cases heavy oxidation wear can be observed, when the amount of debris is lower, its colour is black, and its shape is granular. In case of low oxidation wear the steady value of friction coefficient is higher, although the cross section of the wear track is smaller.
- T4. Comparing nitrided layer produced by conventional and active screen plasma nitriding we can conclude that applying the same process parameters (temperature, process time, gas consumption, pressure) the layer depth is higher for active screen nitriding. The active screen – if the BIAS voltage value is satisfactory for case producing – increase the intensity of nitriding.
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4. INDUSTRIAL UTILIZATION AND POSSIBILITIES FOR THE FURTHER DEVELOPMENT

Firstly, I would like to emphasise that the plasma nitriding is rarely applied in Hungary. So I think the research work like mine can increase the acceptance and reputation of this method. The gas and salt bath nitriding – which are known and widely used in industry – have also good application possibilities, but the low pollution of plasma nitriding is a really important factor in 21st century.

The active screen technology is hardly known in the industry, so I hope this will change. It is important to know, the conventional plasma nitriding furnace can be suitable for this technology with some modifications. Application of active screen plasma nitriding has some advantages:

- compared with other surface modification processes the nitriding result in lower distortion, better heat resistance and higher hardness of the components,
- compared with case hardening the control and thermal stability of the layer is better, the nitrided component does not require post heat treatment, the volume change is minimal,
- the plasma nitriding compared with other nitriding methods means lower pollution and lower consumption.

In connection of my research results, the following industrial utilization potential can be assumed:

- the knowledge of the influence of BIAS voltage on layer structure for different steel types can help further reduction of process costs, and the planning or modification of plasma nitriding furnaces,
- applying active screen the intensity of nitriding is higher, so the process time can be shorter, or during the same time higher layer depth can be produced,
- applying active screen the uniformity of nitriding layer is better, which means better performance, especially in case of components with smaller size.

The research of active screen offers a lot of opened questions for scientists. These research fields can be:

- the influence of screen geometry (hole size, type) on layer structure and properties,
- the influence of the screen material on layer structure and properties,
- the hole size of the specimen, which can be nitrided applying active screen, and its dependence on process parameter
- non-conductive materials (i.e. polymers) nitriding potential and surface properties.

5. LIST OF PUBLICATIONS RELATED TO THE TOPIC OF THE DISSERTATION

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