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LOW-CARBON SHEET METALS TREATED BY NITROOXIDATION IN FLUID LAYER AND THEIR PROPERTIES

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Abstract: *The paper deals with fluid nitrooxidation technology applied to the low carbon steel sheet metals and influence of this process on mechanical properties, formability, structural changes and weldability of this way treated materials. Experiments showed significant increase of mechanical properties (strength and microhardness), moderate decrease of formability and 10-times increase of resistance to atmospheric corrosion in comparison to untreated materials. The optimal welding method appears to be laser welding.*
Key words: *nitrooxidation, low-carbon steel sheet, welding, forming, corrosion resistance*

1. INTRODUCTION

Progressive and continual demands in properties of engineering materials led to innovation in the field of surface treatment technologies. Surface modification of engineering materials is fast developing field of materials science in recent time. Application of surface treatment to these materials allows them to achieve qualitatively new properties. The tendency towards innovation and modification of known methods of thermochemical treatment was recorded in the present, in order to increase the mechanical, tribological and corrosion properties at simultaneous obtaining of aestethic appearance of products. This group also involves the processes providing nitrogen layer deposition or nitrogen implantation. There are known several methods like plasma implantation, sputtering process by ion beam assistance which belong to the modernest ones, but also to the most financially demanding. That is why the research is focused on not only new methods of surface treatment, but also on innovation already known processes of thermochemical treatment. To the modified unconventional methods of thermochemical treatment belongs also nitrooxidation in fluid layer, at that the oxidation follows the nitrogen surface saturation [1, 3, 4, 5].

The nitrooxidation process developed by German company Degussa is quite well known as TENIFER QPQ process in nitrogen salts, but application of this process by utilizing fluid technology is not so known despite its less environmental side effects and lower cost comparing to nitrogen salts and cyanides typical for process TENIFER QPQ mentioned above [1].

The nitrooxidation process is applicable for components at which increased and lifetime are necessary (particularly for components operating in humid environments). Due to increased strength the weight of component is decreased, at maintaining other required mechanical properties. This phenomenon is in present utilized in automotive industry, in technology of consumer and special equipment.

This paper deals with analysis of characteristics of low-carbon steel sheets after nitrooxidation treatment.

The aim of this paper was to review the microstructure and properties changes of low-carbon deep-drawing steel sheets after nitrooxidation process.

2. EXPERIMENTAL

Low-carbon deep-drawing steel DC 01/DIN EN 10130-9 thickness 2 mm, treated by nitrooxidation process in fluidised layer was used in this investigation. Its chemical composition is given in Table 1. Fluidised bed was composed of Al2O3 with grain size of 120μm. The waft of the fluidised layer during treatment was provided by gaseous ammonia, during oxidation using a vapour of distilled water, supplied to the furnace chamber. Conditions of this treatment were as follows: nitridation at 580°C/120 minutes, oxidation at 380°C/5 minutes, air cooling.

Table 1. Chemical composition of experimental steel [wt. %]

EN code	C [max %]	Mn [max %]	P [max %]	S [max %]	Si [max %]	Al [min %]
DC 01	0,12	0,60	0,045	0,045	0,1	-

3. RESULTS

- **Structural analysis**

The nitrooxidation treatment produced a characteristic surface layer about 300 μm in thickness (Fig. 1). This was composed of two zones – about 70 μm thick compound layer, with continuous thin Fe_3O_4 a Fe_2O_3 layer of ~ 3 to 5 μm in thickness and ε-phase ($Fe_{2-3}N$), about 10 μm in thickness.

Layer of ferritic matrix with needle-shaped γ′- Fe_4N nitrides (Fig. 2) was connected to the ε-phase. Its thickness was about 50 - 60 μm. The 200 μm thick diffusion layer was connected to the compound layer and formed the transition to the steel substrate. The diffusion layer was composed of ferritic matrix with dispersed fine α′′- $Fe_{16}N_2$ precipitates.

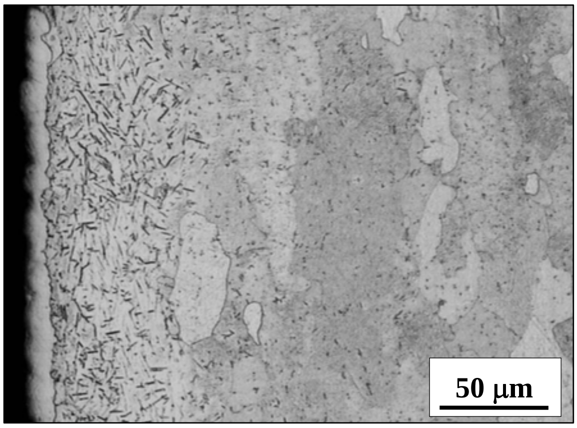

Fig.1 Microstructure of nitrooxidised low-carbon steel DC 01

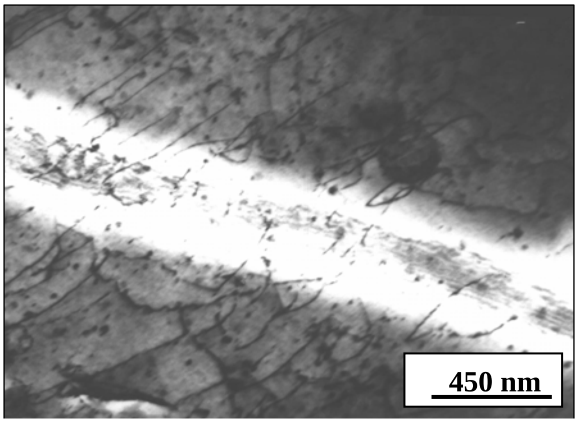

Fig. 2 The close up of γ′-Fe_4N massive particle in interaction with dislocations

- **Mechanical properties**

Improvement of the maximum strength of 41% was observed. Surface microhardness was increased of 653 % compared to basic state steel.

- **Formability**

9% decrease of cupping „h“ (IE) during the Erichsen cupping test and 1,5 % increase of deep- drawing coefficient „m“ during Fukui deep-drawing test were observed for nitrooxidised steel. Precipitated nitrides had no influence on the microstructure changes during forming and caused no failure in the surface layer of bended nitrooxidised steel.

- **Weldability**

There were tested three welding methods: GMAW, resistance welding and laser welding.

The surface oxidic layer caused problems during first two methods welding. There were difficulties with arc ignition and stability [2]. Should the GMAW method be required, the surface oxidic layer has to be removed, but on the other hand it may lead to reduction of corrosion resistance. The microstructure of weld joint after GMAW process with pre-removed oxidic layer is on Fig. 3.

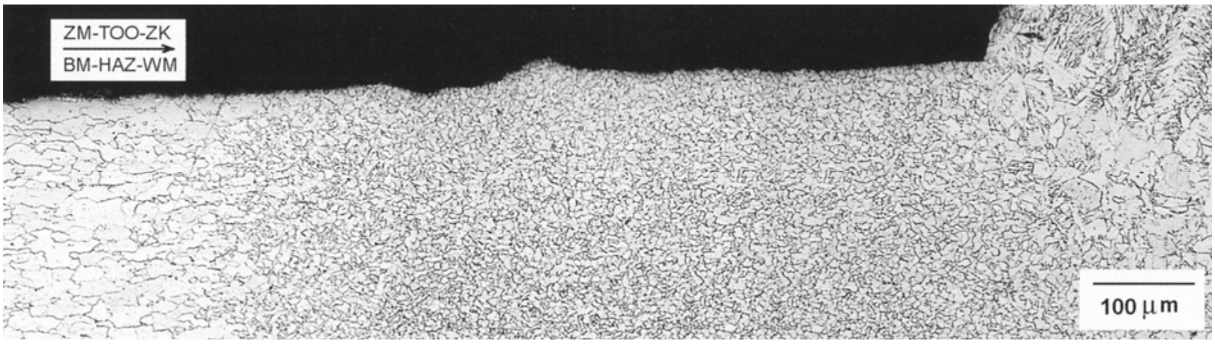

Fig. 3 Microstructure of laser welded joint (base material (BM)- weld metal(WM) - heat affected zone HAZ))

The oxidic layer brought obstructions also during resistance welding due to its low electrical and thermal conductivity. There had to be used ultra low welding parametres with hugely prolonged welding time.

The optimal welding method of nitrooxitation treated low-carbon steel sheets appears to be laser beam welding. Welding parameters are in Table 2. The close up of a laser welded specimen after tension test is on Fig. 4.

Table 2. Laser welding parameters

Laser type	Phase laser IPG 2300W
Protective gas	Linde LASGON C1 (15 % CO_2, 35 % He, 49 % Ar)
Welding speed [$m.min^{-1}$]	3
Laser beam power [W]	2000

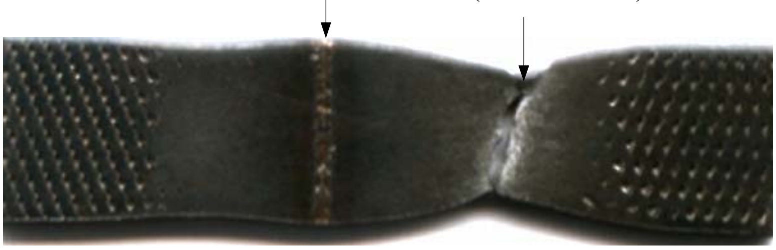

Fig. 4 Sample break in base material after laser welding

The nitride dissolvation, observed at all welding methos was the lowest (approximately 1 mm from weld metal neighbourhood) due to narrowest HAZ (Fig. 5).

Fig. 5 Microstructure of laser welded joint (weld metal - base material - heat affected zone)

Concerning multi-phase composition of nitrooxitation treated low-carbon steel sheets the were not observed any undesirable manifestation of microstructure change. The presence of secondary created nitrides or dessolving of nitridic phases due to welding had the positive influence on microstructure of overheated part of HAZ, where usually the coarse grain can be observed. The nitrides also positively affected the weld joint strength.

- **Corrosion properties**

Nitrooxidised steel showed 10-times increased resistance to atmospheric corrosion compared to basic state steel. Only 2% of sampled area was attacked after 240 hours in condensed water test cabinet.

Table 3. The results of corrosion test of material DC 01

STAV	Evaluation of corrosion changes at sample surface during and after corrosion test in test cabinet according to STN 03 8131				
	Check after 16 hrs	**Check after 48 hrs**	**Check after 72 hrs**	**Check after 144 hrs**	**Check after 240 hrs**
Basic (without nitrooxidation)	without corrosion	Corrosion at 3 % of area	Corrosion at 10 % of area	Corrosion at 60 % of area	Corrosion at 80 % of area
After nitrooxidation	without corrosion	without corrosion	Presence of 2 corrosion points	Presence of 3 corrosion points	Presence of 6 corrosion points

4. CONCLUSIONS

The microstructure and properties changes of low-carbon deep-drawing steel sheets after nitrooxidation process in fluidised bed were investigated.

It was shown that nitrooxidation had a beneficial influence on mechanical properties, formability as well as corrosion resistance. Due to this effect the weight of products could be decreased, at maintaining of required formability and also improved lifetime.

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