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COMPARISON OF LASER AND ELECTRON BEAM WELDING OF STEEL SHEETS TREATED BY NITROOXIDATION[1]

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Abstract

The nitrooxidation process in fluid environment significantly increases mechanical properties of treated materials. On the other hand the surface layer created by this process brings several problems during arc welding. This paper deals with influence of nitrooxidation process on structural changes, weldability, mechanical properties and formability. Specimens welded by CO_2 laser and EBW were examined by visual inspection, microstructure analysis and microhardness analysis. Visual inspection of specimens welded by LBW proved lack of root penetration at maximal welding speed from selected welding speed range (30 to 60 mm/s) and good looking weld at welding speed of 40 mm/s. However, EBW process produced weld joints with wider heat affected zone in comparison to laser beam welding and weld joints which were much more prone to porosity. The structure has not showed any abnormalities in phase composition, which could be expected due to existing nitrides in surface layer of materials welded after their nitrooxidation.

Keywords: Nitrooxidation; Laser beam welding; Electron beam welding.

COMPARAÇÃO DE SOLDA POR LASER E POR FEIXE DE ELÉTRONS EM CHAPAS DE AÇO TRATADAS POR NITRO-OXIDAÇÃO

Resumo

O processo de nitro-oxidação em ambientes fluídos aumenta significativamente as propriedades mecânicas dos materiais tratados. Por outro lado, a camada superficial criada por esse processo traz vários problemas durante a soldagem por arco. Esse trabalho lida com a influência do processo de nitro-oxidação em mudanças estruturais, soldabilidade, propriedades mecânicas e conformabilidade.As amostras soldadas com laser CO_2 e por EBW foram examinadas através de inspeção visual, análise de microestrutura e análise da microdureza. A inspeção visual das amostras soldadas através de LBW provou a falta de penetração na raiz na velocidade máxima de solda em uma faixa de velocidade de solda selecionada (30 à 60 mm/s) e uma solda de boa aparência à uma velocidade de solda de 40 mm/s. Entretanto, o processo de soldagem EBW produziu juntas soldadas com uma zona mais afetada pelo calor em comparação à solda por raio laser, e juntas soldadas muito mais propensas à porosidade. A estrutura não mostrou nenhuma anormalidade na composição da fase, que poderia ser esperada devido a existência de nitretos na camada superficial dos materiais soldados depois da nitro-oxidação.

Palavras-chave: Nitro-oxidação; Solda por raio laser; Solda por feixe de elétrons.

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INTRODUCTION

Properties of new materials are often focused on increasing mechanical, tribological and corrosion characteristics. Surface modification of engineering materials is fast developing field of materials science in recent time. Among the often used surface modification processes belong processes providing nitrogen layer deposition or nitrogen implantation. There are known several methods e. g. plasma implantation and sputtering process by ion beam assistance. However, these processes pertain to the most financially demanding. That is why the research is concentrated not only on new methods of surface treatment, but also on innovation of already known thermochemical treatment processes. One of them is nitrooxidation in fluid layer where oxidation follows the surface saturation by nitrogen.

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.

The nitrooxidation process is applicable for components where lifetime increase is necessary (particularly for components operating in humid environments). There can be also lowered the weight of components due to a higher material strength and comparable other required mechanical properties.[1] These factors are at a moment applied in automotive industry, consumer goods industry and special equipments.

Research and development of new materials should go hand in hand with new technologies of their processing, especially welding. This paper deals with possibilities of laser beam welding of low-carbon steel sheets after nitrooxidation treatment.

Material Structural Charactreristics

The nitrooxidation treatment produced a characteristic surface layer about 300 μm in thickness (Figure 1**Figure 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 0.6 to 0.7 μm in thickness and ε-phase ($Fe_{2-3}N$) with approximate thickness of 10 μm. The oxidic layer is being formed immediately after nitridation process, but its thickness is lower, i. e. 0.3 to 0.4 μm.

Layer of ferritic matrix with needle-shaped γ´- Fe_4N nitrides (Figure 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.[2]

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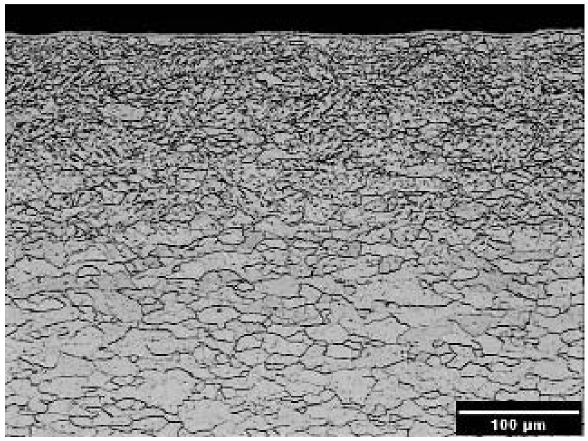

Figure 1. Microstructure of nitrooxidised low-carbon steel DC 01.

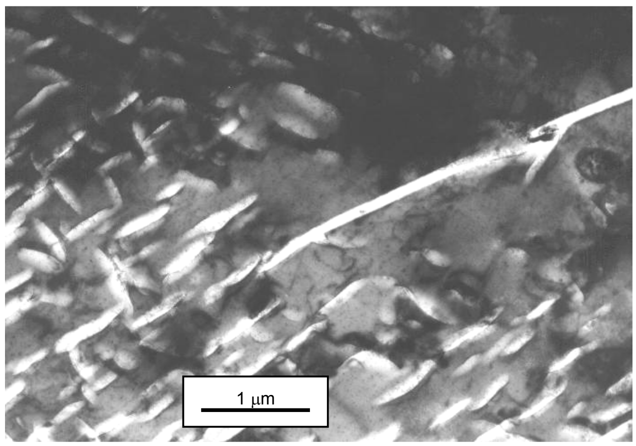

Figure 2. Detail of ferritic matrix with needle-shaped γ´- Fe_4N nitride.

Figure 3 represents the surface oxicic layer after nitrooxidation process.

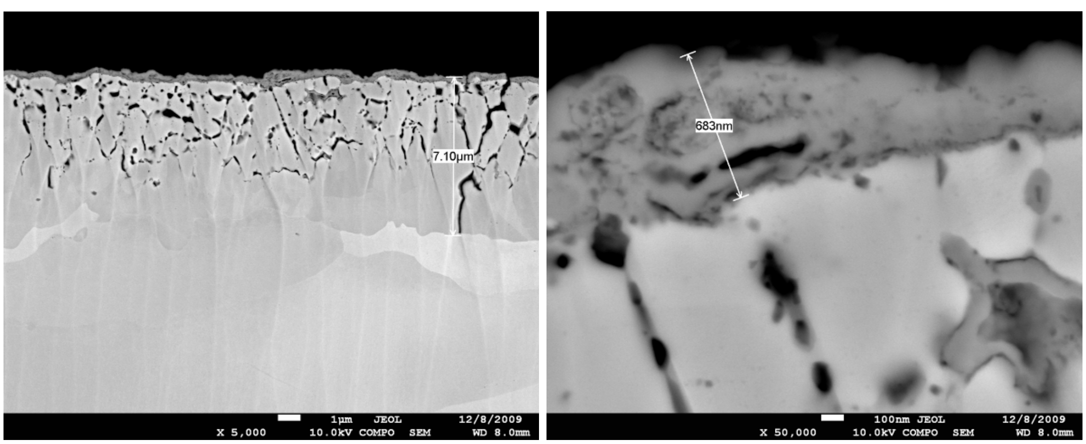

Figure 3. Presence of surface oxidic layer after 5 minute nitridation.

Mechanical Properties of Material

Measurements of mechanical properties consisted of tensile strength test and microhardness measurements.

The transverse tensile test was carried out according to standards STN EN 895 and STN EN 10002-1:2001. There was used 12 samples divided in to four groups (three samples in each group) with different treatment: group 1 – without treatment, group 2 - nitridation 580 °C/45 min , oxidation 350°C/5 min, group 3 - nitridation 580 °C/ 45 min, oxidation 350°C/10 min and group 4 - nitridation 580 °C/45 min, oxidation 350°C/15 min. Data obtained and calculated from the tensile test are shown in Table 2, the typical tensile test graph of steels with and without nitrooxidation treatment is shown in Figure 4 (graph showing the test result after surface treatment represent other two group as the results were similar).

Table 1. Measured values at tensile test

Group	Re [MPa]	Rm [MPa]	A [%]
1	200	282,3	31,2
2	308	377,3	23,7
3	304	400,7	23,7
4	307	383	23,3

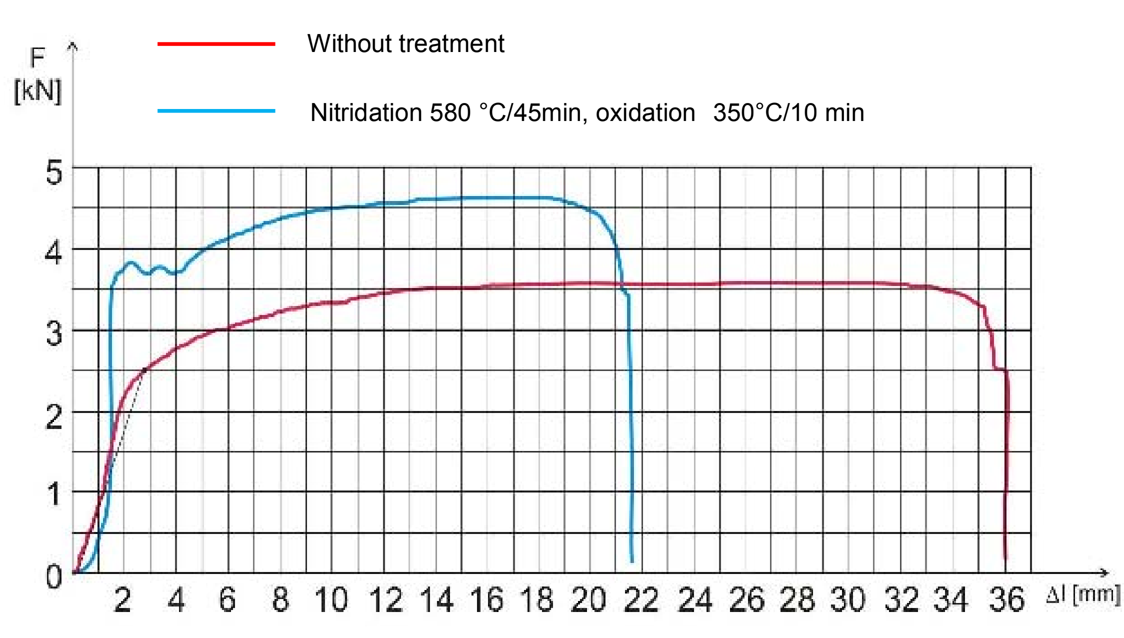

Figure 4. Tensile test graph of selected samples.

The tensile test exhibited maximum strength enhancement of 34 to 42 % for nitrooxidised materials and also yield strength enhancement of 52 to 54 % in comparison to base material without treatment. The ductility decreased approximately by 7.5 %. Differences among treated groups were insignificant and represented about 6 %. That proved the estimation that the oxidation stage in nitrooxidation process has minimal or no influence on mechanical properties of treated materials.

Maximal hardness of 1130 HV 0.01 was observed in the nitrooxidation layer. Surface microhardness increased by 653 % compared to steel without nitrooxidation treatment.

Formability

During the Erichsen cupping test there were observed 9 % decrease of cupping „h“ (IE) and 1,5 % increase of deep- drawing coefficient „m“ of Fukui deep-drawing test 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.

Experimental

The base material used in experiment was low-carbon deep-drawing steel DC 01/DIN EN 10130-9 with thickness of 1 mm, treated by nitrooxidation process in fluidised layer. Chemical composition of used material is given in Table 2. Fluidized medium was composed of Al_2O_3 with grain size of 120 μm. The waft of the fluidized medium 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 2. 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	-

As previous experiments showed problems with weldability by GMAW[3] and resistance welding methods due to surface oxidic layer being on the other way necessary for good corrosion properties, the laser beam welding was carried out. This method is used in an automotive industry for welding of coated thin steel sheets.[7]

The CO_2 laser Ferranti Photonics AF 8 with 10.6 μm wavelength was used. Table 3 shows the used welding parameters.

Table 3. Laser beam welding parameters

Laser type	Ferranti Photonics AF 8 CO_2
Protective gas	Ar 99,996% (18 l/min)
Welding speed v_z[mm/s]	30, 40, 50, 60
Laser power [W]	2000

Nitrooxidised material was welded by electron beam welding on PZ EZ ZH1 machine with various parameters at First Welding Company, Inc., Slovakia. In order to exclude potential errors during positioning and fixing base materials, the penetration welds (further named as welds) on a one piece of material were carried out. Operating pressure in electron beam gun and welding chamber was $4x10^{-4}$ Pa and $4x10^{-2}$ Pa respectively. Many welds were rejected due to porosity and spatter observation (spatter can be seen in Table 4). Considering this, oscillation of the beam (forward-backward) has been applied in welding process. Variable parameters in this research were:

- the length of the oscillation: 4 and 5.5 mm
- oscillation frequency: 40, 60 and 120 Hz
- focus current: 720, 726, 736 mA
- welding speed: 5, 10, 15, 20 mm/s
- welding current: 3, 5, 10, 12, 15 mA

There also have been done tests with more welding passes. The sample selected as the best result had three passes (Table 4) in order to get suitable, poruses free weld. First pass was considered as a cleaning one where oxides were tried to be removed. The second pass was the penetrating pass and the third one was for the "cosmetic" modification of the surface.

Table 4. Welding parameters

Pass No.	Welding voltage [kV]	Welding current [mA]	Welding speed [mm/s]	Focusing current [mA]	Frequency [Hz]
1	40	3	10	736	60
2	40	10	10	736	60
3	40	5	10	736	60

RESULTS

Weld surfaces, weld roots and cross-sections as dependence on LBW parameters are shown in Table 5.

Table 6 shows weld surfaces, weld roots and cross-sections as dependence on EBW parameters of specimens having the lowest spatter and lack of porosity.

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Table 5. Laser beam weld surface and cross-section appearance

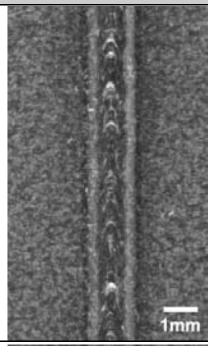
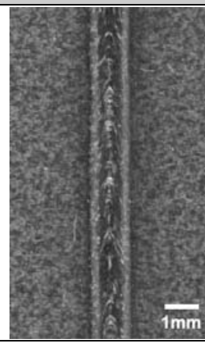
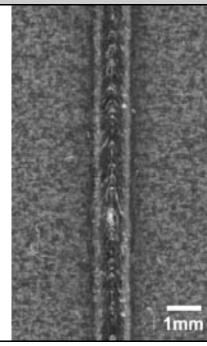
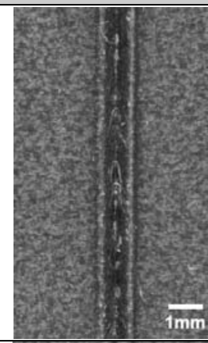
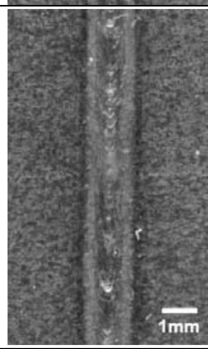
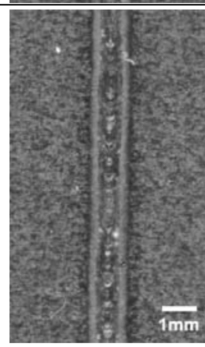
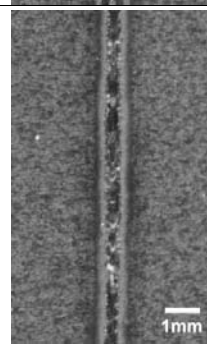
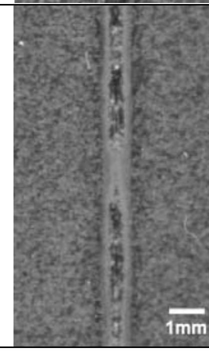
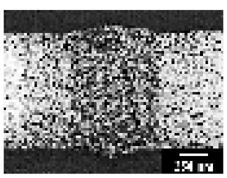
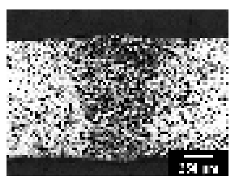
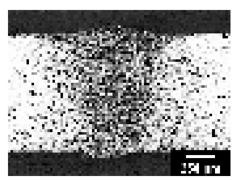
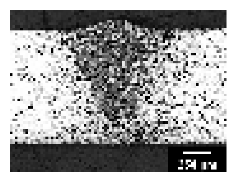
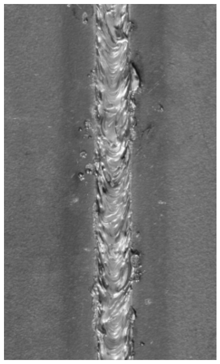
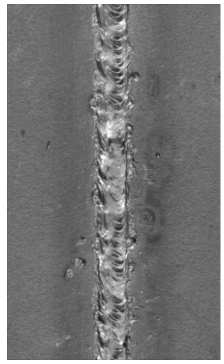
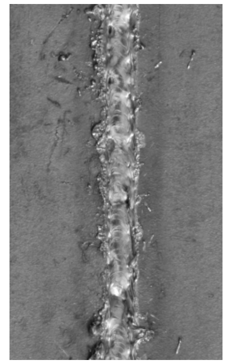
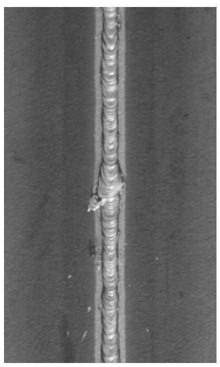
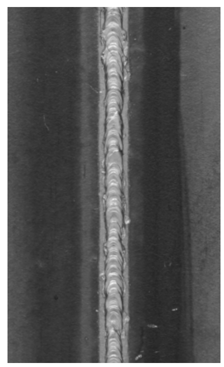
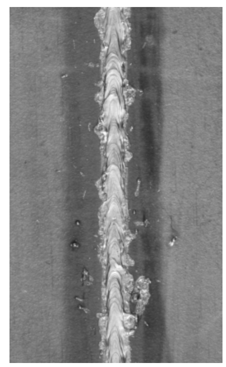
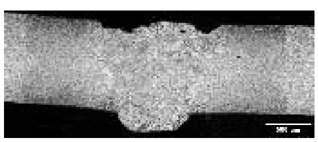
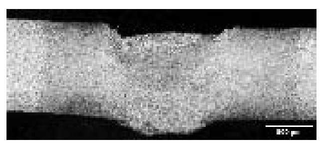
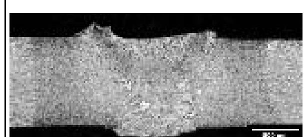
	Welding speed [mm/s]			
	30	40	50	60
Weld surface				
Weld root				
Cross-section				

Table 6. Surface, root and cross section appearance of electron beam welds

Sample No.	**503**	**502**	**606**
No of passes	**1**	**3**	**3**
Weld surface			
Weld root			
Cross-section			

A close up of EB weld macrostructure is shown in Figure 5.

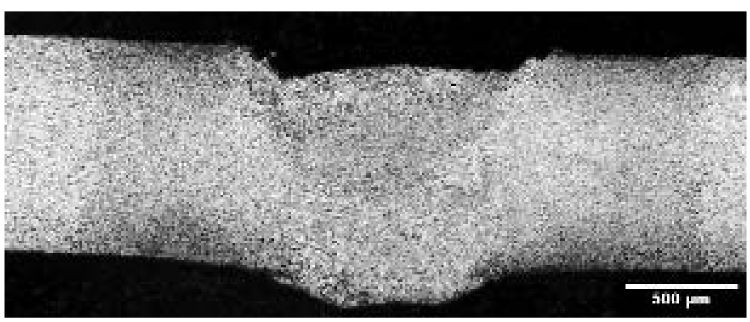


Figure 5. Macrostructure of the three pass weld

The microstructure of LBW joint (welding speed of 40mm/s) is shown on Figure 6 where particular areas of weld joint can be seen – parent material, HAZ and weld metal.

Figure 6. Microstructure of laser weld joint (weld metal- HAZ- parent material) of materials treated by nitrooxidation.

Microhardness measurements of laser beam welds are shown in Figure 7 and Figure 8.

Table 7 sumarizes average value of microhardness in relevant section of the electron beam joint, Figure 9 shows microhardness progression through this weld joint.

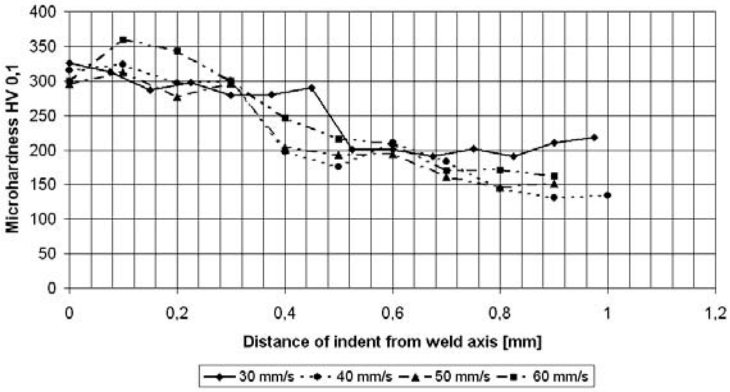


Figure 7. Microhardness as a dependence on distance of weld axis in upper part of weld joint.

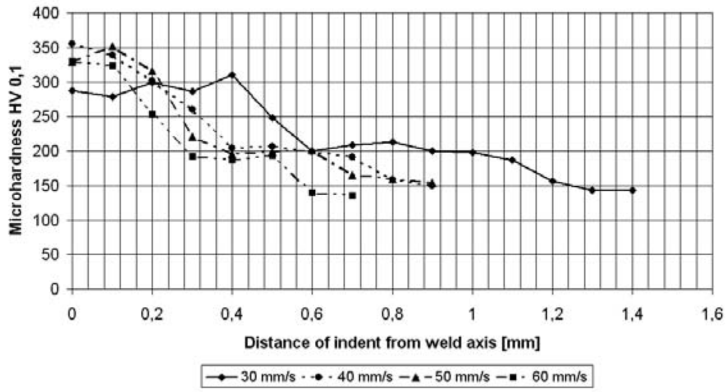


Figure 8. Microhardness as a dependence on distance of weld axis in root part of weld joint.

Table 7. Approximate values of microhardness of electron beam weld

	Surface	Middle	Root
Weld metal	152.7	159.1	185.7
Heat affected zone	161.3	134.5	181.6
Base material	154.8	124.8	180.2

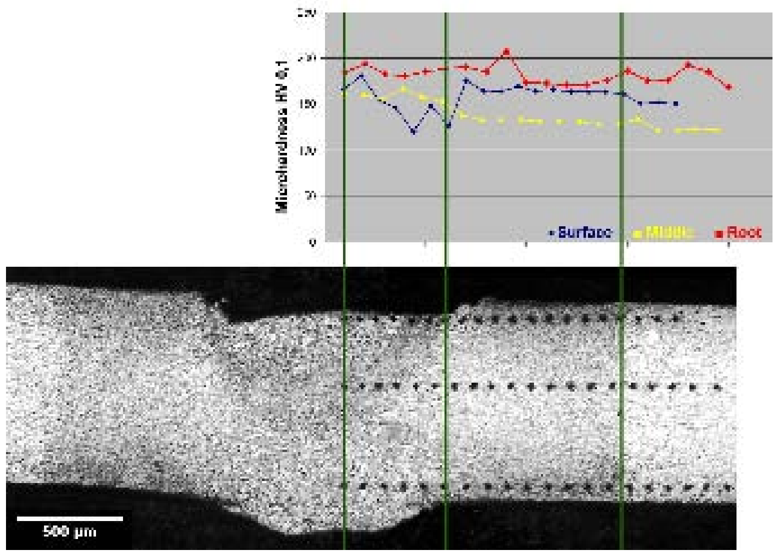


Figure 9. Microhardness measurements of three pass electron beam weld.

DISCUSSION

Visual inspection of laser beam welds proved incomplete penetration at welding speed of 60 mm/s. Other welding speeds had full penetration with lack of defects and regularly formed weld. Comparing the HAZ width, the welding speed of 30mm/s had the greatest heat affection. From cross-section point of view, the welding speed of 40 and 50 mm/s look similar, but weld root surface pattern in 50mm/s welding speed showed some irregularities. Because of this, the recommended optimal welding speed for thickness of 1 mm should be between 40 and 50 mm/s.[4]

On the other hand, electron beam welds had problems with weld bead surface regularity and excessive spatter.[6] However, the multipass welding mode has positive influence on weld bead surface regularity (compare cross sections of samples 503 and 502 in Table 6). Shape of the weld cross sections was characterised by a surface concavity presented on all welds. In direct comparison of laser and electron beam welds (Table 5 vs Table 6

Table 6), there was obvious difference in weld bead dimensions and weld cross section shape, amount of spatter and weld metal porosity for the benefit of laser beam welding.

The width of the weld made by electron beam welding was double in comparison to laser beam welding, the width of heat affected zone was even triple. This was probably caused by lower cooling speed in vacuum during electron beam welding.

The laser beam welding caused the nitride dissolvation up to distance of 1 mm from the weld joint boundary. The microstructure consisted of acicular ferrite and ferrite precipitated along boundaries of columnar crystals.

The structure has not showed any abnormalities in phase composition, which could be expected due to existing nitrides in surface layers of materials welded after their nitrooxidation.

Microstructure of high tempetarure HAZ consisted of polyedric ferrite. The undesirable grain coarsing was also not observed in this part of weld joint.

Based on microstructural analysis there can be assumed that existing nitrides prevent grain coarsing in the HAZ area heated over temperature A_3.

Microstructure of parent material which had partially deformation textute consisted of polyedric ferrite, tercial cementite precipitated at the grain boundaries.

Electron beam weld microstructure depended on counts of welding passes. The microstructure of single pass welds consisting of polyedric ferrite had coarser grain structure (Table 6, cross-section of sample 503).

There was also observed presence of upper bainite. Microstructure of welds made by three passes (Table 6, cross-section of sample 502, 606, Figure 5) was different, as every subsequent pass has influence on microstructure of the previous pass. The microstructure of upper part was formed by coarse accicular ferrite and polyedric ferrite. There were also sporadically observed grains of upper bainite. The ferritic matrix had secondary precipitated carbides and nitrides. Structure of weld root was very similar to the upper part. Significant difference in weld microstructure was observed in the middle part of the weld.

This was caused by the application of the third welding pass resulting to annealing of the previous, i. e. second pass. The microstructure of this weld area consisted of fine polyedric ferrite with a slight heterogenity of grain size.

On the contrary, the microstructure of the high temperature heat affected zone in upper, middle and root part of weld joint was very similar. It consisted of polyedric ferrite with fine and almost equal grain size. There was not observed any grain coarsing in this part of the weld.

The microstructure of heat affected zone to base metal transition for all three monitored weld parts consisted of polyedric ferrite with precipitated nitrides in ferritic matrix and grain boundary. There was a significant difference in grain size of heat affected zone and base metal structure.

Microhardness measurements of laser beam welds showed maximal hardness of 360 HV 0.1 in weld metal of specimen welded by 60mm/s welding speed. This specimen also presented the highest microhardness variances, probably due to the highest welding speed and thus the highest cooling rate. This estimation can be supported by microhardness measurements of specimen welded by lowest welding speed (30 mm/s) which had also the lowest microhardness variance due to the lowest cooling rate. The differences between microhardnesses in upper and root part of weld joints weren't significant.

The microhardness measurements of electron beam welds showed the smallest microhardness of 120 HV 0.1 near sample surface and a bit higher microhardness

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(172.6 HV 0.1) in heat affected zone. Next microhardness drop to approximately 150 HV 0.1 was observed in base material near the surface. Middle section of electron beam weld has shown stable microhardness of approximately 157 HV 0.1 decreasing continuously through the heat affected zone up to the base material to approx. 122 HV 0.1. The highest microhardness values of approximately 180 HV 0.1 were observed in the root section of the weld metal. The reason of microhardness fluctuations were probably caused by the presence of upper bainite in the microstructure and probable placement of indent into this phase.

CONCLUSIONS

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

Concerning the multiphase composition of sheets after their nitrooxidation and especially presence of three types of nitrides: $Fe_{2-3}N$ (ε - phase), Fe_4N (γ´ - phase) a $Fe_{16}N_2$ (α´´- phase) and oxidic phases (Fe_3O_4, Fe_2O_3), there has not been observed any undesireable demonstration of microstructural change in analysed weld joints. Analysed microstructures of weld joint areas had typical character corresponding to welding of structural steels.

Some problems have occurred during GMAW and spot resistance welding due to surface oxidic layer.[5] On the other hand, laser beam welding seems to be suitable welding technology.

The presence of secondary precipitated nitrides and dissolvation of nitridic phases due to welding process had positive influence on microstructure of high temperature HAZ. There was no grain coarsing in this part of weld joint being otherwise typical for this area and therefore had positive influence on weld joint strength increase.

The structure did not show any abnormalities in phase composition, which could be expected due to existing nitrides in surface layers of materials welded after their nitrooxidation.

Microhardness of the specimen with optimal welding speed (40 mm/s) reached maximum value of 325 HV 0.1 in weld metal near sheet surface and 355 HV 0.1 in root part of weld joint respectively. The heat affected zone showed further microhardness drop to approximately 200 HV 0.1 while typical microhardness of base material was roughly 150 HV 0.1.

It was shown that nitrooxidation had a beneficial influence on formability as well as corrosion resistance. Having proved the good mechanical properties of weld joints which are at a moment subject of further research there can be assumed that the weight of welded products could be decreased, with preserving required formability and also improved lifetime.

The results of electron beam welding of nitrooxidation treated steel sheets were inconsistent.

On one hand, the microhardness measurements allow to expect good plastic behaviour of the weld metal and heat affected zone. There was also not observed any grain coarsing of high temperature heat affected zone.

On the other hand, electron beam welding was more sensitive to precise adjustment of welding parameters and because of the surface oxidic layer being deteriorated in vacuum much more prone to spatter and weld metal porosity.

Concerning comparison to laser beam welding the width of heat affected zone was three times wider, the weld width was double. This widening was probably caused by lower cooling speed due to welding in vacuum. Laser beam welds were spatter free and had more suitable weld shape in cross-section. Contrary to the electron beam welding, the laser welds did not exhibit the concavity of weld surface.

As there was not proved decrease of heat affected zone width which is important for minimising deterioration of surface nitrooxidation layer and thus keeping the corrosion resistance as low as possible, it is not recommended to use electron beam welding for this way treated materials. Based on promising results from CO_2 laser beam welding there will be interesting to investigate the possibilities of using solid state lasers for nitrooxidation treated steel sheets.

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