SCIENTIFIC PAPERS
OF THE UNIVERSITY OF PARDUBICE
Series B
The Jan Perner Transport Faculty
18 (2012)

MATERIAL ANALYSES OF HIGH STRENGTH STEELS FOR AUTOMOTIVE SAFETY PARTS

Eva SCHMIDOVÁ

Department of Mechanics, Materials and Machine Parts

1. INTRODUCTION

Energy save and reduction of the environment load are important tasks of automotive industry together with increasing of passive safety. High stress steel enables weight reduction simultaneously with increasing of safety.

Hot stamping is a widely used method of preparation of high-strength steel car components. The material is formed in a thermo-mechanical process during anizothermal cooling from the austenitization temperature into the specified shape and reaches a fully martensitic structure with strength higher than 1500MPa [1, 2]. The process enables production of stampings with reduced springback, with a great geometric accuracy and a high strength-to-mass ratio. The sections produced in this way are used in crash protection applications, mainly bumper beams, door reinforcements and B-pillars. Current research in this field is oriented around heat treatment with controlled cooling. Reaching an optimal combination of martensite and residual austenite leads to a desired increase of fracture toughness and energy absorption capability at high loading speeds [3, 4].

Usage of precoated steels in hot stamping is one of the methods that improve the quality of high-strength stampings. The role of the coating is protection of the surface against iron scales and decarburisation during hot forming and quenching in the die. A 30-40µm layer based on Al-Si (90% Al) is preferred for martensitic low-alloyed steels. The benefit of a coating based on Al-Si is higher oxidation resistance at high temperature [5].

The structural changes in the surface layer produced by the thermal process reduce re-melting during welding, contributing to the preservation of the passivation effect. In addition, experiments have demonstrated a tendency to creation of a protective layer on the electrodes that increases their durability [6]. At the same time, however, a tendency for the layer to melt into the weld metal has been observed, for example in laser-welded joints. A possible consequence is the creation of brittle inter-metallic phases based on Al-Fe and reduction of joint strength [7,8, 9]. During laser welding of hot forming steel with an Al-Si coating, the coating is diluted into the weld zone, which results in Fe-Al intermetallic phase formation [10, 11].

The presented study focuses on an analysis of the microstructure and surface layer of low-alloy martensitic steel and detection of potential negative impacts on weldability. It specifies material parameters that present a source of dispersion of static and dynamic strength of joints within a standard manufacturing process of high-strength components of the bodies.

2. METHODOLOGY OF ANALYSES

The experiments concerned various meltages of martensitic steels 22MnB5 in a condition for hot stamping. The study of the material's metallurgical weldability was based on an analysis of internal homogeneity and micro-cleanness of the material. The evaluated effect of the surface layer on the weldability was based on the (i) an analysis of the chemical composition of individual sub-layers in crosswise scratch patterns; (ii) evaluation of differences in morphology and chemical composition directly on the surface layer of the stampings. The structural analyses were performed by light and electron microscopy (SE), the chemical composition was analysed by an energy microanalysis method (EDX analysis).

Samples for the structural analyses were taken from areas corresponding to evaluation of the surface layers. All the evaluated stampings showed a martensitic structure with an approximately identical share of bainite and partially tempered martensite.

Study of weldability is based on evaluation of:

i. material response at different levels of thermal interference,
ii. static endurance of the weld joints.

There were carried out structural and phase analyses of the initial quality of the high stressed steel and surface layer, structural changes within the gasp of thermal and voltage cycle of the used technology of the welding. With regard to fundamental demand on fracture goings of the steel and weld joint there were made fractographical analyses. These analyses were made for identification of preponderant fracture tendencies of basic material, weld metal and heat affected zone. In presented selection of results there is mentioned especially dependence of achieved weld endurance on a process of a fracture.

3.STUDY OF FRACTURE BEHAVIOR

There was done fractography analysis of selected parts of profiles to evaluate:

- a fracture mode or local differences in case of inside material imperfection,
- defective part of fracture tracts in connection with structural characteristics.

Ductile fracture was identified as a preferential for martensitic steel and welding joints even at presence of internal defects (Fig.1, 2).

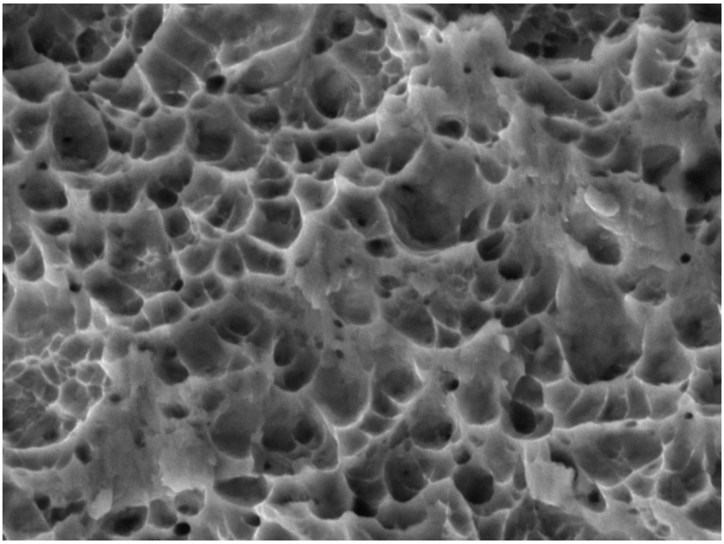

Figure 1 Ductile fracture mode

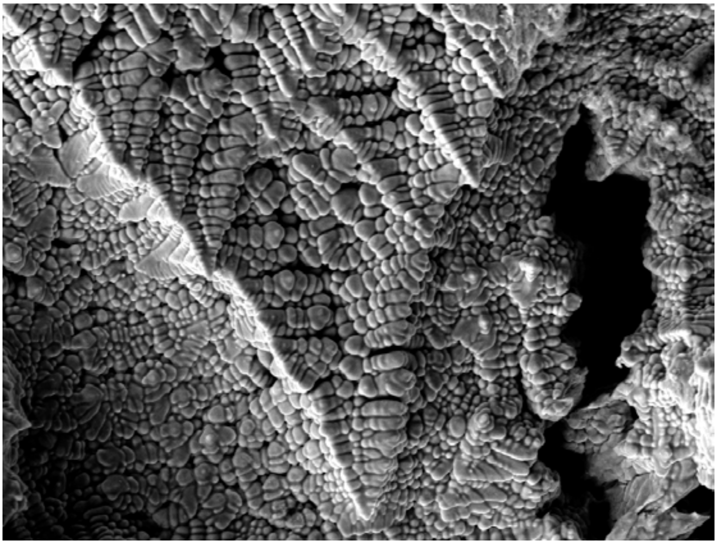

Figure 2 Defect of welding joint

Important influence on the energy consumption during destruction of spot weldings is a tendency of fracture initiation. Three different tendency of fracture were discovered:

a) over boundary weld metal
   This type of infringement was connected with lower level of reached strength of weld joints.
b) cross the weld metal, especially at higher presence of internal defects.
c) along the softened zone, connected with maximum strength and absorbed energy.

A critical effect on the dynamic strength brings a tendency of melting surface material into the weld metal and creation of brittle inter-metallic phases based on Al-Fe. A tendency to the brittle fracture mode instead of a ductile fracture was observed – Fig.3.

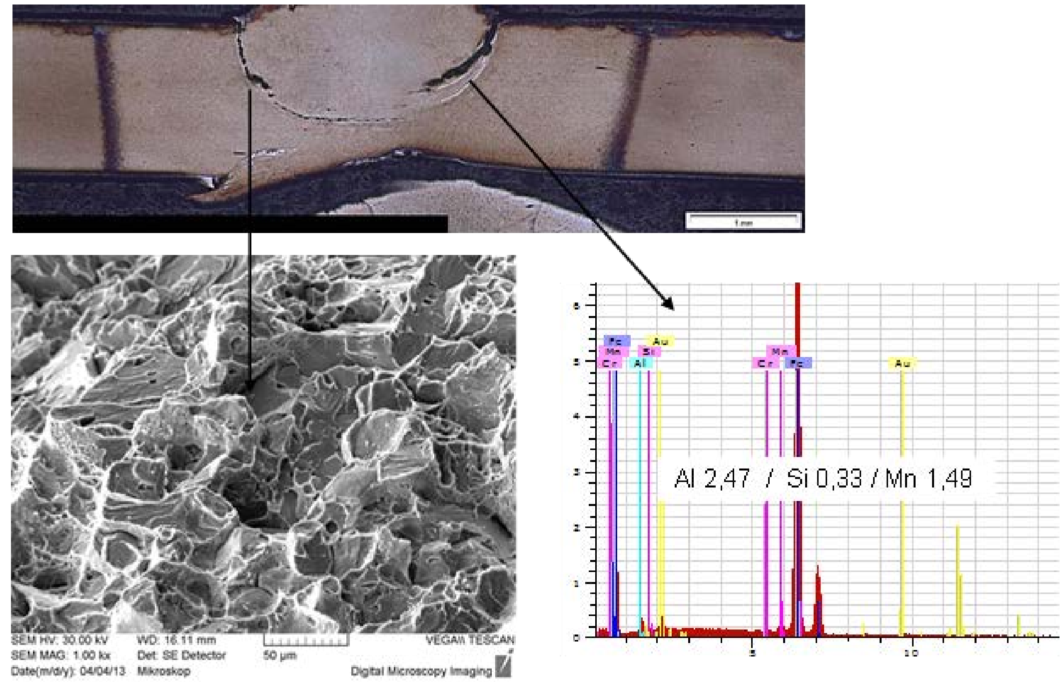

Figure 3 Brittle fracture mode due to presence of intermetallic phases

4. ANALYSES OF SURFACE LAYERS

An example of a typical composition of the surface layer is presented in Fig. 4, 5. The thickness of metallographically distinguishable sub-layers was measured on etched crosswise scratch patterns (positions 1 to 4 in Fig. 5).

A difference of the diffuse - inter-metallic layer thickness was detected (in the measured parameter No. 2 in Fig. 5). In addition, a different structure of heterogeneities in the volume of the coating was observed in these stampings. Unlike the remaining set of stampings with typically scattered isolated volumes of these heterogeneities, in stampings with higher intermetallic layer, another continuous "inter-layer" was formed in the volume of the coating. The analyses of local differences in the chemical composition focused on these areas.

Figure 4 Tested stamping

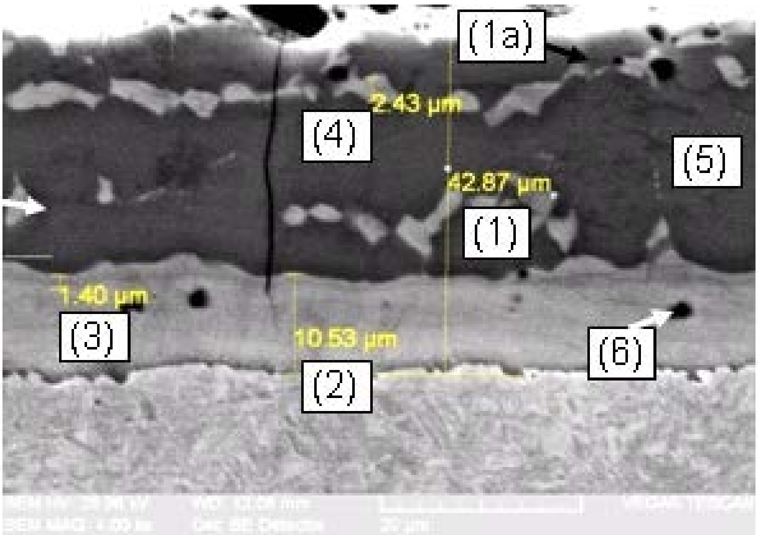

Figure 5 Typical structure of surface layer

The chemical composition of the sub-layers was evaluated on scratch patterns. The positions of individual measurements correspond to measurements of the sub-layer thicknesses and is presented in Fig. 5: (1a) immediate surface layer; (2) diffusion layer bordering martensitic steel; (3) border between the diffusion layer and the remaining volume of the coating; (4) heterogeneities in the coating beyond the area of the diffusion layer; (5) homogenous area of the coating beyond the diffusion layer; (6) surroundings of hollows in the diffusion layer.

Changes in concentration of essential elements (Al-Fe-Si) in the sub-layers of the coat in a line perpendicular to the surface are presented in Fig. 6. Proportions of Si-Fe-Mn elements in individual sub-layers were evaluated in succession to the detected geometric differences in the structure of the coating in the tested series of the stampings. Differences of Al/Si in the individual sub-layers are considered particularly important as regards the evaluated effect on the weldability. The results showed a twofold increase of Al/Si proportion in the basic volume of the layer in stampings, where the deviation in the internal structure of the surface layer was detected. The non-compactness of the immediate surface layer (the 1a area in the mentioned measurement) did not allow a definite evaluation of the chemical composition in the crosswise scratch patterns. For this reason, the measurement was supplemented with a direct measurement from the surface. The measurement showed again an increase of the Al/Si proportion in stampings with identified deviation in the thickness of the inter-metallic layer.

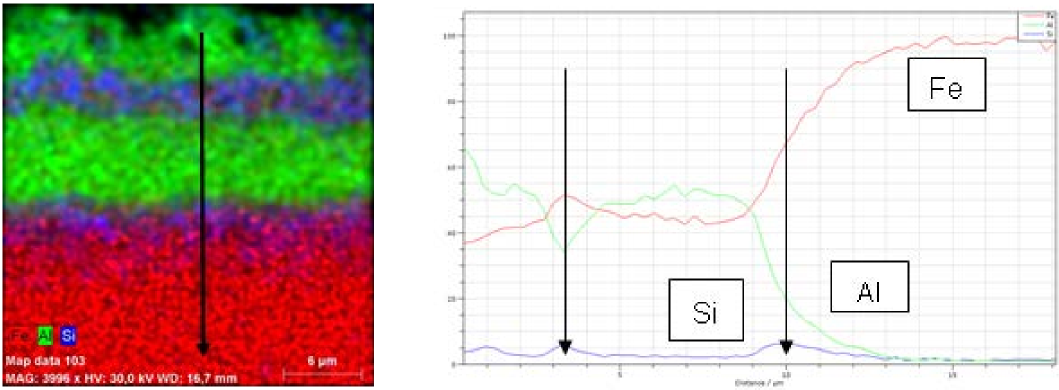

Figure 6 Different structure of surface layer after intensive Al-Si-Fe redistribution

Current results of the monitored relations are given in Fig. 7. It is obvious that the mentioned twofold increase of Al/Si proportion on the surface of the stamping and simultaneously in the basic volume of the coating occurs at the otherwise stabile Al/Si proportion in layers bordering the basic material (layers 2, 3). This redistribution was thus produced by an increase of an inter-metallic layer and, at a certain stage, by the formation of a new inter-layer in the volume of the coat with an approximately identical Fe/Al/Si proportion. The increased Al/Si proportion on the surface of the stampings coincides with the measured parallel decrease of Al in the formed secondary inter-layer. According to the measurement, the increased thickness of the inter-metallic layer was accompanied with increased Fe concentration in the secondary inter-layer.

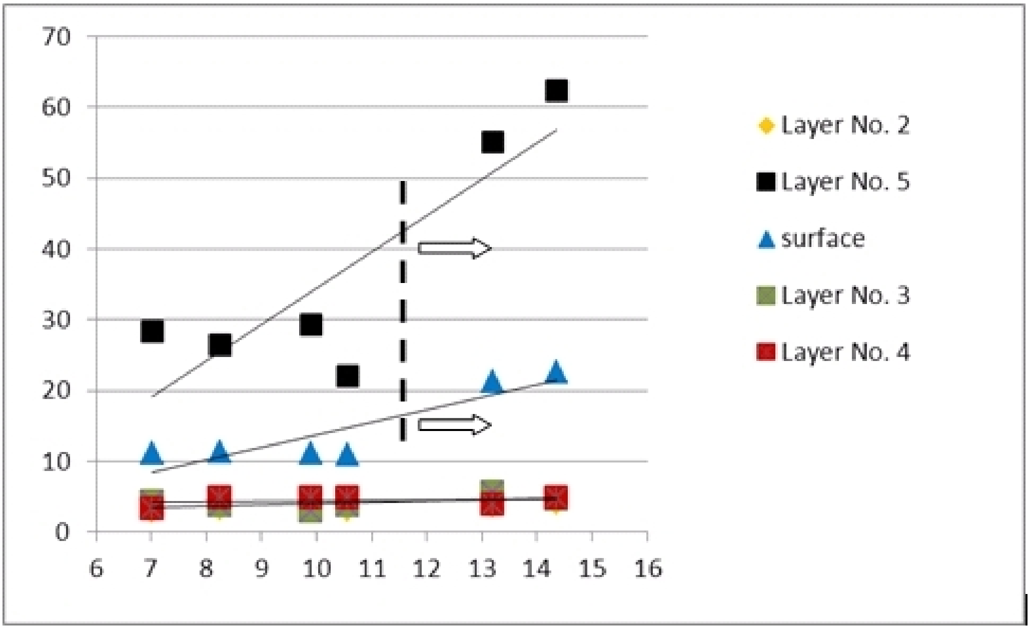

Figure 7 Change of the Al/Si proportion in relation with the thickness of the inter-metallic border of the coating

The mutual interconnection of the detected deviations in the internal composition and the Al/Si proportion manifest the dispersion of the performed heat treatment, particularly the differences in temperatures and the heating period during austenitization. This fact leads to differences in intensity of the diffusing processes that lead to the observed differences in the layers' internal structure. The source of the undesired dispersion of weldability particularly comes from a tendency to form a secondary inter-layer with an increased Fe and Si content. The measurement results suggest that these secondary layers are formed in combination with a thickness of an inter-metallic layer exceeding ca 13µm.

The marked border in Fig. 7 corresponds to the detected limit thickness of the inter-metallic layer. Exceeding this limit leads to forming of another "secondary" layer enriched with Fe, thus an undesired effect on the weldability. A layer of oxide on the surface of the stampings increases with the growing heating period of austenitization. The changes of thickness in the layer of oxide on the surface of the stampings produce different surface colouring caused by interference. Stamped sections with detected diffusion redistribution of the monitored elements in the formation stage of a new "secondary" layer enriched with Si and Fe showed a certain distinction in the surface colouring within the tested series of stampings (difference in the blue-grey colouring intensity). The undesirable changes of the surface layer's internal structure can thus be to some extent indicated by a change of surface colouring.

5. EXPERIMENTAL WELDING

An experimental series of 25 spot welding joints was produced to evaluate the effect of the detected diffusion processes on the weldability. The experiment was based on a comparative evaluation of static strength and fracture behavior of the welded joints, combining materials with extremes of the detected differences in the surface layer's structure. The selection of the welding parameters was based on a preceding optimization of the technological welding process. The detected difference of static strength was 22kN (in joints combining materials with a minimum thickness of the inter-metallic layer) vs. 17.6 kN (in joints of materials with the maximum thickness of the inter-metallic layer).

The curve of the fracture decisively affected the strength of the resistance spot-welds. Higher strength was achieved in a situation where the fracture was initiated in a zone of tempered martensite. Lower strength was connected with initiation of the fracture on the fusion line. The welding thermal cycle in this particular type of martensitic steel causes a local softening of the basic material. The position and extent of this critical zone decisively affects the fracture behaviour and strength of the spot welds. The detected differences in the surface layer's structure affect the current flow during the spot welding. With a view to the stable characteristics of the remaining parameters in the tested series (the chemical composition, the micro-structure, micro-cleanliness, etc.), it is possible to connect the differences in the reach and intensity of the thermal impact during welding with the detected deviations of the coating.

Evaluation of the samples was carried out on the basis of differences reached by thermal effects, which is affected by the interlayer diffusion, and together with a current flow.

To assess the influence on the range of thermal effects there was evaluated:

1) the distance of the fusion zone

2) the distance of the softening zone

From materials with the same quality of the surface layer there were selected representative samples to be spot-welded. After the static test, the scratch pattern was created and subsequently also the micro hardness was created for determination of the heat affected zone.

The comparison showed that the differences in the diffusion zone did not produce measurable differences in regard of the positions of those layers. A comparison of the intensity of thermal effect on the material showed no difference in level of microhardness, but a measurable effect on the wide of tempered zone was observed. There was also found a tendency of precipitation of carbides along austenite grain boundaries. But in the reviewed cases, this trend did not lead to any negative effect on fracture behavior. It was found forming ductile fracture mode, no intergranular fracture.

6. CONCLUSIONS, AIM OF THE NEXT RESEARCH

The presented results led to detection of basic tendencies in the formation of the internal structure of the surface layer in connection with deviations of the introduced heat treatment of martensitic steel 22MnB5. Coating by AlSi is applied during the heating phase, i.e. during heating 5-10 min at 880-950°C. The heat treatment leads to modification of the surface layer and at the same time diffusion of iron into the surface layer and surface oxidation of the layer.

The heat treatment creates a layered sub-structure with uneven distribution of Al-Si-Fe. The performed analyses document diffusion redistribution of Al-Si-Fe elements in the surface layer in connection with changes in the geometry of the layer's internal structure. Increase of the temperature and the heating period leads to an increase of the diffusion layer's thickness and diffusion of Fe into the AlSi coating. Together with the rising Fe/Si, Fe/Al proportion, the volume of heterogeneities enriched with iron increases and the share of Al/Si rises in the immediate surface zone. Forming of a continuous "secondary" inter-metallic inter-layer in the volume of the coating with prevailing Al is particularly important for its impact on weldability. The share of Fe/Si, Fe/Al elements, i.e. the intensity of the impact on the current flow during welding, rises with the austenitization period in this secondary layer.

At the same time, the Al/Si proportion on the surface of the stampings rises, the surface oxidation becomes more intensive and porosity of the immediate surface layer increases. The rise of porosity and the content of oxides on the surface contribute to the instability of the welding process. A higher amount of pores leads to their random collapse, thus changing the current flow. The higher layer of oxides on the surface of the stampings affects the current flow.

The study of the crucial tendencies in diffusion re-distribution of the Al-Fe-Si components in connection with formation of undesirable "inter-layers" was based on an evaluation of local concentrations of these elements in relation to the thickness of the inter-metallic layer. Based on the performed experiments, the maximum limit thickness of the inter-metallic layer has been specified as 13 µm as the limit stage of these changes with a view to the researched impact on the spot welds' quality. Exceeding of this value by dispersion of the austenitization period causes instability of the welding process in the random combinations of the weldments.

The experimental evaluation of the weldability revealed the differences in softened zone. The formation of wider zone of tempered martensite was accompanied by the intergranular carbide precipitation. Regarding the fracture behaviour, the harmful effect on the fracture toughness can be estimated mainly in the case of more intensive precipitation. Limited harmful influence was proved in connection with presence of Al-Si intermetallic phases inside the welding pool. The main goal of next research is evaluation of detected processes on the dynamic resistance of spot weldings. We are supposed to perform the estimation of the significant possibilities to improve the dynamic durability of martensitic high strength steel in mentioned applications.

Předloženo: 31. 5. 2013

References

[1] Liu, H.S., Xing Z.W, Bao J., Song B.,Y. Investigation of the Hot-Stamping Process for Advanced High-Strength Steel Sheet by Numerical Simulation. *J Mater Eng Perform,* 2010, 19:325-34.

[2] Heping, L., Xuejun, J., Han D., Jie, S. Martensitic Microstructural Transformations from the Hot Stamping, Quenching and Partitioning Process. *Materials characterization*, 2011, 62:23-227

[3] Bardelcik, A., Salisbury, C.P., Winkler, S., Wells, M.A., Worswick, M.J. Effect of Cooling Rate on the High Strain Rate Properties of Boron steel. *Int J Impact Eng*, 2010, 37:694-702.

[4] Dancette, S., Fabregue, D., Massardier, V., Merlin, J., Dupuy, T., Bouzekri, M. Experimental and Modeling Investigation of the Failure Resistance of Advanced High Strength Steels Spot Welds, *Engineering Fracture Mechanics*, 78, 2011, 2259-2272.

[5] Karbasian, H., Tekkaya, A.E. A review of hot stamping. *J Mater Process Technol*, 2010, 210:2103-2118.

[6] Maggi, S., Murgia, M. Introduction to the Metallurgic Characteristics of Advanced High-Strength Steels for Automobile Applications. *Weld Int*, 2007, 22:610-618.

[7] Ehling, W., Cretteur, L., Pic, A., Vierstraete, R., Yin, Q. Development of a Laser Decoating Process for Fully Functional Al-Si Coated Press Hardened Steel Laser Welded Blank Solutions. *In 5th International WLT-conference on Lasers in Manufacturing*. Munich, Germany, 2009, p.409-413.

[8] Choi, J.K., Kim, J.K., Shin, S.M., Kim, C.H., Rhee, S.H. A study on Laser Welding Characteristics of 1500MPa Grade Ultra High Strength Steel for Automotive Application. *J. Korean Soc Laser process*, 2010, 13:19-26.

[9] Kim, C., Kang, M.J., Park, Y.D. Laser Welding of Al-Si Coated Hot Stamping Steel, *Procedia Engineering*, 2011, 10:2226-2231.

[10] Ehling W, Cretteur L, Pic A, Vierstraete R, Yin Q. Development of a Laser Decoating Process for Fully Functional Al-Si Coated Press Hardened Steel Laser Welded Blank Solutions. *Proceedings of 5th International WLT-conference on Lasers in Manufacturing*. Munich, Germany; 2009, p.409-413.

[11] Choi JK, Kim JK, Shin SM, Kim CH, Rhee SH. A study on laser welding characteristics of 1500MPa grade ultra high strength steel for automotive application. *J Korean Soc Laser Process* 2010;13:19-26.

[12] Fan DW, Kim HS, Oh JK, Chin KG, De Cooman BC. Coating degradation in hot press forming. *ISIJ Int* 2010;50:561-568.

Resumé

MATERIAL ANALYSES OF HIGH STRENGTH STEELS FOR AUTOMOTIVE SAFETY PARTS

Eva SCHMIDOVÁ

Vysokopevné martenzitické oceli po lisování za tepla jsou široce využívány pro bezpečnostní prvky automobilů, které se vyznačují vysokým poměrem pevnosti k hmotnosti a vysokou rázovou houževnatostí. Odolnost jednotlivých součástí je určena dynamickou pevností svarových spojů. Cílem této práce je analýza svařitelnosti těchto ocelí a jejich degradace v důsledku svařování. Hlavním problémem se ukazuje strukturní nehomogenita a difuze prvků z povlaku na bázi AlSi. V práci jsou předloženy výsledky analýz, kde je ukázána fázová přeměna během svařování a změna lomového chování v tepelně ovlivněné oblasti. Jako hlavní nepříznivý efekt je zjištěna redistribuce Al a Fe. Na základě toho byly navrženy vhodné prarametry povlaku. Vliv austenitizace byl odhadnut na základě reálných vzorků a ověřen na experimetálních svarech.

Summary

MATERIAL ANALYSES OF HIGH STRENGTH STEELS FOR AUTOMOTIVE SAFETY PARTS

Eva SCHMIDOVÁ

The martensitic high strength steels after hot stamping are currently widely used for safety automotive components characterized by a high strength-to-weight ratio and an increased resistance to impact. The durability of each parts is determined by dynamic strength of welded joints. The objective of presented analyses was study of weldability of martensitic steels and relevant degradation processes. The structural heterogeneity and diffusion processes in the AlSi coating have proved as a significant problem. The paper presents a selected results of revealed parameters, which influenced the phase transformation during welding and fracture behavior of heat affected zone. In order to avoid the identified harmful effect of redistribution of Al and Fe, the limited acceptable parameters of coating was proposed. The influence of austenitization process was estimated and the real intensity of discussed effects was evaluated by experimental welding.

Zusammenfassung

MATERIAL ANALYSES VON HOCHFESTEN STÄHLEN FÜR SICHERHEIT IM AUTOMOBILBEREICH PARTS

Eva SCHMIDOVÁ

Die martensitische hochfeste Stähle nach der Warmumformung werden derzeit häufig für Sicherheit Automobilkomponenten durch eine hohe Festigkeit-zu-Gewicht-Verhältnis und eine erhöhte Schlagfestigkeit gekennzeichnet werden. Die Haltbarkeit der einzelnen Teile wird durch dynamische Festigkeit der Verbindung bestimmt. Das Ziel der vorgestellten Analysen war Studie Schweißbarkeit martensitische Stähle und relevanten Abbauprozesse. Die strukturelle Heterogenität und Diffusionsprozesse in der AlSi-Beschichtung haben als wesentliches Problem erwiesen. Der Beitrag stellt eine ausgewählte Ergebnisse offenbarten Parameter, die die Phasenumwandlung beim Schweißen und Bruchverhalten Wärmeeinflusszone beeinflusst. Um die identifizierten schädliche Wirkung der Umverteilung von Al und Fe zu vermeiden, wurde die begrenzten akzeptabler Parameter der Beschichtung vorgeschlagen. Der Einfluss der Austenitisieren Prozess wurde geschätzt, und die wirkliche Intensität der Effekte diskutiert wurde durch experimentelle Schweißen ausgewertet.