

INFLUENCE OF ANNEALING ON MICROSTRUCTURE AND PROPERTIES OF COLD ROLLED SHEET FROM HIGH MN-AL STEEL TYPED

KUC Dariusz¹, WOŹNIAK Dariusz², BEDNARCZYK Iwona¹

¹Silesian University of Technology, Katowice, Poland, EU, <u>dariusz.kuc@polsl.pl</u> ²Institute for Ferrous Metallurgy, Gliwice Poland, Poland, EU

Abstract

The aim of the paper was to determine the parameters of recrystallization annealing for cold-rolled sheets prepared from steel X60MnAl30-9. Tested steel is characterized with high resistance and ductility but due to the addition of 9%Al it meets the criterion of the so-called "light steel". Microstructure and properties of metal sheets are mainly dependent on temperature and time of annealing which in continuous processes should be as short as possible. Tests of microstructure and mechanical properties for given parameters enabled the choice of the optimum parameters of the process to achieve the best possible relation of the resistance properties.

1. INTRODUCTION

Application of new, highly-resistant steels with high formability in automotive industry leads to substantial decrease of vehicle weight [1]. At the same time we can observe improvement of safety during crash thanks to high absorption of energy which the elements made of such steel feature. High-manganese austenitic steel, in which research centres are currently interested, is characterised by extremely high formability and substantial strength. Capability of energy absorption is also much bigger in this case in comparison with conventional steels. Such a set of features can be explained by the presence of alternative strain mechanisms, such as: creation of twins (TWIP effect) [2-6], phase transitions produced by strain (TRIP) and plasticity induced by shear bands [7, 9]. Optimum content of manganese in TWIP steels is ca. 20-35% by mass and the content of coal and other elements in total does not exceed 0.003 - 0.6% by mass. Wide application of TWIP steels is hindered by difficulties connected with their production and processing. Development of this group of steels, implementation to large-scale production and application as structural material is conditioned by improvement of their plasticity in room temperature and hot processing. By proper selection of chemical composition, modification of initial microstructure, grain refinement and application of suitable thermal and plastic working, it is possible to obtain optimum connection of mechanical and plastic properties. Moreover, the procedure of materials selection for automotive industry requires tests of mechanical properties as a function of strain rate, because at high strain velocity, typical for car crash, mechanical strength is substantially changed [1]. Currently at the Silesian Technical University tests of new steel with high content of manganese and aluminium, indicated as X60MnAl30-9 and designed for constructional elements for automotive industry, are carried out [9]. This article presents the results of tests of microstructure and mechanical properties of sheet from Mn-AI steel with duplex austenitic-ferritic microstructure after clod working. Presented results are crucial for elaboration of production technology of this group of steel.

2. EXPERIMENTAL PROCEDURE

The material tested is the high-manganese steel X60MnAl30-9 The steel fabrication technology was developed by smelting in the inductive vacuum furnace VSG 100S manufactured by PVA TePla AG. This type of furnace makes it possible to obtain favorable conditions for obtaining the required composition and high purity steel. Casting took place under an atmosphere of argon, using an intermediate ladle that had been heated to a water-



cooled copper catalyst. The catalyst has a square cross section measuring 100 x 100 mm and a height of 1100 mm.

The next stage was rolling the ingots in Institute for Ferrous Metallurgy, it took place in several stages. Initially, the charge was hot rolled into square sections of 33 mm, the next step was to obtain flat bars of 20 mm thickness, then the material was rolled to a thickness of 12 mm, in the next step the thickness was reduced to 6mm, the next step was to achieve thickness of 3mm and finally flat bars 2 mm. The temperature of the batch heating and steel rolling was chosen by taking into account the plastic properties, the chemical composition, and the final product requirements, and for heating 1180 °C and rolling 1150 °C. Rolling was done at a speed of 1 m / s. Finally, the element underwent cold rolling to a thickness of 1mm. Rolling underwent on 4 passes with the following reduction of thickness: 1.96 mm \rightarrow 1.56 mm \rightarrow 1.29 mm \rightarrow 1.06 mm. Annealing was conducted with furnace Carbolite EAF 11/6 in temperature range 700-1000 °C with holding time of 15 and 30 minutes.

There was a static tensile test performed after rolling of the rods. The rods after rolling were used to prepare samples with round cross-section to test the mechanical properties in accordance with norm PN-EN ISO 6892-1. Tests were conducted on testing machine Zwick/Roell Z100. On the basis of test the following parameters were marked: Ultimate tensile strength (UTS), Yield point ($Rp_{0.2}$), elongation (A5). Hardness measurement was conducted with the use of hardness tester Zwick type 3212002/00. Metallographic test was conducted on light microscope type Olympus GX51 with magnification in range 200÷1000×. In order to conduct quantitative analysis of pearlite areas the images were registered on scanning microscope with magnification up to 15000×. SEM analysis were carry out on scanning microscope Hitachi S-3400N.

3. RESULTS AND ITS DISCUSSION

After cold rolling both the austenite grains and the ferrite bands undergo elongation towards the direction of rolling as presented in **Figure 1a**. In the next stage the material went through the recrystallisation annealing in temperature range of 750 - 1000 °C with holding time of 15 and 30 minutes. After annealing in temperature of 750 °C the division of ferrite bands occurs. After annealing for 15 minutes there were no processes of recrystallization occurring inside the area of austenite grains, which is shown in **Figure 1b**. Not until 30 minutes passed was it visible that partially recrystallized austenite grains are present, as shown in **Figure 1c**.



Figure 1 Description om next page

It was observed that after annealing in temperature of 800 °C and holding time of 15 minutes some areas of small recrystallized austenite grains are present and it is proved by the presence of annealing twins. It is also possible to notice the decrease of the participation of the ferrite bands and the appearance of the small globular divisions, as shown in **Figure 2a**. It was also noticed that after holding for 30 minutes in temperature of 800 °C the austenite grains grow, which can be seen in **Figure 2b**. After annealing in temperature of 850 °C it can be



seen that the further growth of recrystallised grain of austenite occurs and there is a decreased participation of ferrite bands observed, as shown in **Figure 2c**.

In the observation of the structure after annealing in temperature of 900 - 1000 °C it was found that the growth of the recrystallized austenite grains continues with the presence of many annealing twins and the bands of ferrite as well as globular division disappear and now only singular bigger divisions can be seen.



Figure 2 Microstructure of steel after cold rolling and annealing

Results of hardness measurements of each of the samples are shown **Figure 3**. On the basis of the results analysis one can observe a significant drop in the hardness of samples which were annealed in comparison with the initial condition. Together with the temperature increase the hardness of the samples gradually drops. In temperature range from 750 - 1000 °C there was a drop of hardness by about 25 %. Time extension for holding in a given temperature caused an average decrease of hardness by about 1.5 %.

A summary of the results of the mechanical properties tests is shown on **Figure 4** and in **Table 1**. The analysis shows that the higher the temperature of the annealing the lower the strength properties which gradually decrease. There is a significant decrease of the strength after annealing in 800 °C. The drop in strength is directly connected with the increase in plasticity of the given material which in this case improves its productibility. It should be pointed out that even after annealing in temperature of 900 °C the value of tensile strength does not drop below 1000 MPa. Together with the increase of annealing temperature there is a drop in yield stress observed. Elongation increases together with the increase of annealing temperature, but it should be pointed out that it does not exceed 50% in any of the cases.











Annealing temperature, °C / Holding time, min.	R _{p0.2} MPa	UTS MPa	A5 %
700 / 30	950	1180	12.5
800 / 15	805	1100	34
800 / 30	780	1080	36
850 / 15	735	1060	38
850 / 30	700	1045	40
900 / 15	660	1020	43
900 / 30	635	1000	46

Table 1 Results of static tensile test of samples after cold rolled and annealing

SEM analysis conducted for particular areas and characteristic points in the microstructure of steel. The presence of steel, manganese, aluminium and silicon was found present in the micro-areas and was shown in **Figures 5, 6.** In the areas of austenitic matrix (area 1) there is a significant dominance of the manganese content (37 %) and lower content of aluminium (5 %), whereas in the areas of ferrite bands (area 2) there are places observed which are richer in aluminium (6.5 %) and where the content of manganese is lower (28.5 %). The areas 3 and 4 are located in globular divisions areas and show an average aluminium content and are probably divisions of carbides type (FeMn)₄.



Figure 5 Microstructure (SEM) of X60MnAl30-9 steel after annealing at 800 °C/ / 15 min (a) and 900 °C / 15 min (b)

During the observation of substructure with the use of STEM microscope it was found that in case of cold rolling and annealing in 800 °C for 15 minutes the advanced process of recrystallization occurs which is proved by the presence of significant number of sub-grains and the presence of migrations of high-angle grain boundaries (Figure 7).

Dislocations within areas of sub-grains as well as a big number of formed annealing twins in sub-grain areas were also observed. In case of observation of sample annealed in temperature of 900 °C for 15 minutes it was found that the process of recrystallization here is much more advanced. The amount of present dislocations is decreased here and the migration of high-angle grain boundaries is smaller. There are more significantly bigger subgrains present with flat boundaries. The advanced recrystallization process can also be proved with the presence of a large number of annealing twins.









Weight %

	AI-K	Si-K	Mn-K	Fe-K
x60-MnAl-30-9 800C(1)_pt1	4.9	0.4	37.3	57.4
x60-MnAI-30-9 800C(1)_pt2	6.5	0.4	28.5	64.7
x60-MnAI-30-9 800C(1)_pt3	3.6	0.2	35.8	60.4
x60-MnAI-30-9 800C(1)_pt4	2.6	0.2	37.3	59.8

C)

Figure 6 Microstructure (SEM) of X60MnAl30-9 steel after annealing at 800 °C / 15 min (a), (b) EDS spectrum,) c) weight % of elements in selected area



Figure 7 Substructure of X60MnAl30-9 steel after annealing at 800 °C / 15 min (a) and 900 °C/ 15 min (b)

CONCLUSION

1) Tested steel is characterised with two-phase ferritic-austenitic structure. During recrystallisation annealing the fully recrystallised structure is present after annealing in temperature of 800 °C. Further temperature increase and extension of holding time causes the growth of recrystallised grains.



- 2) The ferrite which is present in microstructure on the longitudinal section shows a band structure along the direction of rolling. Together with the annealing temperature increase the amount of ferrite decreases.
- 3) In the substructure, there are well-developed subgrains with a big amount of annealing twins and small density of defects such as dislocations, which prove the proceeding process of static recrystallisation.
- 4) There were differences found between the chemical composition in the areas of austenite and ferrite. The regions of ferrite are richer in aluminium whereas the areas of austenite are characterised with increased amount of manganese.
- 5) It was found that the annealing temperature has influence on the strength properties and plastic properties and that together with the rise of the temperature of recrystallisation annealing the resistance properties and the offset yield strength decrease. Plastic properties in temperature range 800 900 °C slightly increase. There is a significant drop in strength observed after exceeding 750 °C, because above that temperature the hard phase (FeMn)₄ does not form. It was stated, for all described options that the tensile strength is bigger than 1000 MPa.
- 6) The most beneficial option when taking into account the manufacturing technology is the option with annealing at a temperature of 800 °C and holding time of 15 minutes. Elevation of temperature and holding time may lead to disadvantageous oxidation of the material as well as to grain growth. When the need arises to prepare more complex burrs the annealing in temperature 850 900 °C can be applied.
- 7) It was stated that there is a possibility to prepare metal sheets from steel X60MnAl 30-9 with high strength values, including the yield point on the level of 800 MPa with elongation to rupture not exceeding 40 %. It shows, that the tested steel can be applied as the element of motor-car body for vehicles built from thin metal plates with thickness of about 1mm. Metal sheets prepared from this type of steel may prove to be a better alternative to the currently applied metal sheets from DP steel (dual-phase steel).

ACKNOWLEDGEMENTS

The work was supported by the Ministry of Science and Higher Education within the framework of the BK-225/RM0/2017

REFERENCES

- [1] MULTI-AUTHOR WORK: ULSAB AVC "Body structure materials" TTS No4, 2001
- [2] GRÄSSEL O., KRÜGER L., FROMMEYER G., MEYER L.W. High Strength Fe-Mn-(Al, Si) TRIP/TWIP steels developments properties-application, International Journal Plasticity t. 16, 2000, pp. 1391-1409
- [3] FROMMEYERR G., BRUX U. Microstructures and mechanical properties of high-strenght FeMn-Al-C Light TRIPLEX Steels, Steel Research International, t. 77, 2006, pp.627-633.
- [4] HAMADA S. A. Manufacturing, mechanical properties and corrosion behaviour of high Mn Twip steels, Acta Universitatis Ouluensis, C281, 2007
- [5] FROMMEYERR G., BRUX U. NEUMANN P. Supra-ductile and high-strength manganese-TRIP/TWIP steels for high energy absorption purposes, ISIJ International, t. 43., nr 3, 2003, pp. 438-446
- [6] HOFMANN H, MENNE M, GÖKLÜ S, RICHTER H. Properties of austenitic high manganese steels with induced plasticity (LIP steels). Proc. of Int. Conf. on Steel Future for the Automotive Industry, Wiesbaden, Germany, 2005, pp. 73-80
- [7] SCOTT C., ALLAIN S., FARAL M. & GUELTON N. The development of a new Fe-Mn-C austenitic steel for automotive applications, Rev. Metall nr 6, 2006, s. pp. 293-302
- [8] ADAMCZYK M., KUC D., HADASIK E. Modelling of structure changes in TRIP type steel during hot deformation, Archives of Civil and Mechanical Engineering, vol.IV, No.3 2008, pp. 85-91;
- [9] KUC D., HADASIK, E.; NIEWIELSKI G. et al. Structural and mechanical properties of laboratory rolled steels highalloyed with manganese and aluminium, Archives of Civil and Mechanical Engineering, No. 12 (3), (2012), pp. 312-317