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REDUCTION OF MACROSEGREGATION IN CONCAST SLABS OF DEEP DRAWING STEEL

REDUKCJA MAKROSEGREGACJI W CIĄGŁYCH WLEWKACH PŁASKICH ZE STALI GŁĘBOKOTŁOCZNEJ

Results of laboratory and industrial tests related to development and implementation of the comprehensive technology of production and continuous casting of Bw11 low carbon steel for cold drawing of special products are presented. The aim of material tests was to assess the impact of the applied technological processes on reduction of central macrosegregation in slabs and plates. Segregation of alloying elements and impurities in steel, occurring during the process of concast slab solidification, results in formation of the central segregation zone. In heavy plates rolled from concast slabs, the zone is disclosed during the deep etch test, and constitutes material faults which disqualifies plates from application for cartridge cases. In the segregation band of higher hardness, revealed by means of the Oberhoffer's reagent, intensified pearlite banding, characteristic strips of fine pearlite and numerous non-metallic inclusions – mostly elongated MnS sulphides, were found.

In order to reduce central segregation in concast slabs, the following treatments were performed:

– reduction of P and S content in steel,
– modification of non-metallic inclusions by means of Ca addition,
– using Ti and B microadditives for the purpose of modification of steel solidification structure,
– reduction of the degree of steel overheating prior to casting,
– increasing intensity of concast slabs cooling,
– soft reduction of concast slab in the final phase of solidification.

By means of the standard technology modified in the above manner, plates without the central segregation were manufactured in the steel plant (Huta Stali Częstochowa). The produced plates fulfil conditions of the acceptance test specifications, moreover, their usability was verified by means of laboratory press forming tests as well as tests of industrial cartridge casing forming.

Keywords: central macrosegregation, concast slab, deep-drawing plates, low carbon steel

W niniejszej publikacji przedstawiono wyniki badań laboratoryjnych i przemysłowych, związanych z opracowaniem i wdrożeniem kompleksowej technologii wytwarzania i odlewania ciągłego stali niskowęglowej Bw11, przeznaczonej do tłoczenia na zimno wyrobów specjalnych. Celem badań materiałowych była ocena wpływu zastosowanych procesów technologicznych na redukcję makrosegregacji środkowej we wlewkach i blachach. Zachodząca podczas krzepnięcia wlewka ciągłego segregacja pierwiastków stopowych i zanieczyszczeń zawartych w stali powoduje utworzenie środkowej strefy pasm segregacyjnych. W blachach grubych walcowanych z wlewków ciągłych strefa ta ujawnia się podczas próby głębokiego trawienia i stanowi wadę dyskwalifikującą blachy do zastosowań na łuski amunicyjne. W paśmie segregacyjnym, o podwyższonej twardości, ujawnianym za pomocą odczynnika Oberhoffera, stwierdzono zintensyfikowaną pasmowość perlitu, charakterystyczne pasemka drobnopłytkowego perlitu oraz liczne wtrącenia niemetaliczne, w głównej mierze wydłużone siarczki MnS.

W celu ograniczenia segregacji środkowej w wlewkach ciągłych zastosowano:

– ograniczenie poziomu P i S w stali,
– modyfikację wtrąceń niemetalicznych za pomocą wapnia,
– mikrododatki Ti i B w celu modyfikacji struktury krzepnięcia stali,
– zmniejszenie stopnia przegrzania stali przed odlewaniem,
– zwiększenie intensywności chłodzenia wlewków ciągłych,
– zabieg miękkiego dogniatania wlewka ciągłego w ostatniej fazie krzepnięcia.

Za pomocą tak zmodyfikowanej technologii wytworzono w Hucie Stali Częstochowa blachy bez segregacji środkowej, które spełniły wymagania odbiorowe, a ich przydatność została zweryfikowana za pomocą laboratoryjnych prób tłoczności oraz prób przemysłowego tłoczenia łusek.

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1. Introduction

Plates of Bw11 steel, used for cartridge casings, due to deep drawing combined with thin walls drawing, need to be characterized by isotropy of structure and properties. Plates rolled from the cast strand, casting according to the standard technology, do not meet these requirements due to occurrence of the central macrosegregation. Macrosegregation constitutes a significant internal defect in concast slabs, which persists after homogenizing annealing and rolling. In the rolled products, the quenched segregation bands reduce plasticity, resulting in delamination and tearing of material during the cold drawing, and possess a risk of products cracking while in use. Segregation bands constitute a sharp contrast in macrostructure images in the macroetching test, and are considered to be such defects which qualify plates as rejects.

The present publication presents results of investigation of segregation band structure as well as description of the comprehensive technology of steel melting and continuous slab casting, implementation of which allows to obtain plates with the central segregation considerably reduced. These plates meet acceptance tests specifications and gives a positive result in the special product forming test.

2. Macrosegregation and methods of its reduction

The central macrosegregation occurs during slab solidification, as a result enriching with alloy elements and impurities contained in steel of its central zone [1,2]. Conditions of concast slab crystallization differ from thermodynamic equilibrium, and as a result of which constitutional supercooling occurs ahead of the solidification front. Alloying and additive elements diffuse from the δ- ferrite dendrites to the interdendritic space, while the highest level of enrichment may be observed in the central zone of the slab, which solidifies at the latest, forming central macrosegregation bands. In this zone, as a result of carbon and alloying elements concentration, bulging of ferrite/pearlite microstructure as well as increase of pearlite and/or bainite fraction occurs. In phosphorus enriched areas, etched by means of the Oberhoffer's reagent, simultaneous segregation is observed of sulphur and manganese, which form MnS sulphates, vulnerable to deformation during a rolling process.

In order to reduce macrosegregation, numerous technological treatments are applied in the process of steel melting and continuous casting, which result in more uniform distribution of alloying components and non-metallic inclusions in the solidifying slab. Amongst these, three most effective ways may be distinguished.

The first method includes improvement of metallurgical quality of steel by proper scrap selection, ladle treatment of liquid steel, modification of non-metallic inclusions by means of Ca, making that microadditives modify the structure of slab crystallization [3, 4]. *The second method* consists of reduction of columnar crystals fraction in the structure of solidifying slab in favour of the equiaxed crystals, *inter alia*, by low superheat casting as well as electromagnetic stirring of the mould; below the mould and in the final slab solidification zone.[4, 5]. *The third method* consists processing of the solidified skin of the slab and two-phase zone in the find area of the solidification crater. For this purpose, mechanical "soft" reduction is used (MSR) and/or intensified cooling in the final phase of slab solidification (Thermal Soft Reduction: TSR) [4,6]. As a result of the slab skin pressing in the proximity of solidifying liquid steel cone, closure there occurs as well as forced flow of the liquid in a direction opposite to casting. In this way, the macrosegregation zone is reduced, and a portion of liquid steel enriched with segregation elements is mixed with the rest of the unsolidified steel. Elimination of the results of contraction (cavity pipes, central line cracks and porosity) in concast slabs is additional effect of steel continuous casting using the SR system.

3. Goal, scope and methodology of tests

A comprehensive technology was developed and verified on a low carbon steel melting and continuous casting and the purpose of which was to reduce the central segregation in concast slabs and heavy plates. Tests were conducted in order to explain the essence of the central macrosegregation and effectiveness of the modified standard technology of plates manufacturing at the industrial conditions by comparison of the structure of concast slabs as well as plates' structure and properties.

Macrostructure investigations were carried out with the Baumann print method as well as macro etching tests. The Baumann prints and macroetching tests were carried out on cross sections of the entire gauge and width of slabs and plates. Disks were ground, and then the Bauman prints were made. Next, the disks were subject to the macro etching in aqueous solution of HCl in 1:1 proportion.

Microstructure investigations of concast slabs and plates following rolling, as well as quenching and tempering were carried out on metallographic specimens subjected to the Nital or Oberhoffer's reagent etching. Microstructure was analysed by means of the light microscopy, scanning electron microscopy (Philips XL30), as well as transmission electron microscopy (JEM 2000

FX equipped with ISIS system for microanalysis of chemical composition in micro-areas).

Investigations of non-metallic inclusions occurring in plates were carried out on non-etched metallographic specimens, conducted on the plane parallel to the rolling direction. Qualitative assessment of non-metallic inclusions was carried out with a comparative method, according to the reference standard scale. A quantitative assessment of inclusions was carried out by means of IBAS 2000 computer image analyzer. Microanalysis of chemical composition of inclusions was carried out by means of EDAX energy dispersive X-ray spectrometer scanning electron microscope Philips XL30.

Plates' properties were determined in tensile tests on samples longitudinal and transverse to the rolling direction and plate gauge. Impact tests were conducted on standard V-notch samples from plate sections subject to strain ageing.

Physical simulation of drawing was made on plates with sections dimensions: 300×30×19 mm. These samples were initially bent and then subjected to a hydraulic press drawing at the conditions reproducing the technological process of the cartridge case manufacturing. *Industrial press forming tests* were carried out in metal stamping plant (Tłocznia Metali "Pressta"), where several dozens of cartridge cases were manufactured in the line with the current technology. The final stage of tests included *dynamic tests*, carried out at the military training area.

4. Modification of heavy plate manufacturing technology in order to reduce segregation

Bw11 low carbon steel for cold drawing of special products, with the chemical composition according to PN-H-84023-10 standard: 0.09 to 0.13% C, 0.30 to 0.50% Mn, max. 0.13% Si, max. 0.025% P, max. 0.025% S, 0.03 to 0.07% $Al_{solub.}$ was subjected to testing. Industrial heats were made in the steel plant (Huta Stali Częstochowa), in KONEL electric converter. The examined steel was subject to ladle treatment and vacuum degassing in a VD chamber. A continuous casting method was applieded to produce 10 heats (Table 1) with different: content of micro-additives, degree of non-metallic inclusions modification, casting rate, cooling intensity and application of slab soft reduction (SR).

Preparation of high purity homogenous steel included:

– Reduction of the content of deleterious elements: P, S, O, N, H,

– Reduction of Al content and adding microadditives of Ti or Ti+B,

– Modification of non-metallic inclusions by means of Ca addition,

– Homogenizing of metal bath by means of argon bubbling.

The following techniques were applied during a *continuous steel casting* to reduce segregation:

– Reducing casting temperature to 17-26°C above liquidus,

– Soft reduction (SR) of solidifying slab,

– Increasing intensity of secondary cooling.

One heat (no. 254342) was made by means of the hitherto applied technology, typical for carbon steels, while modifications were introduced into the other heats, in order to reduce macrosegregation (Table 1). During casting with the application of SR, the soft-reduction zone of the slab was located between 16 and 21 metre from steel meniscus in the mould. The taper of strand

TABLE

Characteristics of tested material of Bw11 steel

Heat number	Technology modification				
	Micro-additives	Ca treatment	Bath superheating over liquidus temperature, °C	Slab cooling intensity litres of water per kg of steel	Soft reduction SR
254342			50	1	
512845	0.002% B; 0.015% Ti	0.0017% Ca	26	1.5	
512846	0.018% Ti	0.0020% Ca	25	1.5	
512847		0.0016% Ca	24	1.5	
516149		0.0025% Ca	20	2	
516150	0.011% Ti	0.0025% Ca	21	2	
517701	0.023% Ti	0.0029% Ca	20	2	From 225 to 220.5 mm
518870	0.025% Ti	0.0025% Ca	22	2	From 225 to 220.5 mm
523279	0.014% Ti	0.0010% Ca	23	2	From 225 to 220.5 mm
523280		–	17	2	From 225 to 220.5 mm

drawing system was adjusted by means of spacers installed on the segments' columns. The casting rate was increased from 0.9 to 1.2 m/min in order to adjust slab solidification cone (computer-calculation based on the system model) to the end of the soft reduction zone in the CCM machine. Intensity of cooling of slabs casting with an application of the SR technology was doubled compared to the reference melt. Slabs were cast into the mould of cross-sections: 225 mm × 1300 mm and 220 mm × 1500 mm, moreover, prior to the plate rolling they were subjected to annealing at temperature of 1250°C. 19 mm gauge plates were rolled at the temperatures ranging from 1200°C (initial rolling temperature) to 850°C (final rolling temperature).

5. Identification of central macrosegregation

Simulation of steel solidification during cooling carried out by means of a computer programme Thermo-Calc has shown that fraction of austenite solidifying directly from the liquid phase below 1500°C exceeds 10 mass % [7]. It was ascertained that in the liquid phase remaining in the solidifying slab, carbon, manganese, sulphur, phosphorus and silicon shown a significant increase of segregation, while final sulphur and phosphorus enrichment compared to the average composition of steel could even be multiplied (up to several dozens).

Plates from reference heat no. 254342 tapped and cast in a classic way (without application of the modified technology) were not qualified for cartridge case forming due to segregation bands occurring in the central zone (Fig.1). Segregation bands visualized by means of the Oberhoffer's reagent have shown that they are characterized by reduced propensity to etching and increased hardness (Fig. 1b). The Nital etching have shown increased density of pearlite strips in these bands (Fig. 1c). In the segregation band in relation to the other sections of plate, concentration of MnS sulphides elongated towards the rolling direction as well as TiN nitride precipitations were observed (Fig 2a). Moreover, a presence of very fine pearlite bands strongly elongated towards the rolling direction was recorded. They were not observed outside the segregation area.

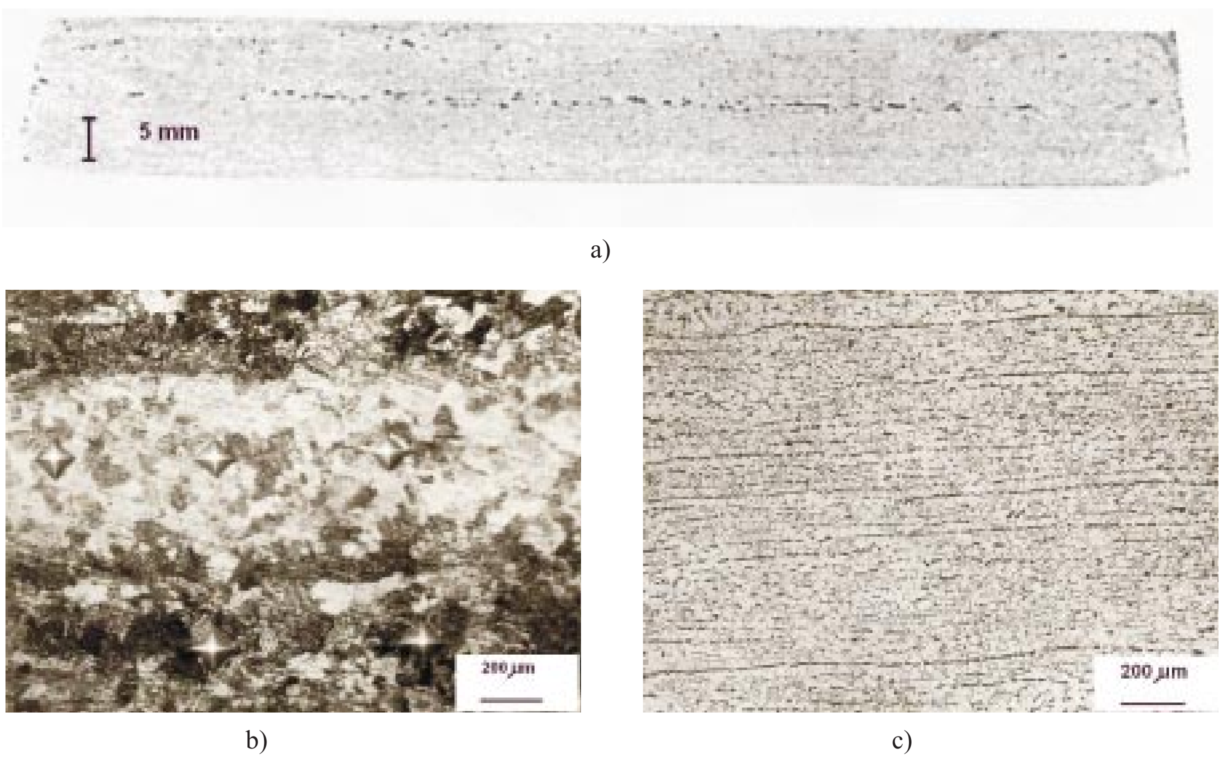
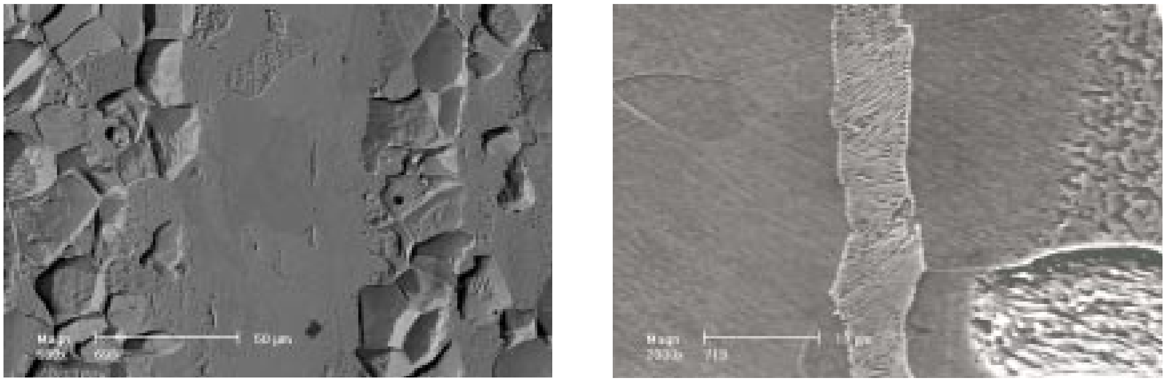


Fig. 1. Structure of segregation band in the plate of Bw11 steel, heat no. 254342: a) macrostructure of plate cross-section after macro-etching, b) segregation band revealed using the Oberhoffer's reagent, c) ferritic and pearlitic structure in segregation bands presented in Fig. b)



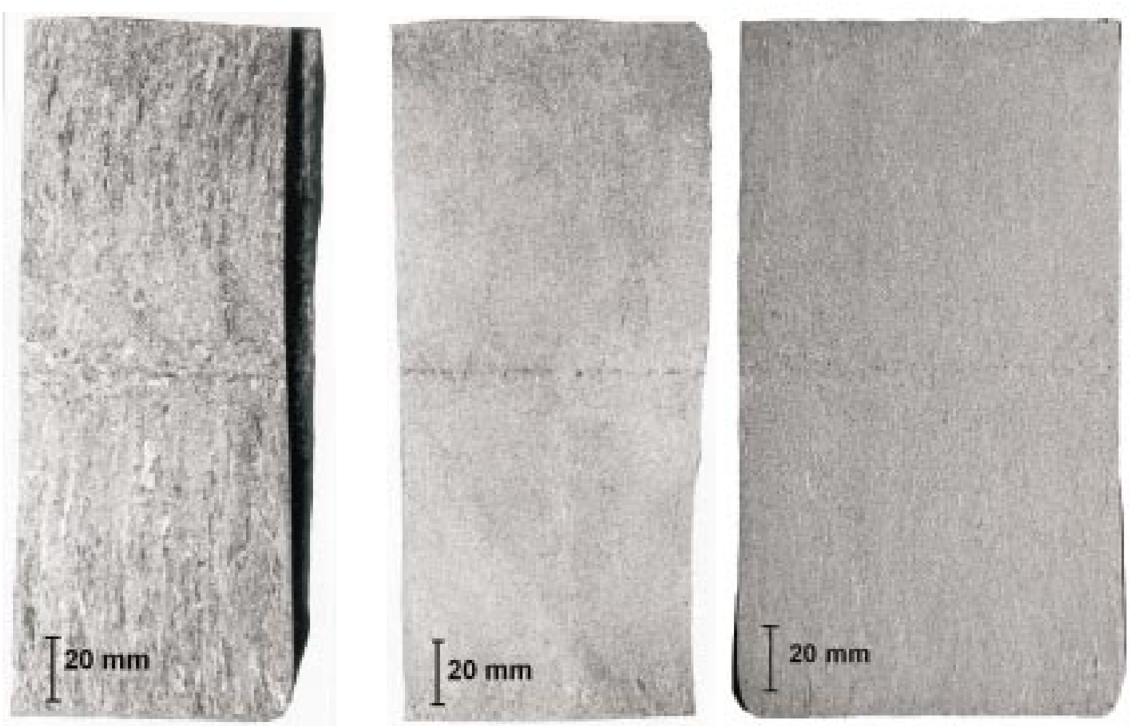
a) b)

Fig. 2. Scanning micrograph of segregation band structure in the centre of the plate gauge a) MnS and TiN precipitation, b) elongated pearlite colonies

6. Assessment of the structure and properties of products manufactured by means of the modified technology

Industrial heats of Bw11 steel met requirements of PN-H-84023-10 standard pertaining to the basic chemical composition as well as additional requirements pertaining to restrictions on chemical composition and content of residual elements and micro-additives in the finished steel. The applied technology of melting and furnace refining allowed to obtain phosphorus content in the finished steel on the level of 0.008-0.009% as well as a low content of the other undesirable impurities. A ladle treatment as well as a vacuum degassing ensured obtaining very low content of sulphur, ranging from 0.001 to 0.004% and hydrogen at the level of 2-3 ppm.

Analysis of the images of micro- and macrostructure of slabs has shown qualitative diversity which can be justified with the applied manufacturing technology (Fig.3).



a) b) c)

Fig. 3. Macrostructure of cross section of concast slabs of Bw11 steel heats: a) no. 254342 (without microadditives and modifiers), b) no. 512846 (Ti, Ca/S=0.66), c) no. 517701 (Ti, Ca/S=2.42)

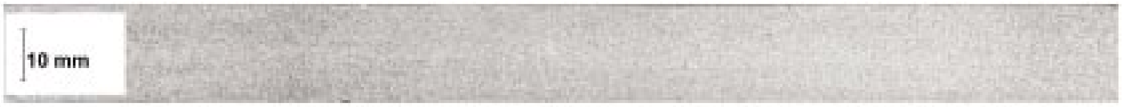


Fig. 4. Macrostructure of the central section of plate after macroetching, heat no. 517701

Compared to the reference melt (Fig.3a), introduction of Ti resulted in a more homogenous macrostructure, characterized by fine grains and reduction of the crystallization zones, typical for the low carbon steel (Fig. 3b). Only the surface zone of frozen crystals is clearly outlined. Comparative tests of slabs microstructure have shown that ferrite and pearlite in the heats with Ti, form homogenous structures of equiaxial grains, while in the steel without Ti – form banded, acicular structures. Owing to modification of sulphate inclusions, intensive cooling and soft reduction of solidifying slab, considerable homogeneity of macrostructure and disintegration of segregation bands was observed (Fig. 3c).

Investigations of macrostructure of plates after macroetching have shown that an average fraction of the segregation bands in the plate width was as follows:

• above 16% in plates of heat no. 254342, without special treatments during slab casting,

• ca. 6% in plates of heats without soft reduction (SR) with moderate slab cooling,

• ca. 4% in plates of heats without soft reduction (SR) with intensive slab cooling,

• ca. 2% in plates of heats made with application of the soft reduction (SR) and intensive slab cooling, in that ca. 0.1% – in plates of heat no.517701.

This data shows that P and S level, Ca/S ratio and continuous casting parameters have impact on the number and size of segregation bands in the Bw11 steel. Comparing macro- and microstructure images of plates of different Bw11 steel melts, it was ascertained that the highest fraction of segregation bands occurs in the plates of heat no. 254342, carried out in line with the classic technology, while the desired, and fully satisfying structure quality, without the central line segregation bands characterizes plates of heat no. 517701 made according to the modified technology (Fig. 4). The new technology included full modification of sulphate inclusions by means of Ca, intensive cooling as well as soft reduction of the solidifying slab.

In plates of heats made in line with the modified technology, as a result of P and S content reduction as well as effective refinement of steel along with modification of non-metallic inclusions by means of Ca, degree of steel contamination with non metallic inclusions did not exceed standard 1 according to the reference standard scale. K1 inclusion ratio determined for the selected plates was 8.3, in that 1.1 for sulphides and 7.2 for oxides. Content of inclusions in plates, estimated by the method of image analysis was ranging from 0.04 to 0.08%. The average shape factor calculated as a ratio of the largest size to the smallest size, was 1.5, and the average size – ca. 10 μm. A sulphur susceptibility to segregation causes that a pure reduction of its content does not eliminate manganese sulphides from segregation bands. Only simultaneous reduction of sulphur content and application of steel treatment with calcium resulted in the evenly distributed globular oxide-sulphides composed of calcium aluminate with calcium sulphide outside layer surface emerging in place of aluminium oxides and manganese sulphides (Fig. 5). Moreover, TiN nitrides dispersive precipitation occurrence was ascertained in or on the boundaries of ferrite grains or in dislocations.

In case of all plates, the required level of mechanical properties compliant with PN-H-92210-2 standard R_m and A_5, was achieved, as well as a high contraction level, ca. 70-80% and impact resistance ranging from 320 to 360 J/cm^2. Similar values of contraction in all three directions: longitudinal, crosswise and in the direction of plate gauge, as well as impact strength measured on the samples longitudal and crosswise to the direction of rolling prove plates' isotropy.

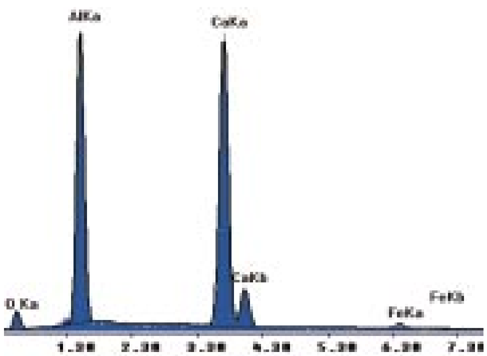
Properties of 8 mm quenched and tempered plate were as follows:

$$R_{0,2} = 260MPa, \quad Rm = 360MPa, \quad A_5 = 35\%$$

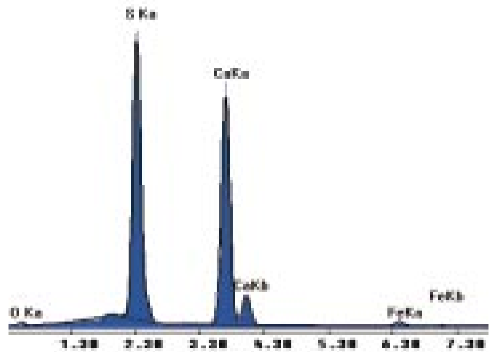
Also the properties of steel during cold rolling of flats with draft ranging from 0.29 do 0.69 were investigated. In case of strain with draft of 0.69 value of R_m increased by more than 50%, while A_5 elongation decreased by 68% compared to its initial value. Strain hardening has a crucial impact on cartridge cases, as the required strength of the bottom above 588 MPa, is achieved by double upset forging following quenching and tempering or normalizing.

Above all, plates for cartridge cases should be characterized by ability to plastic deformation, applied in the used press forming and drawing process producing extremely thin wall.

a)



b)



c)

Fig. 5. Globular non-metallic inclusions in Bw11 of heat no. 517701 (Ca/S = 2.42). a) scanning micrograph of the inclusion, b) core microanalysis results xCaO-yAl_2O_3, c) results of microanalysis of outside layer surface of CaS calcium sulphide

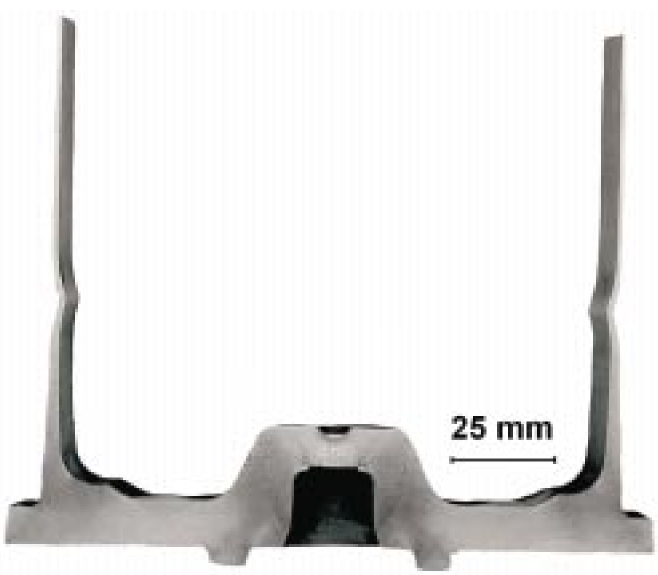


Fig. 6. Image of the macrostructure of Bw11steel pressed product, without segregation. Longitudal section after macroetching. Heat no. 517701

Simulation tests of drawability, consisting of drawing the walls of bent U-shaped flat of initial gauge 18 mm, to a final gauge of 1.3 mm, reflecting approximately thickness of cartridge case, gave positive results and constituted a basis for undertaking industrial test of products' press forming.

On the basis of simulation tests, plates of 517701 [8] heat were qualified to press forming of special products. No discrepancies from the currently binding process were found in the case of this material during press forming: dishing, cupping, 1st, 2nd, 3rd pass as well as bottom pressing. Results of measurements carried out after each interoperational recrystallization annealing were compliant with the values assumed. Results of mechanical properties tests of the wall and bottom of the finished products met the requirements of technical documentation:

– wall: R_m = 333 – 441 MPa, A_{10} = from 20% to 25% ,

– bottom: R_m = min. 588 MPa.

Macrostructure in the entire section of the product was homogenous and without the central segregation, except the for local macrostructural changes resulting from the press forming process (Fig. 6). Subsequent dynamic tests have shown proper response of products. Based on the obtained results of press-formability, industrial batch of plates was made in order to complete the order for special products placed in the metal stamping (forming) plant.

7. Conclusions

1. Segregation of alloying elements and impurities occurring during concast slab solidification results in formation of segregation bands in the central zone. In heavy plates this zone is revealed with macroetching test and constitutes the defect which disqualify plates from the application for cartridge cases. Material for this application should be characterized by full isotropy of structure and properties. During deep drawing of plates, combined with thin wall drawing (stretching), hardened segregation bands result in delamination and tearing of material
2. In the segregation band of the higher hardness, exposed by means of the Oberhoffer's reagent, intensified pearlite banding, inclusions of elongated MnS sulphates, characteristic strips of fine lamellae pearlite and TiN were stated.
3. In order to reduce the deleterious effect of central segregation on mechanical properties, the following treatments were performed:
   – reduction of P and S content in steel,
   – modification of non-metallic inclusions by means of Ca,
   – Ti and B microadditives for the purpose of modification of steel solidification structure ,
   – reduction of the degree of steel overheating prior to casting,
   – increasing intensity of concast slabs cooling,
   – soft reduction of concast slab in the last phase of solidification.
4. By means of the modified technology, a range of industrial heats was made in the steel plant (Huta Stali Częstochowa). Plates without the centre line segregation were obtained from the heat in the case of which intensive cooling, soft reduction of slab and full modification of inclusions were applied. This heat is characterized by low content of sulphur (0.001%), phosphorus (0.008%) and oxygen (20 ppm), Ti at the level of 0.011% as well as high Ca/S ratio of 2.42. Only composite globular calcium aluminate inclusions with calcium sulphide coat, evenly distributed in the matrix were found in plates, while elongated MnS inclusions were not found.
5. Plates manufactured by means of the modified technology met the acceptance test specifications, moreover their usability was verified by means of laboratory press forming tests as well as tests of industrial cartridge casing forming.

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