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Effect of deformation-induced martensite on the microstructure, mechanical properties and corrosion resistance of X5CrNi18-8 stainless steel

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ABSTRACT

Purpose: The aim of the paper was to determine the effect of deformation-induced martensite on the microstructure, mechanical properties and corrosion resistance of X5CrNi18-8 austenitic steel.

Design/methodology/approach: The investigations included observations of the microstructure on a light microscope, researches of mechanical properties in a static tensile test, microhardness measurements made by Vickers's method and corrosion resistance test examined using weight method. The analysis of the phase composition was carried out on the basis of X-ray researches. The amount of martensite α ' in the obtained microstructures was investigated with ferritescope magnetic tester. The observations of the surface morphology after corrosive tests were carried out using Scanning Electron Microscope. The scope of this study was to achieve the correlations between the mechanical, corrosion and structural properties of cold rolled stainless steel.

Findings: Plastic deformation in a cold working of austenitic stainless steel induced in its structure martensitic transformation $\gamma \rightarrow \alpha'$. The occurrence of martensite α' in the investigated steel structure has an essential meaning in manufacturing process of forming sheet-metals from austenitic steel.

Research limitations/implications: The X-ray phase analysis in particular permitted to disclose and identify the main phases on the structure of the investigated steel after its deformation within the range 10 - 70%. The results of the ferritescope measurements allowed determining the proportional part of α ` phases in the structure of investigated steel in the examined range of cold plastic deformation. The microscope observations of the surface samples subjected to corrosion resistance test in 30 wt% H₂SO₄ solutions permitted to evaluate kinds and the rate of corrosion damages.

Originality/value: A wide range of practical applications of 18/8 steel sheets is warranted by both their high corrosion resistance and high plastic properties.

Keywords: Austenitic stainless steel; Plastic deformation; Mechanical properties; Weight loss method; H₂SO₄ solution

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PROPERTIES

1. Introduction

The chemical, machinery, food, automotive, building, nuclear and shipbuilding industry requires increasingly steels with good combination of excellent corrosion resistance, high mechanical properties and good formability. Metastable austenitic stainless steels, well-known as 18/8 types combine these properties, making them very attractive [1-4].

Austenitic stainless steels contain most often about 16-25% wt. chromium, 0.1% wt. carbon and up to 7% wt. molybdenum, and not less than 7.5% wt. nickel, which is indispensable to obtain of the single-phase structure γ . In the annealed state these steels are characterized by high plasticity and relatively low strength (R_m about 550 MPa; R_{p0.2} about 200-250 MPa). After thermomechanical treatment common steel such as X5CrNi18-10 can have its yield strength increased to about 1400 MPa, with an elongation over 10% [5-9].

Strain hardening of austenitic stainless steels cannot be obtained by heat treatments, it is possible especially by cold or warm working (drawing, rolling, forging, etc.). During plastic deformation, depending on the steel composition and the cold working variables, stacking faults, two different martensitic phase, ε -martensite (HCP) and α '-martensite (BCC) can be formed. The α ' phase is often called strain-inducted martensite because it is produced be a diffusionless phase transformation. The most probable way of the phase transformation in the 18/8 types of steels is the: $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ or $\gamma \rightarrow \alpha'$. The volume fractions of the particular phases influence the mechanical (strength, strain) and other properties for example corrosion resistance of these steels. The degree of martensite deformations depends on the volume of the applied draft, the chemical composition and plastic deformation temperature [10-13].

Resistant to corrosion of stainless steels can be achieved by dissolving sufficient chromium in the iron to produce a coherent, adherent, insulating and regenerating chromium oxide protective film (Cr_2O_3) on the surface. Iron does not occur in its native state because it combines readily with oxygen and other elements. It is extracted from its ore and given the opportunity, tends to revert to a compound by reacting with the environment. Rusting is an example of this reversion process. The process can be retarded by adding chromium, which at sufficiently large concentrations forms a protective oxide film at the surface. The stainless character occurs when the concentration of chromium exceeds about 12 wt%. The passive film of chromium oxide formed in air at room temperature are only about 1-2 nm thick covers the steel. However, even this is not adequate to resist corrosion in acids such as HCl or H₂SO₄[14].

Corrosion can nevertheless occur if the passive film breaks down, locally or uniformly.

Uniform corrosion can occur in acidic or hot alkaline solutions. Loss by this mechanism can be estimated and allowed for in design. The corrosion rate is very slow when the metal is in the passive state. General corrosion resistance is better at larger chromium contents, but other solutes can be detrimental. In particular, sulphur in solid solution is believed to make passivation more difficult. Unfortunately, sulphur alters the temperature dependence of the surface tension of liquid and causes increasing the penetration during welding of stainless steels. The fluid flow that occurs in the weld pool is such that in the absence of sulphur, shallow (wide) weld pools are obtained resulting in unacceptable joints when the concentration is less than about 0.007 wt%. An even higher sulphur concentration may be used in free-machining stainless steels where precipitated sulphur helps break up the machining chips. Nickel significantly improves the general corrosion resistance of stainless steels, by promoting passivation. Austenitic stainless steels therefore possess superior corrosion resistance when compared with martensitic or ferritic stainless steels (with zero or low nickel concentrations), particularly when in contact with mineral acids [15,16].

Pitting corrosion is the result of the local destruction of the passive film and subsequent corrosion of the steel below. It generally occurs in chloride, halide or bromide solutions. It can be initiated at a fault in the passive layer or a surface defect. The steel underneath the break dissolves leading to a build up of positively charged metal ions, which in turn causes negative charges (e.g. chloride ions) to migrate near the defect. Even in a neutral solution, this can cause the pH to drop locally to 2 or 3, thereby preventing the regeneration of the passive layer. In the passive condition, the current density is of the order of $\mu A \text{ cm}^{-2}$; in the pit, however, it may exceed 1 A cm⁻². The reason why the current density is so large in the pit is that the anodic region is very small in area when compared with the cathodic part (the unpitted steel). For a given corrosion current, this greatly exaggerates the corrosion rate at the pits. Similarly, the concentration of chloride ions in the vicinity of a pit can be thousands of times greater than that in the solution as a whole [17-18].

The anodic dissolution of the steel (Fig. 1.) leads to introduction of positive metal ions (M^+) in solution, which causes migration of Cl⁻ ions. In turn, metal chloride reacts with water according to the reactions:

$$M^{+}Cl^{-} + H_{2}O \rightarrow MOH + H^{+}Cl^{-}$$
(1)

This causes the pH to decrease. The cathodic reaction, on the surface near the pit follows:

$$O_2 + 2H_2O \rightarrow MOH + 4OH^-$$
⁽²⁾



Fig. 1. Generalised illustration of pitting corrosion of stainless steel [19]

The propagation phenomenon is well understood in comparison to mechanism of pit initiation. Initiation process was associated with MnS inclusions which are difficult to avoid in the steel-making process. It appears that the inclusions are surrounded by a Cr depleted region which is believed to cause the initiation.

$$PRE = %Cr + m%Mo + n%N$$
(3)

where: m and n are the factors for Mo and N.

For austenitic stainless steel PRE can be calculated using the following dependence:

$$PRE = %Cr + 3.3\%Mo + \upsilon\%N$$
(4)

where: v = 10 to 30%; usually for these kind of steel v = 16%.

The value of PRE for X5CrNi18-8 steel contain in range from 17.0 to 20.8.

The aim of these investigations was to define the influence of the degree of plastic deformation in the cold rolling process on the microstructure, mechanical properties and corrosion resistance in acidic solution of austenitic stainless steel type X5CrNi18-8. In particular special attention was put on the amount of martensite α' phase occurring in the structure of tested stainless steels after cold rolling.

2. Experimental procedure

The examinations were carried out on metastable austenitic stainless steel grade X5CrNi18-8, resulting from industrial smelting from the UGINE&ALZ (Poland). The chemical composition of the investigated steel, according to PN-EN 10088-1:2007 [20] is presented in Table 1. The material for examinations was delivered in the form of sheet-cutting steel with dimensions about $2 \times 40 \times 700$ mm, subjected to cold rolling ranging from 10%, 20%, 30%, 40%, 50% to 70%, using the sheet mill Quarto type 10502 produced by Skoda. The rolling was conducted at 25°C keeping a constant direction and side of the rolled strip. The steel sheets were sampled for research of the mechanical properties, for microhardness measurements, corrosion resistance, metallographic observations and the X-ray phase analysis.

Table 1.

Chemical composition of the investigated steel

Mass contents, (%)								
С	Cr	Ni	Mn	Si				
0.03	18.07	8.004	1.31	0.39				
Мо	Ν	Р	S	Fe				
0.25	0.044	0.03	0.004	bal.				

Microscopic examinations of the microstructure of austenitic X5CrNi18-8 stainless steels were performed on longitudinal polished microsections and chemically etched in the Baraha's reagent [21]. Samples with dimensions about 10×15 mm were cut from the steel sheets in the delivery and deformed state. Metallographic observations of the microstructure were performed in a light microscope OLYMPUS GX71, equipped with a image analyzer system. Observations were realized with a magnification from 400 to 1000x.

Additionally the average grain size of specimens was determined using the method of counting the slits grains into the image area, according to standard PN-EN ISO 643:2005 [22].

The metallographic investigations permit to assess the steel microstructure type X5CrNi18-8 in the delivery and to define the influence of the degree of plastic deformation on its cold rolled structure with a draft from 10 to 70%. Furthermore to determine the metallographic symptom of draft, shape and size of austenite grains.

The amount of deformation induced α' martensite was determined by magnetic measurements, using a ferritescope (Helmut Fischer, model FMP30), according to the standard PN-EN ISO 8249:2005 [23]. The device was calibrated with δ -ferrite standard samples and the results were converted to the α' -martensite contents with the correlation factor of 1.7. In measurements the standard samples sets type M-0620 were used. The effect of sheet thickness on the results was compensated by means of a correction curve provided by the manufacturer of the device. The magnetic measurements with the ferritescope were performed on the metallographic samples, both in nondeformed and deformed state. Tests were carried out in 5 point measurements of each specimen.

The mechanical properties were determined applying static tensile test and measurements of the microhardness.

The static tensile test were performed on the testing machine ZWICK 100N5A. Dimensions of test samples were determined on the basis of PN-EN 10002-1+AC1:2004 [24] standard and cut from the steel sheet parallelly to the rolling direction.

The microhardness measurements of the investigated cold reduced sheets from steel X5CrNi18-8 were carried out by a microhardness tester (Hauser, model PMT-3), according to the standard PN-EN ISO 6507-1:2007 [25]. Researches were made by Vickers's method on metallographic samples with a load value about 50 g.

In order to study the structural changes taking place during cold plastic deformation on steel X5CrNi18-8 X-ray phase analysis has been used applying the filter cobalt radiation (λ CoK_{α}= 0.179021 nm). The X-ray diffraction patterns were performed on the X'PERT PANalytical diffractometer with the accelerating voltage of 45 kV and current intensity of 40 mA. The data of diffraction lines were recorded by "step-scanning" method in 2 Θ range from 45° to 115° and the 0.1° step and a time of measurements amounting to 2 seconds in one measurement position. The obtained diffraction patterns were analyzed applying the program PCPDFWIN.

Corrosion resistance of investigated steel was examined using weight method according to the standard EN ISO 7384:2001 [26] and EN ISO 3651-2:1998 [27]. Gravimetric tests were carried out in 30 wt% H₂SO₄ solution on samples with dimensions about 20×15 mm. The solutions were prepared using distilled water and analytical grades of chemicals (H₂SO₄). The specimens were cut along to rolling direction from steel sheet-metals in its delivery state and after cold rolling with a draft of 10-70%. Before starting the analysis the samples were cleaned in 95.6% ethanol and weighed on analytical balance to a precision of 0.01 g. The test of corrosion was realized at room temperature, whereas the time of the test was equal 25 days. Value of corrosion loss was calculated using the following dependence:

$$V_c = \frac{\Delta G}{A \cdot t} \left[\frac{g}{m^2 \cdot day} \right]$$
(5)

where: ΔG - mass loss of a sample [g], A - surface of a sample [m²], t - time of the measurement [day].

Linear corrosion rate was determined basing on the dependence:

$$V_p = V_c \cdot k \left[\frac{mm}{year} \right]$$
(6)

where: V_c - value of corrosion loss $\left[\frac{g}{m^2 \cdot day}\right]$, k - ratio including

mass loss of a sample and linear corrosion rate calculated by the formula: $k = \frac{365}{2}$ where ρ - density of material $\left[\begin{array}{c} g \\ g \end{array} \right]$.

For investigated X5CrNi18-8 steel the density of about
$$\rho = 7.9 \left[\frac{g}{cm^3}\right]$$
 was established.

Metallographic macroscopic investigations of samples after gravimetric tests were performed on LEICA MEF4A light microscope. The microscope observations of the surface samples subjected to corrosion resistance test in 30 wt% H_2SO_4 solutions permitted to evaluate kinds and the rate of corrosion damages. In order to observe the morphology of the corrosion products formed on the material surface after weight loss tests the Scanning Electron Microscopy SUPRATM25 produced by ZEISS was used. Observations carried out applying the magnification of 280, 400 and 3000x.

3. Experimental results and discussion

The steel structure in the delivery state shows equiaxial austenite grains about $21 \mu m$ average a diameter with many twins (Fig.2). The metallographic symptoms of deformation in steel structure in that state were not affirmed.



Fig. 2. Microstructure of X5CrNi10-8 austenitic stainless steel in the delivered state

The optical micrographs of 20, 50 and 70% cold rolled specimens from steel type X5CrNi18-8 are shown in Fig. 3.

In a bright field (optical) microstructure, the etchant used brings out the martensite phase as a dark constituent, the austenite phase remaining white.



Fig. 3. Microstructure of X5CrNi18-8 austenitic stainless steel after cold rolling with a draft of (a) 20%, (b) 50% and (c) 70%

The wavy features in the microstructure going in diagonal directions signify the manifestations of the deformation layer on the specimen surface. The small amount of black features seen in Fig. 3a is due to residual fine scratches and inclusions.

Deformation with a larger draft of about 50% causes in the steel structure the increase in the amounts of the dark phases signifying the increase in the amounts of the martensite phase (Fig. 3b). It is apparent that the slip lines and the twins had a bearing to the nucleation and the growth of this phase.

Metallographic observations of steel structure deformed with a maximum degree of deformation, about 70% show the large areas of the dark phase recognized as a martenisie α ' and a few light area coming from the austenite matrix (Fig. 3c).

During the cold rolling process with draft degree over 40% the grain boundaries are faintly visible and considerable size reduction of the steel structure were observed. Because of this, no attempt has been made to measure the average grain size by the standard intercept method.

It is already known that in metastable austenitic stainless steel during plastic deformation the martensite transformation take place [28]. The phase transformation normally leads to formation of two products: one magnetic bbc (α ') and other nonmagnetic and hexagonal close-paced (ϵ). In case of X5CrNi18-8 austenitic stainless steel according to metallographic results the ϵ phase dose not occurs. So, the following martensite transformation $\gamma \rightarrow \alpha$ ' take place.

On the basis of the realized magnetic investigations it was found that the amount of the martensite α ' phase in the investigated steel structure increases within the increases the of draft degree in the cold rolling process.

The volume percentage of strain inducted α ' martensite of cold rolled X5CrNi18-10 steel samples have been presented on Fig. 4.

In the delivery state the X5CrNi18-8 steel characterized the magnetic permeability about 1.05, what allow to classify the studied steel as a paramagnetic material. Perhaps, the occurrence of α ' phase is results of pretreatment of material (cutting, roughing, machining), which causes the eutectoid changes of γ phase.



Fig. 4. Changes of martensitic α ' phases as a function of draft degree in the investigated steel

After that plastic deformation within the range of 10 to 50% the amount of α ' phases in investigated steel structure average from about 1.30 to about 11.10% (Fig. 4), what proves of proceed a martensitic transformation $\gamma \rightarrow \alpha$ '. Amount of formed phases increasing with the degree of deformation and after maximum, 70% of draft the volume of martensite α ' is on the level about 21.0%.

On the basis of the realized tensile tests the tensile strength R_m and yield point $R_{p0.2}$ were determined. The changes of the samples geometry were used to define the elongation A and reduction of area Z of the investigated steel. With the increasing of draft degree the value of the microhardness $HV_{0.05}$ increases too. The results of these investigations permit to define the influence of the degree of deformation in the cold rolling of steel on the ductile, strength properties and microhardness of the examined steel. The results of investigations concerning the mechanical properties have been presented on Fig. 5 and Fig.6.



Fig. 5. Changes of the mechanical properties (R_m , $R_{p0.2}$, HV_{0.05}) in the investigated cold rolled steel depending on degree of deformation



Fig. 6. Changes of the plasticity (A, Z) in the investigated cold rolled steel depending on degree of deformation

The tensile strength of the investigated steel in the delivery state is about 647 MPa, the yield point is about 330 MPa, the microhardness is about 162 $HV_{0.05}$, the elongation is about 52% and the reduction of area is on the level about 48%. After plastic deformation the microhardness and strength properties of the examined steel increases, while the plasticity decreases (Fig. 5, Fig. 6).

With the increasing of deformation within the range of 10-50% the tensile strength of X5CrNi18-8 steel increases from about 785 MPa to about 1257 MPa, the yield point from about 586 MPa to about 970 MPa, the microhardness from about 227 HV_{0.05} to about 357 HV_{0.05}, while the elongation decreases from about 32% to about 1% and the reduction of area from about 45% to about 15%.

Cold rolling of X5CrNi18-8 steel with a maximum draft, about 70% causes the increases of value tensile strength to about 1496 MPa, the yield point to about 1161 MPa, the microhardness to about 400 $HV_{0.05}$, and decreases the elongation to about 0.68% and reduction of area to about 11%.

The results of the influence of the degree of rolling on the values of R_m and $R_{p0.2}$ of the investigated steel were approximated suitably by the function $y = 2.8056x^3 - 31.25x^2 + 230.23x + 438.57$ and $y = 6.2778x^3 - 89.857x^2 + 497.94x - 92.429$. The coefficients of the matching R^2 of the approximate function amounts adequately to 0.993 and 0.9881 which proves good correlation of the approximate function with experimental results (Fig.5).

The dependence of the microhardness of the tested X5CrNi18-8 steel on the degree of deformation in cold rolling was described by the approximating function: $y = 1.7778x^3 - 25.31x^2 + 141.06x + 41$ whose coefficient of matching R^2 is about 0.9953. The value of the coefficient R^2 of the approximate function is close to unity and proves a good correlation of the analytical curve with experimental ones (Fig. 5).

The dependence of the elongation from the degree of deformation described with the aid of the function $y = 0.0744x^3 + 0.8495x^2 - 19.425x + 69.754$, whose coefficient of the matching R^2 is about 0.9877 (Fig.6). Whereas, the dependence of the reduction of area from the degree of deformation described with the aid of the function $y = 0.25x^3 - 3.0595x^2 + 4.0476x + 46.857$, whose coefficient of the matching R^2 is about 0.9999 (Fig. 6).

The difference between microhardness of analysed steel in delivery state and after its deformation with 70% draft (238 $HV_{0.05}$) proves proceeding of strength hardening process (Fig. 5).

The strain hardening of X5CrNi18-8 steel is connected with occurrences of martensite α' phase in it structure. It was found that the microhardness of steel increase with the increasing of amount of α' phase. Fig. 7. show a linear dependence of the microhardness of the tested X5CrNi18-8 steel with the amount of α' martensite induced in cold rolling. This dependence was described by the approximating function: y = 10.277x + 218.35 whose coefficient of matching R^2 is about 0.7788 (Fig. 7).

On the basis of examinations of the mechanical properties it was found that with the increasing deformation of the X5CrNi18-8 steel the strength properties increases, while the plastic properties decrease proportionally to the degree of deformation during the cold rolling.

The X-ray investigation allowed to identify the phase composition of steel type X5CrNi18-8 in the delivery state and after cold rolling with draft degree from 10 to 70%. The results obtained for X5CrNi18-8 samples are presented in Fig. 8 and

in Table 2. On diffraction patterns of steel type X5CrNi18-8 in the delivered state disclosed diffraction lines coming from planes (111), (200), (220) and (311) austenite phases, that confirmed its homogeneous γ structure.



Fig. 7. Vickers microhardness as a function of α ' martensite content for the X5CrNi18-8 steel

First weak peak proceed from martensite α' phases were detected on diffraction patterns in steel deformed with a draft of about 10% on diffraction patterns on the basis of the diffraction lines according to identifications from (110) α' reflection planes, which occurred with matrix lines γ phase from (111) γ , (200) γ , (220) γ and (311) γ .

X-ray investigations of steel X5CrNi18-8 deformed with a draft of 20% also confirmed the occurrence of α ' martensite in its structure. α ' phases were detected on diffraction patterns on the basis of the diffraction lines according to identifications from (110) α ' and (211) α ' reflection planes, which occurred with matrix lines γ from (111) γ , (200) γ , (220) γ and (311) γ reflection planes (Fig. 8).

After cold rolling of steel with a draft of 30%, 40%, and 50% the intensity of austenite peaks gradually decreased and martensite α' peaks appeared in the spectrums. These spectra show a higher thickness reduction in the case of more numbers and a higher intensity of martensite α ' peaks. It was also found that with the increase of deformation the share of the reflection lines $(110)\alpha$ in the dual line with the reflection lines $(111)\gamma$ increases, too. It proves a distinct increase of α ' phase in the structure of the investigated steel. On diffractograms only phases γ and α ' were disclosed. There is no peak indicating the presence of ε martensite. Kumar et al. [29], working with cold rolled X5CrNi18-8 stainless steel, also did not detected the presence of ε martensite. Gey et al. [30] and Humbert et al.[31] characterized deformed X5CrNi18-8 samples and detected the presence of ε martensite. They have used a different apparatus. The transformation was performed in a sub-zero tension test with small deformations. It was showed that ε martensite is only a step of the transformation. Increasing deformation, it transforms to α'martensite. Probably, for the amount of deformation applied in this work, the reaction $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ has been completed. The amount of martensite increases with cold rolling deformation. X ray diffraction technique indicates 14, 46 and 60% of martensite for samples cold rolled 5, 26 and 47% respectively.

2.

Results of the X-ray phase analysis of the X5CrNi18-8 steel after deformation with a range 10-70%

Draft degree Ordinal [%]		Experimental			Identification (ICDD)			
	Angle of reflection [°2 Θ]	Interplanar distance d [Å]	Intensity I/I _{max} [%]	Interplanar distance d _{hkl} [Å]	Intensity [%]	(hkl)	Phase	
	1	51.075	2.0754	57	2.0750	100	111	γ
0	2	59.675	1.7982	65	1.7961	45	200	γ
	3	89.525	1.2703	100	1.2697	26	220	γ
	4	111.275	1.0836	41	1.0828	30	311	γ
10	1	51.175	2.0716	76	2.0750	100	111	γ
	2	52.275	2.0310	24	2.0268	100	110	α'
	3	59.825	1.7938	52	1.7961	45	200	γ
	4	89.625	1 2692	100	1 2697	26	220	ν
	5	111 425	1.0826	44	1.0828	30	311	ν γ
	1	51 175	2 0716	38	2 0750	100	111	1 2
	2	52 125	2.0710	21	2.0750	100	110	<u>a'</u>
	3	59.825	1 7938	21	1 7961	45	200	v
20	4	89.675	1.7550	100	1 2697	26	220	ν γ
	5	99.875	1.1688	17	1.1702	30	211	α'
	6	111.375	1.0830	26	1.0828	30	311	γ
	1	51.125	2.0731	42	2.0750	100	111	γ
	2	52.125	2.0361	39	2.0268	100	110	ά
30	3	77.075	1.4359	27	1.4332	20	200	α'
	4	89.625	1.2692	100	1.2697	26	220	γ
	5	99.425	1.1726	36	1.1702	30	211	α'
	6	111.625	1.0813	31	1.0828	30	311	γ
40	1	51.125	2.0678	47	2.0750	100	111	γ
	2	52.125	2.0361	40	2.0268	100	110	α'
	3	77.075	1.4359	29	1.4332	20	200	α'
	4	89.575	1.2698	100	1.2697	26	220	γ
	5	99.475	1.1723	36	1.1702	30	211	α'
	6	111.425	1.0826	30	1.0828	30	311	γ
50	1	51.175	2.0716	65	2.0750	100	111	γ
	2	52.325	2.0288	57	2.0268	100	110	α'
	3	76.975	1.4375	38	1.4332	20	200	α'
	4	89.675	1.2687	100	1.2697	26	220	γ
	5	99.475	1.1723	59	1.1702	30	211	α'
	6	111.175	1.0843	37	1.0828	30	311	γ
70 -	1	51.225	2.0694	57	2.0750	100	111	γ
	2	52.175	2.0346	53	2.0268	100	110	α'
	3	76.975	1.3921	51	1.4332	20	200	ά
	4	89.575	1.2689	100	1.2697	26	220	γ
	5	99.425	1.1726	94	1.1702	30	211	α
	6	111.475	1.0823	34	1.0828	30	311	γ

X-ray investigations of steel X5CrNi18-8 deformed with a maximum draft of about 70% disclosed diffraction lines coming from planes (110) α ', (200) α ' and (211) α ' martensite α ' phases, which occurred with matrix lines γ from (111) γ , (220) γ and (311) γ reflection planes.

Moreover, the intensities of $(111)\gamma$ and $(220)\gamma$ diffraction lines of Fe- γ phase in X5CrNi18-8 steel do not correspond to the standard intensities, so it could be that the effect of texture of Fe- γ phase is observed [32]. This phenomenon is decidingly the limiting factor determining the quantity of α ' and γ phases. Therefore, this problem is currently the subject of intensive research.

In agreement with X ray diffraction results, it was found that in structure of investigated steel the martensite transformation proceed according to the sequences $\gamma \rightarrow \alpha'$. The occurrence of martensite α' in X5CrNi18-8 stainless steel confirms the results of metallographic observations, magnetic measurements and mechanical investigations.



Fig. 8. X-ray diffraction patterns of steel X5CrNi18-8 in delivery and deformed state

The realized corrosive investigations with gravimetric method permit to determined the influence of degree of plastic deformation on unitary mass loss (V_c) and the linear corrosion rate (V_p) of cold rolled samples from austenitic steel type X5CrNi18-10 immersed in 30 wt% H₂SO₄ solution. The investigations were carried out in sulphuric solution (30 wt% H₂SO₄) because the behaviour of stainless steel in chloride solution (3.5 wt% NaCl) is already known. The mass loss of a samples (V_c) was counted with dependence (5), whereas the linear corrosion rate (V_p) was determined using the formula (6). The ratio including mass loss of a sample and linear corrosion rate carries out about 0.0462 for investigated steel grade X5CrNi18-10.

On the basis of the carried out corrosion test in 30 wt% H_2SO_4 solution it has been found that for steel in delivery state the value of the linear corrosion rate (V_p) was equal 4.5 mm/year and the mass loss (V_c) of about 96 g/m²day. That testifies of a little corrosive resistance and according to the standard EN ISO 7384:2001 steel X5CrNi18-8 in delivery state is characterized by eighth degree of corrosion resistances.

The results of the corrosion test samples after plastic deformation within the range from 10 to 70% causes a small worsening of resistance on corrosion, what marks by increases of both average value of V_p to about 5.0 mm/year and the average value of V_c to about 102 g/m²day. On the basis of calculation values of V_p as well as value of V_c and according to the standard PN-78/H-04608 steel demonstrate ninth degree of corrosion

resistance. It was found that cold rolled samples shows a little lower resistance on corrosion in 30 wt% H_2SO_4 . solution than material in delivery state.

Performed metallographic macroscopic investigations samples of X5CrNi18-8 steel immersed in sulphuric solution permitted to affirm that with the increases of plastic deformation of steel the resistance on pitting corrosion decreases. For investigated austenitic stainless steel the pitting index (PRE), determined using equation (4), was equal 19.6. The results of the macroscopic observations of surface samples after corrosion test in 30 wt% H₂SO₄ solution carried out for 25 days are presented in Figs. 9-15.



Fig. 9. Surface of undeformed samples after immersed in 30% H₂SO₄

The front surface of steel samples in delivery state after corrosion tests in sulphuric solution undergo a uniform corrosion and on its lateral surface some pits were observed (Fig. 9).

The macroscopic metallographic observations of sample surface after corrosion test using weight method and previously cold rolled with a draft of 10-50% showed diverse intensity of pits, micropores and corrosive cracks (Figs.10-14). Moreover, on the surface samples the effects of cold rolling were observed.



Fig. 10. Surface of samples deformed with a draft of 10% and immersed in $30\% H_2 SO_4$



Fig. 11. Surface of samples deformed with a draft of 20% and immersed in $30\% H_2 SO_4$



Fig. 12. Surface of samples deformed with a draft of 30% and immersed in $30\% H_2 SO_4$



Fig. 13. Surface of samples deformed with a draft of 40% and immersed in $30\% H_2 SO_4$



Fig. 14. Surface of samples deformed with a draft of 50% and immersed in $30\% H_2 SO_4$



Fig. 15. Surface of samples deformed with a draft of 70% and immersed in $30\% H_2 SO_4$

However sample from X5CrNi18-8 steel deformed with maximum draft, about 70% become almost total digestion as results of immersed in 30 wt% H_2SO_4 solution (Fig. 15).

Fractographic analyses of sample surface put to corrosion tests allowed to evaluate the type and the degree of corrosion damages.

The conducted morphology observations of surface samples of X5CrNi18-8 steel after exposition in 30 wt% H_2SO_4 solution for 25 days showed diverse character of corrosion products (Figs.16-19).

In delivery state on surface samples the local damages assume shape of cracks, fissure, micropores and pits were observed (Fig. 16).



Fig. 16. Micropores and corrosion pits on a specimen surface in delivery state after immersion tests in 30 wt% $\rm H_2SO_4$ solution



Fig. 17. Numerous, wide pits on a specimen deformed with a draft of 40 % after immersion tests in 30 wt% $\rm H_2SO_4$ solution

It was found, that sample of X5CrNi18-8 steel deformed with 40% draft degree and put to the corrosion test in 30 wt% H_2SO_4

solution revealed on surface a wide pits with diversified size, cracks as well as linear arranged corrosion failure. Beside the numerous micropores were disclosed on surface samples (Figs. 17, 18). Additionally on samples surface the effect of cold rolling was observed.

Cracked passive layer was also observed, what could be a result of rapid penetration of corrosive medium into interior of investigated specimens (Fig. 19).



Fig. 18. Corrosion pits on a specimen deformed with a draft of 40 % after immersion tests in 30 wt% H_2SO_4 solution; higher magnification of the steel surface shown in Fig. 19



Fig. 19. Cracked passive layer on a surface of the cold rolled specimen with a draft of 40% after immersion tests

4. Conclusions

The obtained results of investigations lead to the following conclusions:

- 1. In the delivery state the X5CrNi18-8 steel structure discloses typical grains of γ solution with about 21 µm in diameter, ensuring the following mechanical properties: tensile strength R_m about 647 MPa, yield strength $R_{p0.2}$ about 330 MPa, elongation *A* about 52%, reduction of area *Z* about 58% and microhardness about 162 HV_{0.05}.
- 2. Plastic deformation of steel X5CrNi18-8 in cold rolling with a draft from 10 to 70% causes the increase of the strength properties: R_m from 785 MPa to 1496 MPa, $R_{p0.2}$ from 586 MPa to 1161 MPa and microhardness from 227 HV_{0.05} to 400 HV_{0.05} and decreasing of plasticity *A* from 32% to 0.68%, Z from 45% to about 11%.
- 3. The high mechanical properties of the investigated cold rolled steel determined the structure of elongated austenite grains with martensite α ' phase with a microhardness 400 HV_{0.05}, resulting from the transformation of the induced plasticity.
- 4. The amount of α 'phase in X5CrNi18-8 steel depends on the degree of plastic deformation. The increasing deformation within the range of 10-70 % induces in its structure a martensitic transformation $\gamma \rightarrow \alpha$ ', increasing part of the α 'phase from 1.30 to about 21.0 %.
- 5. X-ray investigations of austenitic steel deformed with draft from 10 to 70% confirmed the occurrence of α' martensite in the steel structure. α' phases were detected on diffraction patterns on the basis of the diffraction lines according to identifications from (110) α' and (211) α' reflection planes, which occurred with matrix lines γ from (111) γ , (220) γ and (311) γ reflection planes.
- 6. Corrosion tests carried out using weight method revealed that X5CrNi18-8 steel showed a little corrosion resistance in sulphuric solution, what is connected with a occurrence of martensite α '.
- Metallographic observations of samples surface after corrosion tests in 30 wt% H₂SO₄ showed corrosion damages diversity.

References

- [1] S. Lamb, Handbook of Stainless Steel and Nickel Alloys, CASTI Publishing INC./ASM International, 2001.
- [2] A. Baron, W. Simka, G. Nawrat, D. Szewieczek, A. Krzyżak, Influence of electrolytic polishing on electrochemical behaviour of austenitic steel, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 55-58.
- [3] A. Kurc, E. Kalinowska-Ozgowicz, The influence of the martensite α' phase occurring in the structure of cold rolled austenitic Cr-Ni steel on its mechanical properties, Archives of Materials Science and Engineering 37/1 (2009) 21-28.
- [4] L.A. Dobrzański, Z. Brytan, M.A. Grande, M. Rosso, Corrosion resistance of sintered duplex stainless steel evaluated by electrochemical method, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 317-320.

- [5] L. Peğuet, B. Malki, B. Baroux, Influence of cold working on the pitting corrosion resistance of stainless steels, Corrosion Science 49 (2007) 1933-1948.
- [6] W. Ozgowicz, A. Kurc, Structure and properties of forming austenitic X5CrNi18-9 stainless steel in a cold working, Journal of Achievements in Materials and Manufacturing Engineering 33/1 (2009) 19-26.
- [7] H. Abreu, S. Carvalho, P. Neto, R. Santos, V. Freire, P. Silva, S. Tavares, Deformation inducted martensite in an AISI 301LN stainless steel: Characterization and influence on pitting corrosion resistance, Materials Research 10 (2007) 359-366.
- [8] A. Pardo, M.C. Merino, M. Carboneras, A.E. Coy, Pitting corrosion behaviour of austenitic stainless steels with Cu and Sn additions, Corrosion Science 49 (2007) 510-525.
- [9] A. Kurc, Z. Stokłosa, Some mechanical and magnetic properties of cold rolled X5CrNi18-8 stainless steel, Archives of Materials Science and Engineering 34/2 (2008) 89-94.
- [10] W. Ozgowicz, E. Kalinowska-Ozgowicz, A. Kurc, Influence of plastic deformation on structure and mechanical properties of stainless steel type X5CrNi18-10, Journal of Achievements in Materials and Manufacturing Engineering 32/1 (2008) 37-40.
- [11] Xu Chunchun, Hu Gang, Effect of deformation-induced martensite on pit propagation behavior of 304 stainless steel, Anti-Corrosion Methods and Materials 51 (2004) 381-388.
- [12] M. Opiela, A. Grajcar, W. Krukiewicz, Corrosion behaviour of Fe-Mn-Si-Al, Journal of Achievements in Materials and Manufacturing Engineering 33/2 (2009) 159-165.
- [13] A. Pardo, M.C. Merino, A.E. Coy, R. Arrabal, F. Viejo, A. M'hich, Corrosion behaviour of AISI 304 stainless steels with Cu coatings in H₂SO₄, Applied Surface Science 253 (2007) 9164-9176.
- [14] L. Zhilin, L. Wei, Q. Juncai, The effect of electrochemically induced annealing on the pitting resistance of metastable austenitic stainless steel, Metallurgical and Materials Transactions A 37 (2006) 435-439.
- [15] B.R. Kumar, B. Mahato, R. Singh, Influence of cold-worked structure on electrochemical properties of austenitic stainless steels, Metallurgical and Materials Transactions A 38 (2007) 2085-2094.
- [16] M. Rutkowska-Gorczyca, M. Podrez-Radziszewska, Influence of cold working process on the corrosion resistance of steel 316L, SIM XXXVII (2009) 319-323.
- [17] J. Baszkiewicz, M. Kamiński, Fundamentals of materials corrosion, Warsaw University of Technology Publishers, Warsaw. 1997 (in Polish).
- [18] M. Ahlers, The Martensitic Transformation, Revista Materia 9 (2004) 169-183.
- [19] E. Perdahcioglu, H. Geijselaers, Influence of plastic strain on deformation-induced martensitic transformations, Scripta Materialia 58 (2008) 947-950.
- [20] European Standard, Stainless steels Part 1: List of stainless steels; Polish version PN-EN 10088-1:2007.
- [21] G.F. Vander Voort, Metallography: Principles and Practice, ASM International, Materials Park, Ohio, 1999.

- [22] European Standard, Steels Micrographic determination of the apparent grain size, Polish version PN-EN ISO 643:2005.
- [23] European Standard, Welding Determination of Ferrite Number (FN) in austenitic and duplex ferritic-austenitic Cr-Ni stainless steel weld metals, Polish version PN-EN ISO 8249:2005.
- [24] European Standard, Tensile Testing of Metallic Materials -Part 1: Method of Test at Ambient Temperature; Polish version PN-EN 10002-1+AC1:2004.
- [25] European Standard, Metallic materials Vickers hardness test - Part 1: Test method; Polish version PN-EN ISO 6507-1:2007.
- [26] European Standard, Corrosion test in artificial atmosphere -General requirements; Polish version EN ISO 7384:2001.
- [27] European Standard, Determination of resistance to intergranular corrosion of stainless steels - Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels corrosion test in media containing sulphuric acid; Polish version EN ISO 3651-2:1998.

- [28] B. Cullity, Elements of X-ray Diffraction, Addison-Wesley Series in Metallurgy and Materials, 1967 (in Polish).
- [29] B. Ravi Kumar, A.K. Singh, B. Mahato, P.K. De, N.R. Bandyopadhyay, D.K. Bhattacharya, Deformationinduced transformation textures in metastable austenitic stainless steel, Materials Science and Engineering A 429 (2006) 205-211.
- [30] N. Gey, B. Petit, M. Humbert, Electron backscattered diffraction study of ϵ/α' martensitic variants induced by plastic deformation in 304 stainless steel, Materials and Metallurgical Transactions A 36 (2005) 3291-3299.
- [31] M. Humbert, B. Petit, B. Bolle, N. Gey, Analysis of the γ→ε→α' variant selection induced by 10% plastic deformation in 304 stainless steel at -60°C, Materials Science and Engineering A 454-455 (2007) 865-867.
- [32] K. Pałka, A. Weroński, K. Zalewski, Mechanical properties and corrosion resistance of burnished X5CrNi18-9 stainless steel, Journal of Achievements in Materials and Manufacturing Engineering 16 (2006) 57-62.