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Structure and properties of forming austenitic X5CrNi18-9 stainless steel in a cold working

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ABSTRACT

Purpose: The aim of the paper is to analyze the influence of the degree of rolling reduction on the structure forming and changes of mechanical properties in cold-rolled sheet-metals of austenitic X5CrNi18-9 stainless steel.

Design/methodology/approach: The examinations contained metallographic observations of the structure on a light microscope and on the scanning electron microscope (SEM), researches of mechanical properties in a static tensile test and microhardness measurements made by Vickers's method. The analysis of the phase composition was carried out on the basis of X-ray researches. In the qualitative X-ray analysis the comparative method was applied. Fractographic tests of the fracture after the decohesion of samples in a static tensile test at room temperature were executed in a SEM.

Findings: It has been found that plastic deformation in a cold working of austenitic stainless steel type X5CrNi18-9 induced in its structure martensitic transformation $\gamma \rightarrow \alpha'$. The occurrence of martensite phases 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 from 10% to 70%. Moreover, the results of the X-ray quantitative analysis allowed to determine the proportional part of martensite phases α ` in the structure of investigated steel in the examined range of cold plastic deformation.

Practical implications: The analysis of the obtained results permits to state that the amount of martensite phases α ' in the investigated steel structure increases with the degree of deformation in the cold rolling. Besides, a good correlation was found between changes of the structure and the effects of investigations of the mechanical properties.

Originality/value: Good correlation between changes of the structure and the effects of investigations of the mechanical properties in the austenitic X5CrNi18-9 stainless steel was found.

Keywords: Metallic alloys; Austenitic stainless chromium-nickel steel; Plastic deformation; Intergranular fracture; Structure and mechanical properties; Austenite and α '- martensite phases; Cold rolling

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1. Introduction

The dynamic development of technology creates the necessity for producing steels with much better mechanical and plastic properties, about the high corrosion resistance, for chemical, machinery, food, automobile, nuclear and shipbuilding industry [1-4].

Commonly it is known that stable corrosion resistance of steels with austenitic structure and chemical composition: 17-25%wt. chromium, 8-30%wt. nickel, max. 0.15%wt. carbon and up to 7%wt. molybdenum, have a very good ductility, formability and resistance to corrosion. This type of steel can be highly strengthened by cold work processes. They are inherently paramagnetic but since martensite is a ferromagnetic phase, the transformation of austenite to martensite makes them ferromagnetic. These steels are known as TRIP steels which is an acronym of transformation induced plasticity. This transformation depends mainly on two factors: stacking fault energy (SFE) differences between austenite and martensite and external applied stress. During the deformation process different transformation sequences take place, such as: $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ or $\gamma \rightarrow \alpha'$. The amount of ε and/or α ' martensite depends on the processing parameters, as stress state, temperature, rate of deformation and the material chemical composition. The metastability of austenitic stainless steels increases with the decrease of SFE [5-11].

Stainless steel with an austenite structure is also characterized favourably by plastic properties at low temperature, because they do not undergo any changes in the brittle state with the lowering of the temperature for the sake of the structure FCC. However the major defect of these steels is a large content of expensive chromium and nickel. Currently applied production technologies of these steels are more and more material- and energy-saving and typical kinds of them contain about 18%wt. chromium and up to 11%wt. nickel (e.g. X2CrNiMo17-13 or X15CrNiSi20-14) [12-19].

Standard austenitic stainless steels used for sheet-metal, e.g. for automotive industry type X5CrNi18-8 contain maximally 18%wt. chromium and only 8-9%wt. nickel. Near to the controlled content of carbon to 0.04%wt. and nitrogen to 0.05%wt. these steels show generally the highest value of the quotient of plastic properties (A about 50-60%) to the yield point ($R_{p0.2}$ about 300-400 MPa) in comparison with different suitable constructional materials currently applied in the techniques, for example: Bake Hardening Steel (11180B, 11260B), Duplex Steel (1.4462), Dual Phase (H300X) and High Strength Low Alloy-HSLA (H320LA) or also some Al-Mg alloys [20-26].

Thus, the aim of the investigations is to determine the influence of cold rolling on the structure and mechanical properties forming in the austenitic stainless steel type X5CrNi18-9, as well as the character of the fracture obtained during the decohesion of samples in a static tensile test at room temperature.

2. Experimental procedure

Experiments were carried out on low carbon metastable austenitic stainless steel type X5CrNi18-9, according to PN-EN 10088:2007 [27], with the following nominal composition:

C = 0.033%, Cr = 18.08%, Ni = 9.03%, Mn = 1.32%, Si = 0.41%, Mo = 0.23%, N = 0.026%, P = 0.026%, S = 0.002% and the balance Fe. The investigated material was supplied in the form of sheet-cutting steel with dimension about $40 \times 2 \times 700$ mm, as a result of industrial smelting from the UGINE&ALZ (Poland). The material was cold-rolled within the range 10%-70% and samples for researches of the mechanical properties, for microhardness measurements, metallographic observations and the X-ray phase analysis were cut. The rolling was conducted at room temperature (about 20°C) keeping a constant direction and side of the rolled strip. Small unitary draft was applied with a different number of roll passes to ensure a summary draft for every single strip.

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

Evaluation of the mechanical properties was made on the basis of measurements of statically tensile tests on the universal testing machine ZWICK 100N5A. Dimensions of test samples were determined on the basis of PN-EN 10002-1+AC1:2004 [28] standard and cut from the steel sheet parallelly to the rolling direction.

The microhardness measurements of the investigated cold reduced sheets from steel X5CrNi18-9 were carried out by a microhardness tester PMT-3 produced by Hauser, according to the standard PN-EN ISO 6507-1:2007 [29]. Researches were made by Vickers's method on metallographic samples with a load of 50g.

Metallographic investigations of the structure were carried out on longitudinal polished microsections of X5CrNi18-9 steel samples applying the optical microscope LEICA MEF4A, equipped with a Leica Qwin image analyzer. In order to distinguish martensite from austenite and to detect nonmetallic inclusions the metallographic specimens were etched in the reagent Mi17Fe. The etching solution was composed of 10cm³ HNO₃, 100cm³ HCl, 90cm³ distilled water, heated to a temperature of about 40°C. Metallographic observations were carried out with a magnification of 100-1000x.

In order to study the structural changes taking place during cold plastic deformation on steel X5CrNi18-9 X-ray phase analysis has been used applying the filter radiation of an anode CoK_{α}. X-ray researches were run by means of an X'PERT PANalytica 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 40° 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 Diffract AT Search/Match.

X-ray quantitative phase analysis was carried out by the Averbach Cohen method [30]. In the calculation of the quantitative share of the phase α ' the respective surfaces of the diffraction lines of the phases γ and α ' were measured by means of a planimeter.

The fractographic investigation of the fracture after decohesion of the samples in a tensile test at room temperature was executed in the electron scanning microscope SUPRATM25 produced by ZEISS with the accelerating voltage 20kV, applying the magnification of 1000-75000x.

3. Results and discussion

The metallographic investigations permit to define the influence of the degree of plastic deformation on the structure of austenitic stainless steel type X5CrNi18-9 and in particular to determine the metallographic symptom of draft, shape and size of austenite grains. The results of metallographic observation have been presented on microphotos (Figs. 1-4). The structure of the investigated steel in the delivery state shows equiaxial austenite grains about 22 μ m average in diameter with many annealed twins and some non-metallic inclusions (Fig.1), mainly about the pointwise character, which identify as oxides, carbonitrides and globular silicate according to the standard.

The structure of the examined X5CrNi18-9 steel in the delivery state doesn't show the metallographic symptom of deformation and its characteristic for stable state, for example after supersaturation.



Fig. 1. Supersaturated austenite structure of X5CrNi18-9 steel in the delivered state; Etching - Mi17Fe; Mag. 1000x



Fig. 2. Structure of the investigated steel after deformation with draft of 20%; Etching - Mi17Fe; Mag. 1000x

In the investigated steel after cold plastic deformation with drafts up to 20% a structure of elongated austenite grains with slip bands, deformation twins and non-metallic inclusions was found (Fig.2). After 30% cold working of the investigated steel structure apart from elongated γ grains with deformation twins, some non-metallic inclusions and few areas of parallel plates characteristic for martensite α ' were observed (Fig.3).

Observations of the structure of steel type X5CrNi18-9 with a degree of deformation, about 40% to 70% shows that in elongated γ grains there are martensite α ' plates (Fig.4).

Considerable cold plastic deformation on the investigated steel X5CrNi18-9 causes privileged crystallographic orientation of the grain in relation to the direction of the plastic working. During the cold rolling process with an increasing degree of deformation the α ' phase is formed, which causes an essential size reduction of the steel structure and its strain hardening. The occurrence of martensite α ' in X5CrNi18-9 steel confirms the results of mechanical investigations.



Fig. 3. Structure of the investigated steel after deformation with draft of 30%; Etching - Mi17Fe; Mag. 1000x



Fig. 4. Structure of the investigated steel after deformation with draft of 70%; Etching - Mi17Fe; Mag. 1000x

The effect of static tensile tests permitted to quantify the influence of the degree of deformation on the mechanical properties of austenite X5CrNi18-9 stainless steel. The results of investigations on the mechanical properties after a tensile test and microhardness measurements have been gathered in Table 1 and in the curves presented in Figs. 5 and 6.

It has been found, that the value of the yield point $R_{p0.2}$, tensile strength R_m and microhardness $HV_{0.05}$ increase with the degree of deformation, but the value of necking Z and elongation A decreases.

The yield point of the investigated steel in the not deformed state is about 300 MPa, the tensile strength about 624 MPa, microhardness about $183HV_{0.05}$, the elongation about 53% and the necking about 54%. With the increasing deformation within the range of 10%-70% the yield point of X5CrNi18-9 steel increases from about 542 MPa to about 1148 MPa, the tensile strength from about 783 MPa to about 1452 MPa, the hardness from about 279 HV_{0.05} to 554 HV_{0.05}, while the elongation decreases from about 35% to about 1% and the necking from about 50% to about 10%.

Table 1.

Micro-hardness and mechanical properties of cold rolled X5CrNi18-9 stainless steel after tensile test

| Degree of plastic deformation [%] | Mechanical properties | | | | | |
|--|-----------------------|--------------------------|--------|--------|------------------------|--|
| | R _m MPa | R _{p0,2} MPa | A % | Z % | $\overline{HV}_{0.05}$ | |
| 0 | 624 | 300 | 53 | 54 | 183 | |
| 10 | 783 | 542 | 35 | 50 | 279 | |
| 20 | 906 | 767 | 24 | 45 | 316 | |
| 30 | 1010 | 877 | 4 | 35 | 405 | |
| 40 | 1141 | 967 | 3 | 23 | 425 | |
| 50 | 1228 | 1059 | 2 | 13 | 445 | |
| 70 | 1452 | 1148 | 1 | 10 | 554 | |



Fig. 5. Changes of the mechanical properties in the investigated cold rolled steel depending on degree of deformation



Fig. 6. Changes of the plasticity in the investigated cold rolled steel depending on degree of deformation

On the basis of the realized investigations of the mechanical properties it was found that with the increasing deformation of X5CrNi18-9 steel the strength properties increase, while the plastic properties decrease proportionally to the degree of deformation during the cold rolling.

X-ray investigations of steel X5CrNi18-9 deformed with a draft from 10% to 70% 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 ', (200) α ' and (211) α ' reflection planes, which occurred with matrix lines γ phase from (111) γ , (220) γ and (311) γ reflection planes. It was also found that with the increase of deformation the share of the reflection lines (110) α ' in the dual line with the reflection lines (111) γ increases, too. It proves a distinct increase of the α ' phase in the structure of the investigated steel. The occurrence of the reflection lines (110) α ' in diffraction patterns influences essentially the improvement of the procedure used in the phase analysis. The results of the X-ray phase analysis have been gathered in Table 2 and in Fig. 7.

The analyzed diffraction lines $(111)\gamma$, $(220)\gamma$, $(311)\gamma$ and $(110)\alpha'$, $(200)\alpha'$, $(211)\alpha'$, analysis phases of cold rolled austenite X5CrNi18-9 stainless steel with 70% draft shows distinct texturing (Fig. 7). This phenomenon is decidingly the limiting factor determining the quantity of α' and γ phases.

On the basis of X-ray quantitative phase analysis it was found that the amount of the analyzed α' phase in the investigated steel structure increases with the deformation in the cold rolling process. In the undeformed state of X5CrNi18-9 steel the α' phase does not occur, but after deformation with a maximum draft of about 70% the amount of martensitic phases is about 40% (Fig. 8). Besides, it was found that in the deformed condition with draft from 10% to 70% content of α' martensite phase in the structure of investigated steel is on the average about 27%.

The results of fractographic examinations permitted to determine the influence of the degree of deformation within the range 10%-70% on the character of the fracture of X5CrNi18-9 steel obtained during the decohesion of samples in a tensile test at room temperature. Findings of fractographic observations were introduced on microfractographies (Figs. 9-13).

| Results of the X-ray | y phase analysis of the ii | ivestigated steel with a c | Iraft of 70% | | | | |
|----------------------|------------------------------|------------------------------|-------------------------------------|--|------------------|-------|-------|
| | Experimental | | | Identification (ICDD) | | | |
| Ordinal number | Angle of reflection 2Θ[°] | Interplanar distance d[Å] | Intensity I/I _{max} [%] | Interplanar distance d _{hkl} [Å] | Intensity [%] | (hkl) | Phase |
| 1 | 51.117 | 2.0742 | 100 | 2.075 | 100 | 111 | γ |
| 2 | 52.121 | 2.0374 | 47 | 2.0268 | 100 | 110 | α' |
| 3 | 77.043 | 1.4372 | 37 | 1.4332 | 20 | 200 | α' |
| 4 | 89.580 | 1.2705 | 96 | 1.2697 | 26 | 220 | γ |
| 5 | 99.360 | 1.1739 | 55 | 1.1702 | 30 | 211 | α' |
| 6 | 111.368 | 1.0837 | 29 | 1.0828 | 30 | 311 | γ |

| Table 2. | | | | | | |
|----------------------------|-------------|---------------|--------------|-----------|----------|----|
| Results of the X-ray phase | analysis of | f the investi | igated steel | with a dr | aft of 7 | 0% |



Fig. 7. X-ray diffraction patterns of steel X5CrNi18-9 with a draft of 70%

In the delivery state in the X5CrNi18-9 steel the ductile fracture with characteristic smoothing convexities and craters occurred (Fig. 9).

After cold rolling with a draft of 20% in the investigated steel there is to be observed a transcrystalline ductile fracture with large areas of plastic deformation and small areas of brittle cracking. Characteristic craters with diversified size and few small separations on plastic deformed surfaces have been found (Fig. 10). Whereas in areas of brittle cracking probably α ' phase occurred. In those places parted flat surfaces were observed with visible lines and ridges. Rolling with a draft of 30% and 40% causes, that in X5CrNi18-9 steel fracture areas of plastic deformation occurred and the large quantities skips distance about smooth surfaces connected with the presented α ' phase. These skips distance are probably the borders of phases γ and α ' (Fig. 11).

Martensite α ' phase existing in X5CrNi18-9 steel structure creating on its fractures smooth terraces (Fig. 12) separated originate from austenite γ phase terraces with surfaces showing plastic deformations (Fig. 13).



Fig. 8. Dependence of the volume fraction of α ' phase on the degree of plastic deformation of the investigated steel



Fig. 9. Ductile fracture of X5CrNi18-9 steel in the delivery state, Mag. 1000x



Fig. 11. Mixed fracture of investigated steel type X5CrNi18-9 after deformation with a draft of 30%, Mag. 5000x



Fig. 10. Transcrystalline ductile fracture of investigated steel type X5CrNi18-9 after deformation with a draft of 20%, Mag. 5000x

Observations on the scanning electron microscope additionally give metallographic microsections of investigated X5CrNi18-9 steel, applying the magnification 5000x and 8000x.

On the basis of the realized investigations it was found that the martensite α ' phase occurs in cold rolled X5CrNi18-9 steel structure with a draft of 40%. Disclosure on microphotographies (Figs.14 and 15) areas with small parallel lines are characteristic for the occurring martensite α ' phase.



Fig. 12. Terraces on the ductile fracture of investigated steel type X5CrNi18-9 after deformation with a draft of 30%, Mag.75000x

4. Conclusions

Based on the analysis of the obtained results of investigated stainless steel type X5CrNi18-9 in the delivery state and after cold rolling process allowed to formulate the following statements:

 In the delivery state the X5CrNi18-9 steel structure discloses typical grains of γ solution with about 22 µm in diameter,

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ensuring the following mechanical properties: tensile strength Rm about 624 MPa, yield strength $R_{p0.2}$ about 300 MPa, elongation A about 53%, reduction of area Z about 54% and hardness about 183 HV0.05.

- The cold rolling of sheet-metal with a thickness of 2 mm within the range 10%-70% causes that in the investigated steel structure the martensite α' phase is formed.
- The occurrence of this phase essentially influences an increase of the value of Rm to about 1452 MPa, R_{p0.2} to about 1148 MPa and HV0.05 to about 554 HV0.05 but decreases A to about 1% and Z to about 10%.



Fig. 13. Ductile fracture of investigated X5CrNi18-9 steel after deformation with a draft of 30%, Mag. 20000x



Fig. 14. Elongated γ grains with areas of martensite α ' and some non-metallic inclusions in investigated steel after deformation with a draft of 40%; Etching - Mi17Fe; Mag. 5000x



Fig. 15. Areas of parallel plates characteristic for martensite α ' in the investigated steel structure after cold rolling with a draft of 40%; Etching - Mi17Fe; Mag. 8000x

- The X-ray phase analysis disclosed in the structure of the investigated cold rolled X5CrNi18-9 steel the occurrence of the martensite α' phase, whose quantity increases with the increasing degree of plastic deformation.
- Deformation of steel with a maximum degree of draft 70% leads to about 40% part quantitative martensite α' phase in the structure of the investigated steel.
- The occurrence of martensite α' phase in the studied steel is confirmed the results of metallographic observations on microsections by the scanning electron microscope.
- The cold rolling of investigated X5CrNi18-9 steel with a draft of about 40% leads to obtained a mixed fracture with large areas of brittle cracking.

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