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## THE EFFECT OF SOLUTION TREATMENT AND NITROGEN ADDITION ON PHASE COMPOSITION OF DUPLEX STEELS

## WPŁW TEMPERATURY PRZESYCANIA I ZAWARTOŚCI AZOTU NA SKŁAD FAZOWY STALI DUPLEX

The present investigations concern the estimation of the phase composition and the analysis of the ferrite-austenite morphology after solution annealing of hot-rolled sheets of duplex type steels. Two commercial grades of nitrogen alloyed ferritic-austenitic stainless steels were examined. The first one, X2CrNiMoN22-6-3, is the mid-alloy duplex steel with nitrogen addition 0.19%. Its chemical composition corresponds to the commercial grade SAF 2205. The second steel, X3CrNiMoCuN25-6-4, is the high-alloy super duplex stainless steel with nitrogen contents 0.26% and chemical composition close to the commercial grade UR52N+.

It was found that solution annealing within the temperature range 1200°C-1050°C markedly changes the volume fractions and hence the morphology of the austenitic phase. The arrangement of the austenite bands or elongated areas on the background of ferrite matrix remains essentially unchanged in comparison to that after hot-rolling. However their shape and continuity strongly depend on the austenite volume fractions, which result from the applied annealing temperature. The addition of nitrogen, which is strong austenite stabilizer, shifts the temperature range of austenite precipitation to higher temperatures and is responsible for the higher austenite volume fractions in the case of both steels.

Keywords: duplex stainless steels, hot-rolled sheets, solution annealing, phase composition, ferrite-austenite morphology, band-like structure

Prezentowane badania dotyczą oceny składu fazowego i analizy morfologii struktury dwufazowej po wyżarzaniu i przesycaniu walcowanych na gorąco blach ze stali typu duplex. Badano dwa komercyjne gatunki ferrytyczno-austenitycznych stali nierdzewnych z dodatkiem azotu. Pierwsza z nich, X2CrNiMoN22-6-3, jest średnio-stopową stalą duplex o zawartości azotu 0.19% a jej skład chemiczny odpowiada gatunkowi SAF 2205. Druga stal, X3CrNiMoCuN25-6-4, jest to wysoko-stopowa stal nierdzewna typu superduplex z dodatkiem 0.26% azotu i składzie chemicznym, który jest zbliżony do składu stali w gatunku UR52N+.

Stwierdzono, że wyżarzanie w zakresie temperatur 1200°C-1050°C w znaczący sposób zmienia udziały objętościowe a w związku z tym morfologię austenitu. Układ pasm lub wydłużonych obszarów austenitu na tle osnowy ferrytu pozostaje zasadniczo niezmieniony w stosunku do tego po walcowaniu na gorąco. Jednakże ich kształt oraz mniej lub bardziej ciągły charakter w istotny sposób zależą od ilości austenitu, która wynika z zastosowanej temperatury wyżarzania. Dodatek azotu, który jest pierwiastkiem silnie stabilizującym austenit, przesuwa zakres wydzielania austenitu do wyższych temperatur i jest odpowiedzialny za zwiększone udziały objętościowe austenitu w obu stalach.

## 1. Introduction

An increasing development of duplex stainless steel (DSS) grades is observed in the recent few decades after recognising the fact that DSS combine the best properties of both constituent  $\alpha$ - and  $\gamma$ -phases [1-4]. The phase composition, owing to which DSS received their proper name, comprises approximate proportions of the ferritic  $\alpha$ -phase (bcc structure) and the austenitic  $\gamma$ -phase (fcc structure) and the volume fractions of both phases usually oscillate within the range 40-60%. The chemi-

cal compositions are frequently related to the Fe-Cr-Ni ternary system. Typical contents of chromium and nickel are within the range 18-26 wt.% Cr and 4-8 wt.% Ni and the carbon content is usually very small (C<0.03 wt.%). In modern grades of DSS the routine alloying element is molybdenum (up to 4.0 wt.% Mo) or molybdenum and tungsten, as well as additions of copper (up to 1.5 wt.% Cu) and nitrogen (0.1-0.3 wt.% N) [1-4].

The whole group of DSS includes a number of grades, whose corrosion resistance depends on the contents of alloying additions. The most frequently applied

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classification criterion is based on the so-called Pitting Resistance Equivalent Number (PREN), which is determined on the basis of the chemical composition according to the formulae of the form [1-4]:

$$PREN = \%Cr + 3.3x(\%Mo + 0.5\%W) + 16xN \quad (1)$$

In consequence the following categories of DSS are distinguished, namely; low-alloy steels (PREN<32), mid-alloy steels (PREN=32-39) and high-alloy, so-called superduplex steels (PREN>39). Modern grades of DSS exhibit very good functional properties due to high corrosion resistance (various types of corrosion), high strength, good plasticity and toughness as well as relatively high heat conductivity and low coefficients of thermal expansion [1-3].

Processes of hot-deformation with intermediate and/or subsequent annealing are usually one of the first manufacturing steps for DSS. These processes affect both, the specific two-phase microstructure which develops in the course of deformation as well as the phase composition resulting from the applied temperatures [5-7]. Each of these structural aspects is very important from the view point of the material behaviour upon further treatment as well as final mechanical properties and their anisotropy [4-8]. That is why the purpose of the present research was to analyse the effect of the temperature of solution annealing on the ferrite and austenite volume fractions and the morphology of the ferrite-austenite microstructure in hot-rolled sheets of DSS. The effect of nitrogen addition was also taken into account since two commercial grades of DSS were examined, i.e. mid-alloy duplex steel and high-alloy superduplex stainless steel, with different nitrogen contents.

### 2. Material and experimental procedure

The present research concerns two commercial grades of the ferritic-austenitic stainless steels of duplex type, i.e. X2CrNiMoN22-6-3 and X3CrNiMoCuN25-6-4 with the chemical compositions given in Table 1.

The first of the examined steels, X2CrNiMoN22-6-3 (denoted by F), is the mid-alloy DSS with 0.19 wt. % of nitrogen addition. The PREN value, calculated on the basis of chromium, molybdenum and nitrogen contents (Eq. 1) is about 35. The chemical composition of steel F corresponds to the commercial grade SAF 2205 (UNS S32205) [1, 4], that is the grade which is today the most frequently produced DSS.

The second steel X3CrNiMoCuN25-6-4 (denoted by S) is the high-alloy DSS with 0.26 wt.% addition of

nitrogen and the PREN value of about 41. The chemical composition of steel S is very close to the grade UR52N+ (UNS S32520), that is the superduplex type of stainless steel. These steels have the higher contents of alloying elements stabilizing ferrite, i.e. chromium and molybdenum. Hence, the increased additions of austenite stabilizers; like nitrogen, nickel and copper, to equalize the proportions of ferrite and austenite [2, 3].

TABLE 1 Chemical compositions of the ferritic-austenitic steels under examination (in wt. %)

Steel	C	Cr	Ni	Мо	N	Cu	W	Mn
F	0.02	22.28	6.05	2.95	0.19	0.12		1.72
S	0.03	25.12	5.82	3.59	0.26	1.49	0.023	0.87

Steel	Si	Al	Со	Nb	V	Р	S	PREN
F	0.51	0.03		0.02	0.11	0.014	0.005	35
S	0.27	0.13	0.068	0.017	0.096	0.018	0.001	41

The material was received in the form of industrially hot-rolled sheets with the thickness within the range 12.5-13.5 mm. The specimens cut of the sheets were subjected to solution treatment. The process included annealing at the temperatures 1200°C, 1150°C, 1100°C and 1050°C for 3 hours with subsequent water quenching.

Microstructure observations of the as received material, that is after hot-rolling (Fig. 2), and after subsequent solution treatment (Figs. 3 and 4) were carried out by means of optical microscopy (Neophot-2), on the (ND-RD) longitudinal sections (LS) and the (ND-TD) cross sections (CS) of the sheets. A considerable number of micrographs from both sections was registered to select the most representative microstructures for each variant of treatment. Afterwards, the estimation of the phase composition and the analysis of two-phase morphology were conducted on the basis of at least 5 representative micrographs for each section, that is about 10 microstructures for each variant of thermo-mechanical treatment. The image analysis was performed by employing computer program MET-ILO, version V.9.11 [9].

Since for the most cases austenite occurred the second phase observed against the background of ferrite matrix, the estimation of the phase composition was based on the assessment of the austenite volume fraction  $(V_V^A)$ . That is why the measurements of the area fractions of the  $\gamma$ -phase  $(A_A^A)$  were conducted. Similarly, the characterization of the specific ferrite-austenite banded structure based on the analysis of the austenite morphology. Two complementary and simple parameters were used, namely; the so-called elongation factor  $(L_E^A)$  and Feret's ratio  $(R_F^A)$ . Schematic illustration explaining differences between both parameters are shown in Figure 1. Feret's ratio is actually the quotient of Feret's diameters ( $R_F^A = Fx/Fy$ ) in two perpendicular directions. The Feret's diameter is the length of the object contour projection on the X or Y axis, which correspond with the RD and ND directions respectively on the longitudinal sections (LS) and TD and ND directions on the cross-sections (CS) of the sheets [8]. The so-called elongation factor is in fact the ratio of the maximum and minimum dimensions of the object contour ( $L_E^A = D_{max}/D_{min}$ ), irrespective of the external co-ordinate system [9].



Fig. 1. Schematic illustration for the measurement and calculation of the so-called elongation factor ( $L_E$ ) and the ratio of Feret's diameters ( $R_F$ )

The qualitative elemental analysis was additionally performed by means of scanning electron microscopy using Energy Dispersive X-ray Spectroscopy (EDS) method. The analysis concerned concentration of major alloying additions, i.e. Cr, Ni and Mo, within the ferrite and austenite areas to estimate the level of element partitioning in both phases after solution annealing at the temperatures 1200°C-1050°C [10, 11].

## 3. Results and discussion

## 3.1. Microstructure after hot-rolling

The specific character of the initial ferrite-austenite morphology is the result of the hot-deformation process applied in the case of both examined duplex steels. After hot-rolling the ferritic  $\alpha$ -phase constituted the matrix with elongated islands and bands of the austenitic  $\gamma$ -phase parallel to the rolling plane (Fig. 2 a, b). The volume fractions of austenite (V<sub>V</sub><sup>A</sup>) in steels F and S were estimated at 38% and 39% respectively. Two-phase microstructures display a certain degree of heterogeneity, which concerns the size and shape as well as the

arrangement of the areas of both component phases. On the longitudinal sections (LS) the areas with strongly pronounced banding predominate. Nearly continuous and strongly elongated bands of austenite with differentiated thickness are observed parallel to RD within the ferrite matrix. Due to the ferrite volume fraction  $(V_v^F > 60\%)$  the bands and elongated areas of ferrite are linked-up in a net-like form (Fig. 2). There are however regions within the  $\gamma$ -phase with the characteristic rhomboidal shapes. Their occurrence after hot-rolling indicates apparently at not fully recrystallized austenite structure. The ferrite-austenite microstructure observed on the cross-sections (CS) of the hot-rolled sheets is more heterogeneous and displays significantly lower degree of banding, that is much smaller elongation along TD. Additionally the flat areas of austenite, which are inclined at some angle to the rolling plane are also observed (Fig. 2 b). That is why the two parameters, i.e. elongation factor  $(L_E^A)$  and Feret's ratio  $(R_F^A)$ , were selected to analyse the morphology of ferrite-austenite microstructure. Due to the pronounced elongation of austenite islands along the RD, the values of both parameters are markedly higher on the longitudinal sections of both steels (Table 2). The fact that the values of both parameters are rather close and relatively high on the two examined sections, indicates at significant degree of directionality and formation of strongly oriented two-phase microstructure. TABLE 2

Volume fractions $(V_v^A)$ and the values of elongation factor $(L_E^A)$ and
the ratios of Feret's diameters $(R_F^A)$ for austenite on the longitudinal
and cross sections of the hot-rolled steels F and S

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	L <sub>E</sub>	R <sub>F</sub> <sup>A</sup>	V <sub>V</sub> <sup>A</sup>
	$(D_{max}/D_{min})$	(Fx/Fy)	[%]
F/HR-CS	3.59	3.22	38.0
F/HR-LS	4.78	4.38	50.0
S/HR-CS	3.53	3.19	39.0
S/HR-LS	4.51	4.16	59.0

Comparison of the results for both steels reveals that in two sections (LS and CS) somewhat higher values of the Feret's ratio and the elongation factor, are in the case of steel F (Table 2). Since both parameters reflect elongation of the austenite islands within the ferrite matrix (along RD and TD), it seems that such result may be explained by solution hardening of the austenitic  $\gamma$ -phase by alloying elements, especially nitrogen. The results of a number of works (see [10, 11]) indicate that after annealing at 1200-1050°C the concentration of nitrogen in austenite may be even up to 7-8 times higher than in ferrite. Taking into account the average contents of nitrogen, 0.19% in steel F and 0.26% in steel S, and assuming approximately equal proportions of both phases, the concentrations of nitrogen in the austenitic phases of both steels may reach values up to about 0.35%and 0.45% respectively. Hence in the case of steel S the austenitic phase might appear less ductile due to the higher nitrogen concentration.

# 3.2. Microstructure after annealing

The most striking result after the solution annealing at the temperatures 1200°C and 1150°C is relatively high amount of the austenitic  $\gamma$ -phase within the microstructure of both steels. In the case of the steel F the volume fractions of austenite (V<sub>V</sub><sup>A</sup>) were evaluated at 36.5% and 40% respectively (Table 3, Fig. 3) and for the steel S at about 39.5% and 42% (Table 3, Fig. 4). It appears that in both examined steels the temperature range for austenite precipitation is shifted to higher temperatures. Additionally for all the applied annealing temperatures the estimated volume fractions of the austenitic phase are higher in the case of the superduplex steel S with higher nitrogen addition. The values of the austenite volume fractions, as estimated for the successive annealing temperatures from the range 1200°C-1050°C, are listed in Table 3 and Figure 5 illustrates the results in the form of the plot  $V_V^A = f(T)$ .



Fig. 2. Microstructures of steel F(a) and steel S(b) after hot-rolling on the cross-sections (CS) and longitudinal sections (LS) of the sheets



Fig. 3. Microstructures of hot-rolled steel F after solution annealing at the temperatures 1050, 1100, 1150 and 1200°C (a-d respectively) on the cross-sections (CS) and the longitudinal sections (LS) of the sheets

TABLE 3 The mean values of austenite volume fractions  $(V_V^A)$  after solution annealing, as measured on the longitudinal and cross sections (LS+CS) for both steels F and S

		Steel F	Steel S
Temp.[°C]	Section	V <sub>V</sub> <sup>A</sup> [%]	V <sub>V</sub> <sup>A</sup> [%]
1050	LS + CS	46.8	49.0
1100	LS + CS	43.5	45.1
1150	LS + CS	39.9	41.7
1200	LS + CS	36.5	39.5

Worth to note is the fact that solidification processes of two-phase austenitic-ferritic steels start up with the formation of ferrite. It is possible to obtain 100% of ferrite within the structure of a steel by applying rapid quenching from temperatures close to solidus. Whereas the slow cooling down to the temperature range 1000-900°C favours diffusion and as the result the  $(\alpha \rightarrow \gamma)$  transformation. At high temperatures, of about 1300°C, austenite nucleates and grows at the ferrite grain boundaries and afterwards along the preferential crystallographic directions within the ferrite grains [1, 5]. In the course of the  $(\alpha \rightarrow \gamma)$  transformation the diffusion of alloying elements takes place. Degree of the  $(\alpha \rightarrow \gamma)$ transformation depends on the relative contents of ferrite and austenite forming elements in the steel, temperature and time of diffusion as well as diffusion rates of alloying elements. The austenite stabilizers (i.e. N, Ni, Cu) diffuse into the  $\gamma$ -phase while the ferrite forming elements (i.e. Cr, Mo) are supposed to concentrate mainly within the  $\alpha$ -phase [10, 11].



Fig. 4. Microstructures of hot-rolled steel S after solution annealing at the temperatures 1050, 1100, 1150 and 1200°C (a-d respectively) on the cross-sections (CS) and the longitudinal sections (LS) of the sheets



Fig. 5. The mean values of austenite volume fractions  $(V_V^A)$  as measured on the longitudinal and cross sections (LS+CS) versus the applied annealing temperature for both steels F and S



Fig. 6. Values of the elongation factor  $L_E^A$  (a) and the Feret's ratios  $R_F^A$  (b) for austenite as measured on the longitudinal (LS) and cross sections (CS) plotted against the annealing temperatures for both steels F and S

Due to the oscillations in local concentrations and the qualitative character of the analysis, the level of partitioning in two examined steels F and S was evaluated based on the concentration difference of a given element within the ferrite and austenite phases ( $\Delta C_{EI} = |C_{EI}^F - C_{EI}^A|$ ). The estimation was conducted for the three major alloying additions, i.e. Cr, Ni and Mo, at two temperatures 1200°C and 1050°C, and the results are listed in Table 4.

TABLE 4

Concentration differences of alloying elements in ferrite and austenite (in wt. %)

	Steel F			Steel S		
Temp. [°C]	$\Delta C_{Cr}$	$\Delta C_{Ni}$	$\Delta C_{Mo}$	$\Delta C_{Cr}$	$\Delta C_{Ni}$	$\Delta C_{Mo}$
1200	<2.0	~2.2	~1.25	~1,9	~2.2	~1.30
1050	~3.4	>2.5	~1.40	<3.7	~2.7	<1.55

In general, the concentration differences of the three alloying elements are not very high in comparison to their average contents in both steels. Additionally, for the temperature 1200°C the concentration difference of Cr, Ni and Mo in both component phases, that is the level of partitioning, is distinctly smaller. Except nitrogen, all the alloying additions are the substitutional elements with lower diffusion rates. That is why it seems that the addition of nitrogen, which is strong austenite stabilizer and interstitial element with high diffusion rate, is responsible for the relatively high austenite volume fractions in both examined DSS at higher temperatures.

After solution annealing significant changes are observed in two-phase microstructure, which strongly depend on the applied annealing temperature (Figs. 3 and 4). These changes concern not only the amounts (volume fractions) of both phases but also the morphology of the second phase, that is austenite. The mean values of the elongation factor ( $L_E^A$ ) and Feret's ratio ( $R_F^A$ ) as measured on the longitudinal (LS) and cross sections (CS) for the applied annealing temperatures are listed in Table 5 and presented in the form of plots in Figure 6a, b. On the longitudinal sections (LS) a considerable directionality of the ferrite-austenite microstructure is still observed. The arrangement and elongated shape of the austenite areas along the RD remained unchanged after annealing. However the bands exhibit distinctly less continuous character, especially at higher temperatures, i.e. 1200°C and 1150°C (Figs. 3c, d and 4c, d). It finds reflection in much smaller values of both parameters in comparison to the microstructure after hot-rolling (Tables 2 and 5). More comparable are the values of the elongation factor and Feret's ratio on the longitudinal sections (LS) after hot-rolling and subsequent solution annealing at

TABLE 5

Steel/Temp./Sec.	$L_E^A$ (D <sub>max</sub> /D <sub>min</sub> )	R <sub>F</sub> <sup>A</sup> (Fx/Fy)	Steel/Temp./Sec.	L <sub>E</sub> (D <sub>max</sub> /D <sub>min</sub> )	R <sub>F</sub> <sup>A</sup> (Fx/Fy)
F / 1050 - LS	3.68	3.45	S / 1050 - LS	4.54	4.27
F / 1100 - LS	3.67	3.45	S / 1100 - LS	4.47	4.10
F / 1150 - LS	2.87	2.61	S / 1150 - LS	3.69	3.45
F / 1200 - LS	2.69	2.48	S / 1200 - LS	2.99	2.78
F / 1050 - CS	2.97	2.69	S / 1050 - CS	3.58	3.22
F / 1100 - CS	2.53	2.19	S / 1100 - CS	3.07	2.76
F / 1150 - CS	2.16	1.77	S / 1150 - CS	3.02	2.63
F / 1200 - CS	1.91	1.55	S / 1200 - CS	2.43	2.14

Values of the elongation factor  $(L_E^A)$  and the Feret's ratios  $(R_F^A)$  for austenite after solution annealing on the longitudinal (LS) and cross-sections (CS) for steels F and S

1050°C. This is mainly due to the change of austenite volume fraction, that increased from 38% and 39% after rolling to about 47% and 49% after annealing in both steels F and S respectively. Higher values of both parameters at 1050°C correspond with more elongated and continuous austenite bands (Figs. 3a and 4a). Therefore it appears that the morphology of the ferrite-austenite microstructure after thermo-mechanical treatment is related with the austenite volume fraction and strongly depends on the temperature of solution annealing applied after hot-rolling.

It seems however, that much more substantial changes within the ferrite-austenite morphology after solution annealing concern the microstructures observed on the cross-sections (CS) of the examined steels (Figs. 3 and 4). This conclusion results especially from the comparison of the values of elongation factor  $(L_F^A)$  and Feret's ratio  $(R_{\rm F}^{\rm A})$  after hot-rolling with those after annealing in the case of steel F (Tables. 2 and 5). In hot-rolled sheets the mean values of both parameters were relatively high, that is  $L_E^A = 3.59$  and  $R_F^A = 3.22$ , whereas after annealing markedly decreased with increasing temperature to  $L_E^{A} = 2.97-1.91$  and  $R_F^{A} = 2.69-1.55$ . In particular this effect is well visible on the microstructures of steel F for the higher annealing temperatures (Fig. 3c, d). On the contrary, the microstructures of steel S display markedly higher degree of structural anisotropy and in consequence higher values of elongation factor and Feret's ratio in comparison to steel F. It should be noted additionally, that in few cases the values of both parameters ( $L_E^A$  and  $R_F^A$ ) after annealing exceeded those after hot-rolling. This situation concerns the microstructures of steel S after annealing at 1050°C and may be explained once again by substantially higher volume fractions of austenite after solution annealing at lower temperature.

# 4. Concluding remarks

Two examined steels exhibit significant degree of banding after hot-rolling but also considerable heterogeneity of the ferrite-austenite microstructure, which concerns the size and shape as well as the arrangement of the areas of both component phases. The estimated volume fractions of austenite are 38% and 39% for steels F and S respectively.

On the longitudinal sections (LS) of the hot-rolled sheets, the bands and strongly elongated islands of austenite (along the RD) are mainly observed on the background of the ferrite matrix. The microstructure on the cross-sections (CS) is more heterogeneous and displays lower degree of banding, that is much smaller elongation of austenite areas along TD. The values of elongation factor and the ratios of Feret's diameters estimated for austenite on both sections (LS and CS) are higher in the case of steel F. It seems that the austenitic phase of steel S is less ductile due to the higher nitrogen concentration.

After solution annealing significant changes are observed in the ferrite-austenite microstructure, which strongly depend on the applied annealing temperature. These changes concern not only the amounts of both phases but also the morphology of the second phase, that is austenite. The volume fractions of austenite, as estimated at temperatures 1200°C-1050°C, changed within the range 36,5-47% for steels F and 39,5-49% in the case of steel S.

After annealing at higher temperatures (i.e.  $1200^{\circ}$ C and  $1150^{\circ}$ C) relatively high amounts of the austenitic  $\gamma$ -phase were found within the microstructures of both steels. Additionally, for all the applied annealing temperatures, the estimated volume fractions of austenite are higher in steel S in comparison to steel F. It seems that the addition of nitrogen, which is strong austenite

stabilizer and interstitial element with high diffusion rate, is responsible for the higher austenite volume fractions in steel S, and shifts the temperature range of austenite precipitation to higher temperatures in the case of both steels.

It appears that the morphology of the ferrite--austenite microstructure after thermo-mechanical treatment is strongly related with the austenite volume fraction. The arrangement of the austenite areas, as observed on the longitudinal sections (LS), remained unchanged after annealing, however at higher temperatures these areas are less continuous. With decreasing temperature and increasing austenite volume fraction the bands of austenite become more continuous. This especially applies to steel S, which displays markedly higher degree of structural anisotropy and in consequence higher values of elongation factor and Feret's ratio in comparison to steel F on both examined sections. On the contrary the austenite morphology on the cross-sections (CS) of steel F is markedly more isotropic, especially at higher temperatures (i.e. 1200°C and 1150°C).

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