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# The influence of hot-working processing on plasticity and structure of duplex steel

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#### **ABSTRACT**

**Purpose:** There are numerous branches of industry where the  $(\alpha+\gamma)$  duplex steels have not yet been sufficiently popularised due to problems with their forming, resulting from different properties of the both phases which make up the material. This paper analyses the influence of temperature and tension rate on the superplastic flow of the  $(\alpha+\gamma)$  duplex steel.

**Design/methodology/approach:** Steel specimens were cold deformed with a 70% rolling reduction. After a solution head treatment (1350°C), the specimens were tensioned in temperatures ranging from 800 to 950°C at a rate of  $v_r$ =15×10<sup>-3</sup>÷3×10<sup>-1</sup>mm/s in a 0.005Pa vacuum. Structural examination was carried out using light, scanning and transmission electron microscopy. A quantitative analysis of structural changes was performed using the "MetIlo" image analysis programme.

**Findings:** This paper has shown the cooperation of structure reconstruction mechanisms during deformation of the investigated steel and attempet the changes that take place in the steel structure during superplastic flow.

**Practical implications:** The research carried out enabled the understanding of the phenomena taking place during deformation and annealing of the investigated alloy. The results will constitute the basis for modelling the structural changes.

**Originality/value:** The results will be used to design the basis for a thermo-mechanical processing technology via rolling and inter-operational annealing of the investigated steel.

Keywords: Superplastic materials; Plastic forming; Microstructure; Hot tensile test

#### **MATERIALS**

## 1. Introduction

High-alloy steels of the duplex type  $(\alpha+\gamma)$  of a ferritic-austenitic structure belong to the group of corrosion-resistant steels. They show higher corrosion resistance in comparison with steels of one-phase ferritic and austenitic structure, which, when coupled with their high strength and lower production costs resulting from a lower nickel content, makes them a material more and more widely used in a variety of industry branches [1-8]. They are used for the production of desulfurisation, desalting and purifying machinery, as well as pipelines, tanks, pump components, or even bridge

structural components [9-12]. However, there are numerous branches of industry where the  $(\alpha+\gamma)$  duplex steels have not yet been sufficiently popularised due to problems with their forming, resulting from different properties of the both phases which make up the material. The literature [6, 7] provides us with some information on plasticity tests conducted on ferritic-austenitic steels so as to induce the superplasticity effect as a result of complex thermo-plastic processes. Most frequently, superplasticity is determined by: heat treatment, cold plastic strain (normally below 50% of draft) and hot tension at a very low rate [6-8]. The deformation process of  $(\alpha+\gamma)$  duplex steels after cold plastic working is frequently carried out at a temperature of 700-950°C,

where precipitation of the  $\sigma$  phase takes place. The presence of the  $\sigma$  phase affects brittleness of the material at a temperature below 400°C, although it does not affect ductility at a temperature of 400÷950°C. In properly selected deformation conditions, the reduced in size precipitations of the  $\sigma$  phase make the steel attain superplastic properties [9, 10]. In spite of long-lasting research on plasticisation of these steels, the explicit determination of the structure reconstruction mechanisms during their deformation processes has proved to be unsuccessful [9-22].

This paper analyses the influence of temperature and tension rate on the superplastic flow of the  $(\alpha+\gamma)$  duplex steel. The influence of tension parameters on the qualitative and quantitative image of structural changes has been determined. The paper is also an attempt at explaining the changes that take place in the steel structure during superplastic flow.

### 2. Material and methodology

The material for the research was a steel sheet of the initial thickness equal  $h_0$ =3mm, made from the X2CrNiMoN22-5-3 steel (grade 1.4462 acc. to EN 10270) after rolling and annealing at 1000°C is shown in Fig.1. After solutioned at T=1350°C steel has ferritic structure with an insignificant amount, i.e. 2.8% of austenite (Fig. 2) and a chemical composition presented in Table 1.

Table 1. Chemical composition of the X2CrNiMoN22-5-3 steel ( %-wt.)

C	Si	Mn	Ni	Cr	Mo	N	S	P
0.017	0.30	1.57	5.45	22.8	3.12	0.169	0.0009	0.019

The steel was subjected to cold deformation by rolling with the total rolling reduction  $\varepsilon_h$  =0.7, 0.9. Next, specimens were made for hot deformation by tension, with the initial measuring basis length of 3mm. Tensile tests were performed by means of a hydraulic strength testing machine, Instron 8801. In order to induce the superplastic flow effect, tension was conducted in the temperature range of T=800÷950°C, at a rate of  $v_r = 15 \times 10^{-3} \div 3 \times 10^{-1} \text{mm/s}$ , in a 0.005Pa vacuum. The deformation process was conducted both until the predetermined elongation and until failure, with the specimens being cooled with compressed air. The elongation of a specimen with the initial measuring basis of 3 mm is designated as  $A_{3mm}$ . After tension, material for structural examination was taken from the specimens. Evaluation of the steel specimens' structures after failure was made at a 2mm distance from the failure point, whilst the structure of specimens deformed until the predefined elongation was evaluated in the place of the greatest localisation of the deformation. Observation of the structures was conducted on an "Olympus" light microscope and on a "Hitachi" S-4200 scanning microscope. The microstructure, including the  $\sigma$  phase, was detected by electrolytic etching in a 20% NaOH solution. A quantitative evaluation of the structure was conducted using the METILO programme [23, 24]. The following quantities were determined:

- surface fraction of individual phases A<sub>A</sub>[%],
- mean grain plane section area (precipitations) Ā [μm²],
- grain plane section variability ratio v(Ā) [%].

Investigation of the substructure was carried out by the thin foil method on a JEM – 100B transmission electron microscope of JEOL company. For an evaluation of misorientation angles between neighbouring subgrains, diffraction examinations were performed on an electron transmission microscope. The microdiffraction technique was used, owing to which diffraction images with Kikuchi lines were obtained. The misorientation angles were determined using the comuter programme.

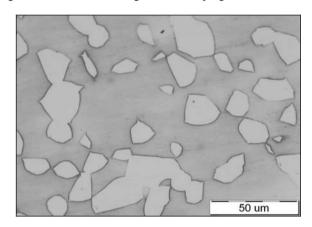


Fig. 1. Structure of the X2CrNiMoN22-5-3 steel after rolling and annealing at 1000°C

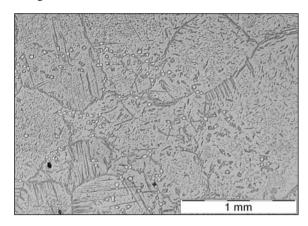


Fig. 2. Structure of the X2CrNiMoN22-5-3 steel after solution heat treatment from T=1350 $^{\circ}$ C, with soaking time of 60 minutes and cooling in water. Surface fraction of ferrite  $A_A$ =97.8%±0.5%

## 3. Results

Tensile curves presenting the influence of tension temperature at a rate  $v_r$ =15×10<sup>-2</sup>mm/s of an earlier cold deformed steel, whose total relative rolling reduction was  $\epsilon_h$ =0.7, on the course of changes of force (F) as a function of elongation  $A_{3mm}$  are shown in Figure 3, and the dependence of temperature on elongation  $A_{3mm}$ , in Figure 3.

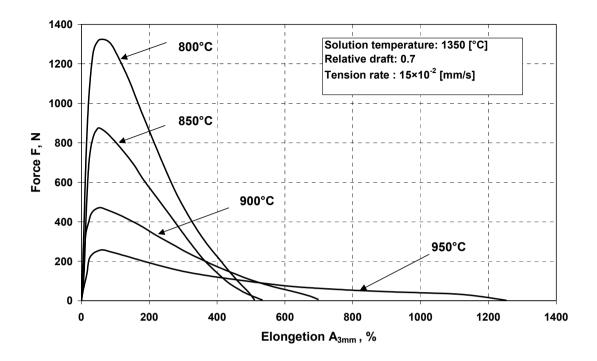


Fig. 3. Influence of tension temperature at a rate  $v_r = 15 \times 10^{-2}$  mm/s on the form of force/elongation curves

After exceeding the value of  $F_{max}$ , tension takes place at a decreasing value of force until failure (Fig. 2). The greatest elongation until failure,  $A_{3mm}\!=\!1250\%$ , is achieved by the steel subjected to tension at a temperature  $T\!=\!950^{\circ}C$  (Fig.3). An increase of tension temperature in the range of  $T\!=\!800\!\div\!950^{\circ}C$  leads to obtaining greater and greater elongation until failure  $A_{3mm}$  (Figs. 3, 4).

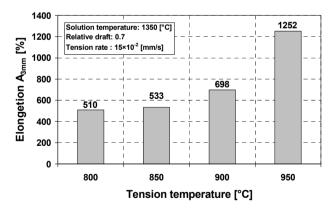


Fig. 4. Influence of tension temperature at a rate  $v_r$ =15×10<sup>-2</sup>mm/s on the value of percentage elongation until failure  $A_{3mm}$ 

The lowest value,  $A_{3mm}$ =510%, was obtained during deformation at T=800°C, whilst the greatest elongation,  $A_{3mm}$ =1252%, at a temperature T=950°C.

The results of the influence of tension rate at T=850°C of the cold deformed steel of a total relative rolling reduction  $\epsilon_h$ =0.9 on

the course of changes of force (F) as a function of elongation  $A_{3mm}$  is presented in Fig. 5, and the dependence of temperature on elongation  $A_{3mm}$ , in Fig. 5.

The tensile curves obtained for the steel investigated show a similar shape (Fig. 5). However, the rate at which the material "softens" during tension after exceeding the maximum force  $F_{max}$ , varies. An increase in the steel tension rate in the range from  $v_r=15\times10^{-3}$ mm/s to  $v_r=3\times10^{-1}$ mm/s at a temperature  $T=850^{\circ}$ C results in a decrease of elongation until failure  $A_{3mm}$ , with the exception of rate  $v_r=3\times10^{-3}$ mm/s (Fig. 4, 5). When tensioning the steel at a rate  $v_r=3\times10^{-3}$ mm/s, the greatest elongation of  $A_{3mm}=1238\%$  was obtained. The lowest value,  $A_{3mm}=371\%$ , was obtained when tensioning at  $v_r=3\times10^{-1}$ mm/s.

Ferrite in the ferritic-austenitic steel after its heating to the investigated range of tension temperature, i.e.  $T=800 \div 950^{\circ}C$ , undergoes transition to austenite and precipitations of phase  $\sigma$  [8], according to reaction  $\alpha \rightarrow \gamma + \sigma$ .

The results of the influence of elongation  $A_{3mm}$  on the changes of structure during tensioning are shown in Figure 7. In the structure of the steel after solution heat treatment from T=1350°C and deformation  $\epsilon_h$ =0.7, followed by tensioning at T=850°C, an increase of the size of sigma phase is observed as the deformation increases.

The results of the quantitative analysis of structural changes depending on elongation are juxtaposed in Table 2. As elongation  $A_{3mm}$  increases, the surface fraction of  $\sigma$  phase grows and changes from 12.9%±0.5% at elongation  $A_{3mm}$ =24%, to a value equal 16.9%±0.5% at  $A_{3mm}$ =932%. The increase of elongation  $A_{3mm}$  is accompanied by growth of the mean plane section area of precipitation, reaching the highest value,  $\bar{A}$ =1.19 $\mu m^2$ , at elongation after failure.

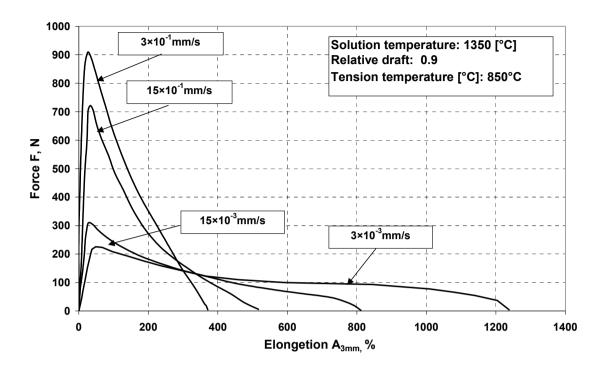


Fig. 5. Influence of tension rate at a temperature 850°C on the form of force/elongation curves

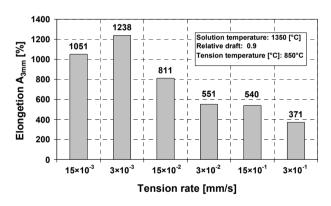


Fig. 6. Influence of tension rate at a temperature of T=850°C on the value of elongation until failure

However, no explicit influence of elongation  $A_{3mm}$  on inhomogeneity of the  $\sigma$  phase precipitation size has been found.

An increase in elongation to  $A_{3mm}$ =200% results in a dislocation density reduction in austenite subgrain in the neighbourhood of sigma phase precipitations. In regions free of precipitations, transformation of the subgrain structure is observed. In region free of precipitations, transformation of the subgrain subsubstructure is observed (fig. 8). The mean misorientation angle between austenite subgrains increases to ca. 20°.

Both narrow- and wide-angle bundaries are present in the ranges from 2 to 34°, where as much as 35% of their fraction is present at ca. 22°. Different stadia od dynamic recovery were observed and in consequence, different processes of dynamic recrystallization (Fig. 9).

The influence of the tension process temperature seems to be significant from the point of view of the structure. The influence of temperature on the surface fraction of  $\sigma$  phase precipitations, the phase size and inhomogeneity, are presented in Figures 10-12.

The maximum surface fraction of  $\sigma$  (A<sub>A</sub>=19,2±0,5%) phase precipitations was obtained after steel tensioning at T=850°C (Fig. 10).

After tensioning at T=800°C and T=900°C, the surface fraction of the  $\sigma$  phase precipitations is similar and amounts ca.  $A_{A}$ =13%.

The lowest surface fraction ( $A_A$ =8,1±0,4%) was obtained after tensioning at T=950°C. An increase of the tension temperature in the range of T=800÷950°C leads to a more than sixfold increase of the  $\sigma$  phase precipitation size from the value  $\bar{A}$ =0,32 $\mu$ m<sup>2</sup> at T=800°C to  $\bar{A}$  =2.12 $\mu$ m<sup>2</sup> as obtained after tensioning at T=950°C (Fig. 10).

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The structure of steel also depends on the tension rate. The influence of rate on structural changes after failure is presented in Figs 13÷15. An increase of tension rate leads to a reduction of the  $\sigma$  phase fraction from A<sub>A</sub>=17.7±0.4% to A<sub>A</sub>=10.8±0.5% after tensile tests at T=850°C (Fig. 13).

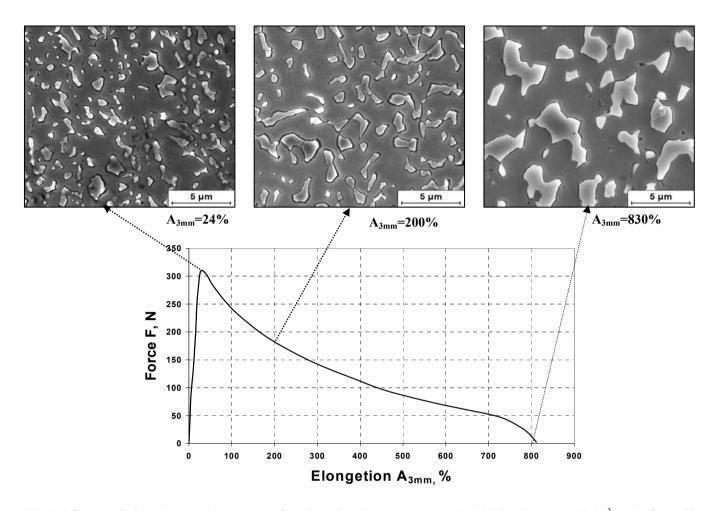


Fig. 7. Influence of elongation on the structure of steel tensioned at a temperature T=850°C and rate  $v_r$ =15×10<sup>-3</sup>mm/s after cold deformation  $\epsilon_h$ =0.7

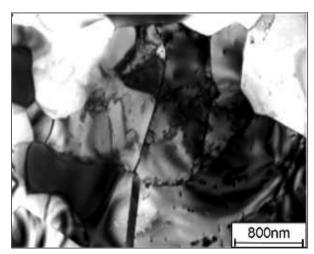


Fig. 8. Substructure of duplex steel after deformation at a temperature T=850°C and rate  $v_r\!\!=\!\!15\times\!10^{\text{-3}}\text{mm/s}.$  Deformation of the sample 200%

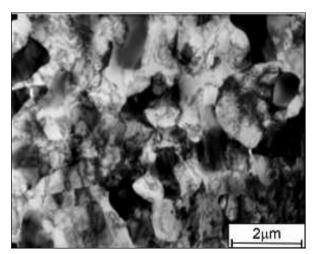


Fig. 9. Substructure of duplex steel after deformation at a temperature T=850°C and rate  $v_r\!=\!15\times10^{\text{-3}}\text{mm/s}.$  Deformation of the sample 200%

Table 2. Results of the influence of elongation  $A_{3mm}$  on the quantitative description of structural changes

	$A_{3mm}=24\%$	$A_{3mm}=200\%$	$A_{3mm} = 830\%$
Surface fraction of phase $\sigma$ , [%]	12.9	13.1	16.9
Mean area of the $\sigma$ phase precipitation, $\bar{A}$ [ $\mu m^2$ ];	0.34	0.39	1.19
Inhomogeneity of the $\sigma$ phase precipitation, $v(\bar{A})$ [%],	85	57	148

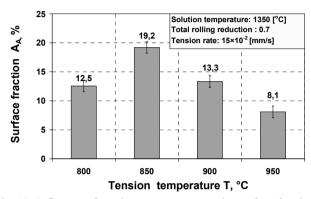


Fig. 10. Influence of tension temperature on the surface fraction of  $\sigma$  phase precipitations

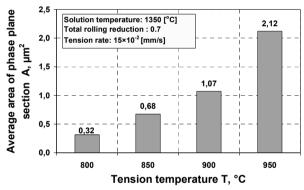


Fig. 11. Influence of tension temperature on the size of  $\boldsymbol{\sigma}$  phase precipitation

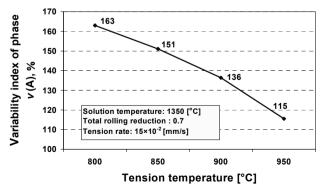


Fig. 12. Influence of tension temperature on inhomogeneity of the  $\sigma$  phase precipitation size

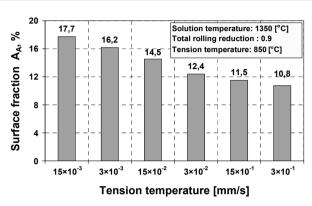


Fig. 13. Influence of tension temperature on the  $\sigma$  phase surface fraction

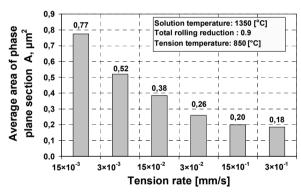


Fig. 14. Influence of tension rate on the size of  $\boldsymbol{\sigma}$  phase pre cipitations

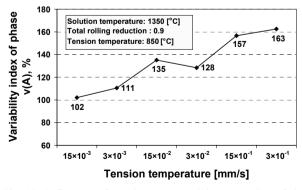


Fig. 15. Influence of tension rate on inhomogeneity of the  $\sigma$  phase precipitations' size

This results, inter alia, from the shorter and shorter time of test duration at higher tension rates. As the tension rate increases, the mean area of the  $\sigma$  phase precipitation plane section reduces from  $\bar{A}=0.77\mu\text{m}^2$  to  $\bar{A}=0.18\mu\text{m}^2$  obtained when tensioning at a rate  $v_r=3\times10^{-1}\text{mm/s}$  (Fig. 14). Inhomogeneity of the  $\sigma$  phase precipitations shows a growing trend as the tension rate increases (Fig. 15).

#### 4. Conclusions

The research carried out allow evaluating the influence of tension parameters on the obtained elongation until failure and changes in the structure of the X2CrNiMoN22-5-3 steel solutioned from T=1350°C and subjected to cold deformation,  $\epsilon_h$ =0.7. It was shown in previous papers that solution heat treatment of the investigated steel from the temperature of 1350°C results in obtaining a ferritic structure. This, in turn, results in obtaining higher values of deformation until failure when compared to specimens deformed from a solutioned material from lower temperatures in the ferritic-austenitic scope [2]. The obtained elongation until failure amounting to  $A_{3mm}$ =370÷1252% testifies to the occurrence of the superplastic flow effect in the investigated range of tension parameters. The effect in the ferritic-austenitic steel in connected with the transition  $\alpha$ - $\gamma$ + $\sigma$  which takes place during the deformation process.

Increasing elongation during tensioning results in an increasing fraction and size of the  $\sigma$  phase in the material structure. The  $\sigma$  phase fraction in the steel structure after tensile tests at a tension rate of  $v_r=1.5\times10^{-2}$  mm/s depends on the temperature at which the process in conducted and it is in compliance with the CTW diagram [14, 24].

The precipitation process is the most intensive at temperature  $T=850^{\circ}C$ . As the tension temperature increases in the range of  $T=800\div950^{\circ}C$ , the percent elongation until failure,  $A_{3mm}$ , grows (Fig. 3, 4). The temperature increase is conducive to more intensive dynamic processes of structure reconstruction. The greatest elongation,  $A_{3mm}=1252\%$ , was obtained at  $T=950^{\circ}C$ , where the structure is still composed of austenite and  $\sigma$  phase precipitations. A continued increase of the process temperature in accordance with the equilibrium diagram [25] would cause precipitation of the ferritic  $(\alpha+\gamma)$  phase, without a fraction of  $\sigma$  phase precipitation grains which hinder the growth. This, in turn, would lead to a grain growth and have an adverse effect on deformability of the X2CrNiMoN22-5-3 steel.

As the tension rate increases in the range of  $v_r = 15 \times 10^3 \, s^1 \div 3 \times 10^{-1} \, mm/s$ , decreasing elongation,  $A_{3mm}$ , is observed. A decreasing tension rate results in an increase of the fraction and size of the  $\sigma$  phase precipitations. This is connected with diffusion of the elements, nucleation and growth of the  $\sigma$  phase precipitations.

The austenitic matrix is under continuous reconstruction. Also, continuous recrystallisation takes place, which enables the formation of new, free of defects, equiaxial grain. An increase of the misorientation angle with increasing elongation also testifies to the structure reconstruction.

This paper has shown the cooperation of structure reconstruction mechanisms during deformation of the investigated

steel, i.e. continuous dynamic recrystallisation, which consists in an increase of the grain misorientation angle as the deformation grows, being accompanied by grain growth, as well as dynamic recrystallisation, which consists in nucleation and growth of new grain along with a precipitation process of the  $\sigma$  phase which inhibits the growth of the newly formed grain.

The results obtained are vital from the point of view of design of the thermo-mechanical processing technology for the investigated steel.

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