

HOT TENSILE TESTING OF SAF 2205 DUPLEX STAINLESS STEEL

VROČI NATEZNI PRESKUSI DUPELEKS NERJAVNEGA JEKLA SAF 2205

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The changes to the microstructure of the duplex stainless steel SAF 2205 during hot tensile tests were investigated. The dominant restoration mechanisms for ferrite and austenite were dynamic recovery (DRV) and dynamic recrystallization (DRX), respectively. Also, the effect of temperature on the deleterious phase precipitation was investigated. The specimens were tested with hot tensile deformation tests in the temperature range from 800 °C to 1100 °C. The tensile strength of the investigated steel decreased rapidly in the temperature range from 800 °C to 950 °C and slowly at the testing temperatures from 1000 °C to 1100 °C. It was found that SAF 2205 has excellent hot-working properties at temperatures between 950 °C and 1100 °C. The differences in the mechanical properties of austenite and ferrite along with the precipitation of the σ -phase represent the most important restrictions during hot working. Optical microscopy was used for the microstructure-evolution analysis in the duplex stainless steel during the hot tensile tests.

Keywords: duplex stainless steel, hot tensile test, microstructural evolution, intermetallic phases, σ -phase

Preiskovali smo spremembe mikrostrukture dupleks nerjavnega jekla SAF 2205 med vročimi nateznimi preskusi. Prevladujoča mehanizma mehčanja v feritu in avstenitu sta bila dinamična poprava in/ali dinamična rekristalizacija. V dupleks nerjavnem jeklu je bil raziskan vpliv temperature na izločanje škodljivih faz. Vzorce smo vroče natezno preskušali v temperaturnem območju med 800 °C in 1100 °C. Natezna trdnost preiskanega jekla se je hitro znižala v temperaturnem območju med 800 °C in 950 °C ter počasneje pri temperaturah preskusov med 1000 °C in 1100 °C. Ugotovili smo, da ima SAF 2205 odlične vroče preoblikovalne lastnosti v temperaturnem območju med 950 °C in 1100 °C. Največje omejitve pri preoblikovanju predstavljajo razlike v mehanskih lastnostih med avstenitom in feritom ter izločanje σ -faze. V raziskavi je bila uporabljena optična mikroskopija za analizo razvoja mikrostrukture v dupleks nerjavnem jeklu med vročimi nateznimi preskusi.

Ključne besede: dupleks nerjavno jeklo, vroči natezni preskusi, razvoj mikrostrukture, intermetalne faze, σ -faza

1 INTRODUCTION

Duplex stainless steels (DSS) have the advantage of low price, pitting-corrosion resistance, stress-corrosion-cracking resistance, resistance to intergranular corrosion, high mechanical strength, corrosion-fatigue resistance, wear resistance, super plastic behaviour and good weldability.¹⁻⁸ But they have a severe disadvantage in being difficult to hot work.^{2,9,10} The DSS microstructure generally consists of around 50 % δ -ferrite and 50 % γ -austenite, which gives them excellent properties, but also provides for a narrow window in the hot-working process. During hot working, ferrite and ferritic stainless steels undergo dynamic recovery (DRV),¹¹ which means that subgrains develop in the microstructure, as is the case for the hot working of duplex stainless steels,¹² while austenite and austenitic stainless steels undergo a high degree of strain hardening and then dynamic recrystallization (DRX).⁸

While the austenite is dominated by high-angle grain boundaries (HAB), ferrite shows a substantial amount of subgrain boundaries, i.e., low-angle grain boundaries (LAB). Such a contrasting behaviour of austenite and ferrite phases is due to the different stacking-fault ener-

gies of these phases, which imposes different deformation mechanisms.¹³

Therefore, when hot working duplex stainless steels, DRV is the initial softening mechanism. But austenite undergoes DRV only at high temperatures and low strain rates. At low strain rates and high temperatures DSS exhibit superplasticity; therefore, allowing elongations that exceed 1000 %.⁶ Superplasticity can be linked to grain-boundary sliding, but grain-boundary sliding also causes cavity formation, which in turn causes stress concentration, and if these stresses cannot be released at sufficient rates, the cavities nucleate.¹⁴ The cavities, provided with appropriate conditions, then undergo the processes of growth and coalescence and form larger cavities that can lead to failure.¹⁴ Another factor that negatively influences the mechanical properties is the precipitation of secondary phases,¹⁵ especially the σ -phase, which occurs in the temperature range from 700 °C to 1000 °C and is promoted by Cr, Mo and Si.^{12,16,17} The σ -phase reduces the ductility and toughness of the steel.¹² Increased steel hardness is an indicator of σ -phase precipitation.¹² The precipitation phenomena in duplex stainless steels occur mainly in the δ -ferrite,

Table 1: Chemical composition SAF 2205 in mass fractions, (w/%)**Tabela 1:** Kemijska sestava jekla SAF 2205 v masnih odstotkih, (w/%)

C	Si	Mn	P	S	Cr	Ni	Cu	Mo	V	Ti	Nb	Al	N
0.021	0.32	1.58	0.026	0.002	22.95	5.3	0.26	2.742	0.15	0.005	0.008	0.012	0.141

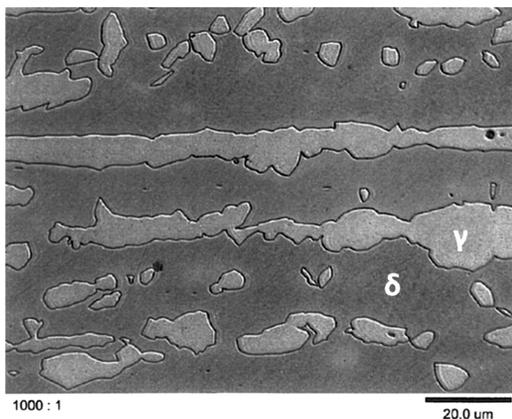
because the diffusion rates are much faster than in the austenite. The genesis of the precipitates can be attributed to the large amount of alloying element and is promoted by the instability of the ferritic matrix at the high temperatures. The morphology and location of the secondary phases are well known and described,¹⁸ they precipitate both at triple points and grain boundaries, and their growth occurs towards the unstable ferrite, also due to the diffusion behaviour of the involved elements.

2 MATERIALS AND EXPERIMENTAL PART

Duplex stainless-steel grade SAF 2205, with the chemical composition given in **Table 1**, was used in the experimental work.

The initial microstructure is presented in **Figure 1**. Both phases have an almost fully recrystallized microstructure produced by the annealing treatment at 1050 °C before deformation testing.

The tensile test specimens of SAF 2205 were made according to DIN 50125. The specimens were cut out from the hot-rolled plates longitudinal to the rolling direction. Tensile tests were performed at elevated temperatures of (800, 850, 900, 950, 1000, 1050 and 1100) °C on a 500-kN INSTRON tensile test machine with a special furnace mounted to ensure high-temperature conditions. The specimens were heated at a rate of 700 °C/h, and were held at the deformation temperature for 15 min. The deformation speed was 5 mm/min, which gives a logarithmic strain rate of 0.0014 s⁻¹. During the hot tensile tests the force and extension were recorded simultaneously. At the end of the tensile tests, the specimens were air cooled.

**Figure 1:** Initial microstructure of SAF 2205 duplex stainless steel, austenite (light) and ferrite (dark), light microscopy**Slika 1:** Začetna mikrostruktura dupleks nerjavnega jekla SAF 2205, avstenit (svetel) in ferit (temen), svetlobna mikroskopija

The samples for metallographic analysis of the tensile specimens were taken 10 mm from the fracture surface in the contraction area, longitudinal to the testing direction. The samples were etched with a solution of 30 g KOH, 30 g K₃Fe(CN)₆ and 100 mL distilled water. The etching causes the phases in the microstructure to colour differently: the σ -phase is dark, the δ -ferrite is lighter and the γ -austenite is the brightest in colour. The metallographic samples were observed with an optical microscope (Microphot FXA, Nikon). The content of the magnetic δ -ferrite was determined by a ferritoscope instrument FISCHER MP30.

3 RESULTS AND DISCUSSION

The initial microstructure of the steel is composed from austenitic grains (light) that are oriented in the rolling direction and recrystallized ferrite grains (dark). Individual "islands" of austenite grains are located in the ferrite areas. Specimens that were tested at (800, 850 and 900) °C contain σ -phase, while those that were tested at higher temperatures contain no σ -phase.

The specimen's microstructures after the tensile tests are shown in **Figures 2a** to **2g**. Cavitations or cracks that formed at high deformations are visible in **Figures 2d** to **2f** at temperatures from 950 °C to 1050 °C. The **Figures 2d** to **2f** show some cracks that nucleated at the austenite/ferrite interfaces and propagated along the interface towards the softer ferrite phase. The same phenomenon was also found by M. Faccoli and R. Roberti.¹⁹ Due to a notable difference in strength between the ferrite and austenite, voids are formed at the austenite/ferrite interfaces. Deformation at high temperatures, however, allows dynamic restoration (DRV and DRX) to take affect and reduce the work hardening, thus reducing the probability of void formation.

It is clear that the hot tensile strength of the steel at deformations up to $\varphi = 0.5$ gradually increases at temperatures around 800 °C, and this can be partially said for the test at 850 °C. But at the temperature of 900 °C the tensile strength does not change significantly due to the established equilibria between the strain hardening and the softening effects. **Figure 2a** shows the microstructure of the sample tested at the lowest temperature. The austenite phase appears in the form of elongated islands. In the sample tested at the lowest temperature (**Figure 2a, 2b**) their distribution is not homogeneous and the austenite/ferrite interfaces are affected by σ -phase precipitation.

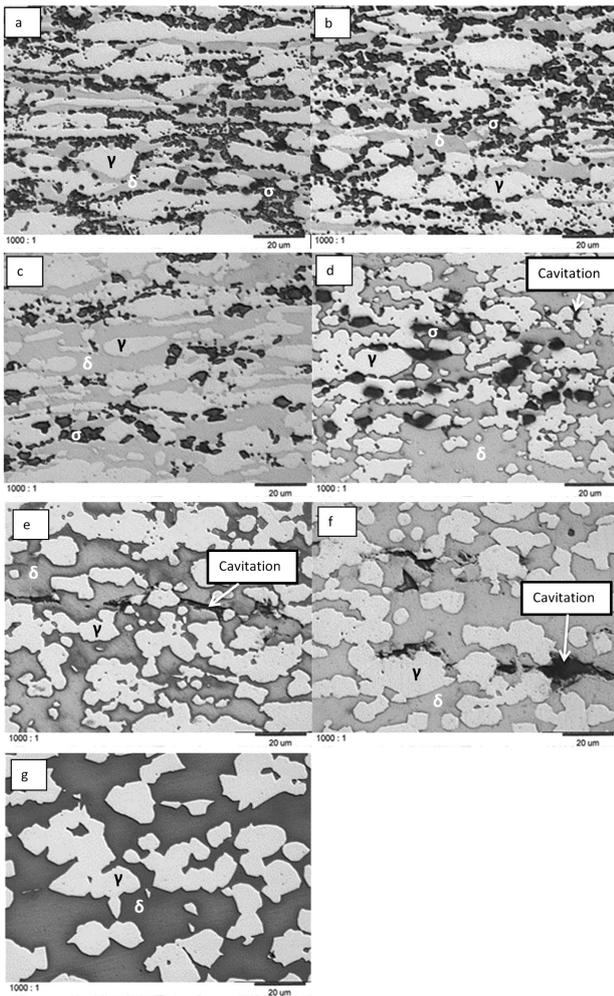


Figure 2: Microstructures of specimens after tensile tests at different temperatures, light microscopy

Slika 2: Mikrostruktura preskušancev po vročih nateznih testih pri različnih temperaturah, svetlobna mikroskopija

At higher temperatures from 950 °C to 1100 °C there is a significant drop in tensile strength due to the softening effects, and the initial strengths are not reached.

Figures 2d to 2g show more equiaxed austenitic grains, a sign of the recrystallization process.

As the temperature increases, the austenite volume fraction decreases and the austenite phase islands are progressively reduced in length and appear more discontinuous; γ -islands which are almost totally dissolved assume a globular shape. According to A. Iza-Mendia et al.,²⁰ austenite produces non-deformed regions at this temperature. The strain difference between the two phases is, therefore, responsible for the severe shear strains at the phase boundaries, sliding or even cracks.

The stress-strain curves for the tests at different temperatures (from 800 °C to 1100 °C) at the strain rate of 0.0014 s⁻¹ are shown in **Figure 3**. According to **Figure 3**, the optimum hot-working temperature range at the strain rate of 0.0014 s⁻¹ should be between 950 °C and 1100 °C.

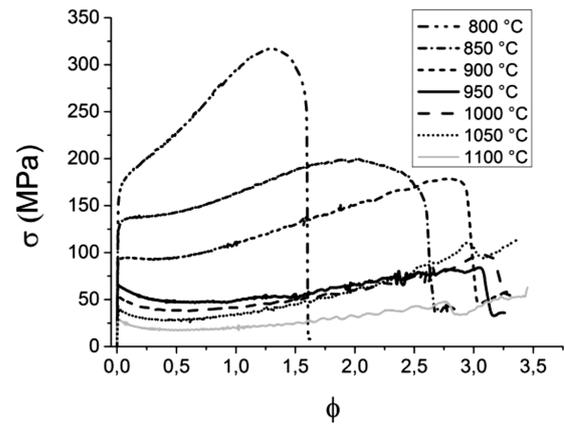


Figure 3: Stress-strain (logarithmic) diagram for hot tensile tests at different temperatures

Slika 3: Graf napetost – (logaritemska) deformacija vročih nateznih preskusov pri različnih temperaturah

The worst hot-working properties are at 800 °C. Generally, the flow stress rises to a maximum at the commencement of the straining before dropping to a steady-state level. The shape of the curves also changes as the material and deformation conditions are altered. At high temperatures, above 950 °C, the flow curves have the characteristic shape expected for materials that show dynamic recovery. As the deformation temperature is decreased the shape changes with rapid work hardening, followed by extensive flow softening. These changes become more dramatic when the austenite volume fraction is further increased. As the volume fraction of austenite in the ferrite matrix is increased, the ductility also decreases

The results of the tensile strength and the reduction of area that were obtained from hot tensile tests are summed up in **Figure 4**. The best hot-working properties are achieved at higher temperatures, as the material has the lowest tensile strengths and the highest contractions and elongations (**Figure 4**).

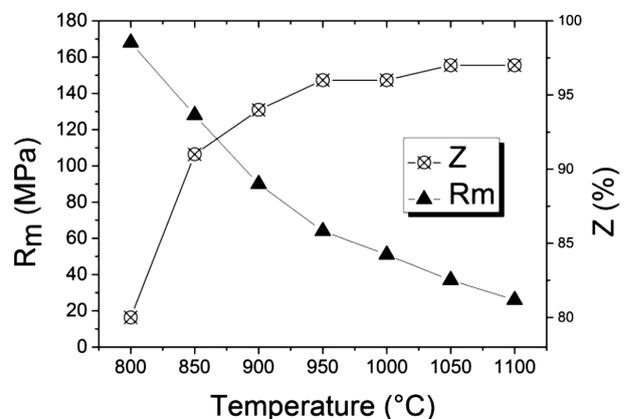


Figure 4: Tensile strength and contraction of SAF 2205, depending on the testing temperature in the range from 800 °C to 1100 °C

Slika 4: Natezna trdnost in kontrakcija SAF 2205 v odvisnosti od temperature preizkušanja v temperaturnem območju od 800 °C do 1100 °C

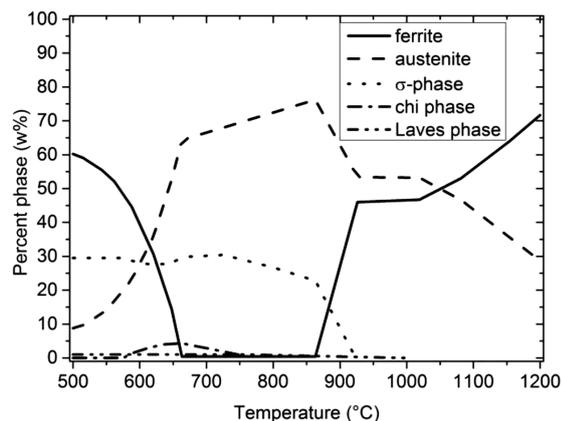


Figure 5: Calculated weight fractions of equilibrium phases in SAF 2205 in the temperature range from 500 °C to 1250 °C

Slika 5: Izračunani masni deleži ravnotežnih faz v SAF 2205 v temperaturnem območju od 500 °C do 1250 °C

There is a rapid increase in the reduction area as the temperature rises from 800 °C to 850 °C, from 850 °C on it gradually increases with temperature, while the fall in tensile strength is more continuous.

The high-temperature mechanical properties and hot-working properties depend mostly on the microstructural changes. DRV can be observed as a polygonisation of the subgrains; it is more pronounced at high temperatures and lower deformation speeds and lower loads. DRX can be observed as the formation of new, equiaxed grains. Another process that can influence the hot-working properties is the precipitation of the intermetallic phases from the matrix, namely the σ -phase.

The equilibrium phase composition for SAF 2205 was calculated with Thermo-Calc, the results are presented in **Figure 5**.

As shown in **Figure 5**, σ -phase formation does not occur at temperatures above 930 °C, while the sample at 950 °C still contains some σ -phase. With a decrease of temperature, the austenite weight fraction increases significantly from 28 % at 1200 °C up to 67 % at 860 °C. The change in austenite volume fraction influences the mechanical behaviour of the duplex stainless steel because of the large difference in strength between the ferrite and austenite.

The content of ferrite in the samples was measured with the ferritoscope. The results of the measurements are presented in **Figure 6**.

Figure 6 shows the evolution of the δ -ferrite volume fraction at different test temperatures; the amount of δ -ferrite increases between 850 °C and 1000 °C. According to **Figure 5**, the amount of σ -phase formed is at least a factor of 5 greater than the amount of chi phase. Predictions by Thermo-Calc are relatively good for the amount of ferrite at 1000 °C; the predicted value is about 48 % compared to 46 % measured. Above 850 °C, the amount of austenite decreases considerably with the deformation temperature, which is attributed to the $\gamma \rightarrow \delta$ transformation. The content of ferrite rises at

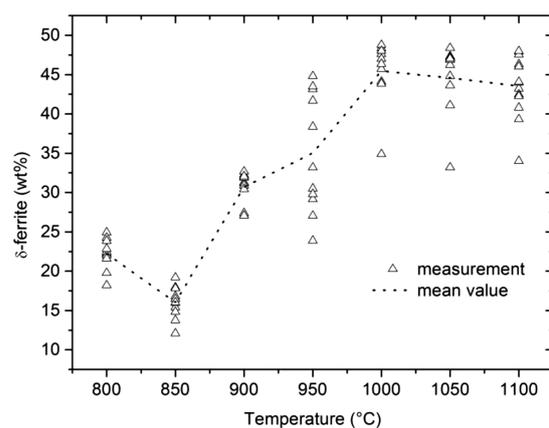


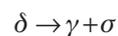
Figure 6: Measured δ -ferrite weight fraction and mean values at different temperatures in the range from 800 °C to 1100 °C

Slika 6: Izmerjen masni delež δ -ferita in srednje vrednosti pri različnih temperaturah v območju od 800 °C do 1100 °C

temperatures from 950 °C to 1100 °C; it is about 45 % at 1100 °C, the rest is austenite.

The hot-working properties greatly depend on the austenite and ferrite content in the steel microstructure; their ratio depends on the temperature, as shown on **Figure 5**. The softening mechanism in ferrite is DRV; it can be observed as the subgrain formation, while the softening mechanism in austenite is the DRX, which is discontinuous, and it mostly occurs on ferrite–austenite phase grain boundaries. The lower driving force for ferrite strain softening is compensated with a higher diffusion rate and a higher mobility of dislocations in a cubic body-centred lattice, that contribute to a faster strain softening process in ferrite.

Another important factor that contributes to the difficult hot-working properties of DSS is the occurrence of the σ -phase. The σ -phase is formed by the eutectoid transformation:



The presence of the σ -phase is the most prominent at 900 °C. Long exposure times at temperatures up to 900 °C and deformation are sufficient conditions for σ -phase formation during hot tensile tests. Duplex stainless steels are more susceptible to the precipitation of intermetallic phases than austenitic steels due to the high Cr and Mo contents and higher diffusion rates in the ferrite phase. The precipitation reaction of σ -phase in austenite is sluggish due to the slow diffusivity of the solute atoms. As the precipitation continues, Cr and Mo diffuse to the σ -phase, leading to the depletion of these elements in the ferrite, especially the Mo content. Therefore, Mo from the inner region of the ferrite diffuses to the σ -phase. The σ -phase nucleates preferentially at the ferrite/ferrite and ferrite/austenite boundaries, and then grows into the adjacent ferritic grains. Mo is the main element controlling secondary-phase precipitation. The σ -phase is a hard, brittle, non-magnetic intermetallic phase, with high Cr and Mo contents.

The partitioning of the elements between the ferrite and austenite takes place during DSS and contributes to the difference in hot strength between the ferrite and austenite. This means that some alloying elements can dissolve preferentially in one phase compared to the other, depending on the nature of the considered chemical element: austenite or ferrite stabilizer. At both extremes, Mo is the element that segregates mostly to ferrite, whereas C and N tend to leave the ferrite. The high N content in austenite is important, as the solute-strengthening effect tends to increase the hot strength of the austenite. The partition of elements changes with temperature. The content of ferrite-forming elements in the austenite phase decreases with temperature, whereas the austenite stabilizer content increases. As a consequence the hot-deformation behaviour of duplex steels can be different at the beginning and at the end of the hot-deformation process. The higher the temperature, the more uniform the element partitioning is between ferrite and austenite.

4 CONCLUSIONS

The following conclusions can be drawn from the experimental work:

The SAF 2205 duplex stainless steel has excellent hot-working properties at temperatures between 950 °C and 1100 °C, while hot working is not recommended at 800 °C.

Difficulties in hot working due to σ -phase precipitation can be accurately predicted by Thermo-Calc software.

The σ -phase is mostly observed below 950 °C. The microstructure of the steel is ferrite + austenite at room temperature, ferrite + austenite + σ from 800 °C to 950 °C and ferrite + austenite at higher temperatures 1000 °C to 1100 °C. The specimens that were tensile tested at lower temperatures (800–900 °C) broke at lower strains due to severe σ -phase precipitation and diminished softening mechanisms. These specimens did not show cavity occurrence 10 mm from the fracture surface, as the fracture was more localized, while the higher temperature tests from 950 °C to 1050 °C had cavities occurring even 10 mm from the fracture surface. The highest testing temperature, however, resulted in a microstructure that showed no apparent damage. This suggests that the dominant reason for failures at temperatures from 950 °C to 1050 °C is the difference in mechanical properties between the ferrite and austenite that are further increased by the differences in the softening mechanisms.

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