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# Effect of microstructure on impact toughness of duplex and superduplex stainless steels

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Properties

## ABSTRACT

**Purpose:** of this paper is to study the effect of heat treatments and resulting changes in microstructure on mechanical properties, mainly impact toughness, of commercial 2205 duplex stainless steel and higher alloy superduplex 2507 grade.
**Design/methodology/approach:** Both steels were submitted to ageing treatments in the temperature range of 500-900 °C with exposure time periods 6 minutes, 1 hour and 10 hours. Light microscope examinations, hardness measurements and impact toughness tests were performed in order to reveal microstructure and changes in mechanical properties.
**Findings:** Obtained results confirm that high temperature service of duplex stainless steels should be avoided. Precipitations of secondary phases (mainly σ phase) strongly deteriorate mechanical properties of steels but some amounts of these phases could be acceptable in the microstructure depending upon the application of the steel.
**Research limitations/implications:** Presence of secondary phases in duplex stainless steel microstructure can be very harmful for its corrosion resistance. This phenomenon is not considered in this study.
**Practical implications:** The accidents during exploitation and errors in processing of duplex stainless steels can result in undesired temperature growth over 500°C. Such events brings question whether the steel can be still exploited or not. The aim of present study is to reveal the effect of thermal cycles on structural changes and mechanical properties of duplex stainless steel and establish the highest acceptable time-temperature conditions for safe operation of the steel.
**Originality/value:** Information available in literature does not clearly indicate what amount of secondary phases existing in duplex stainless steel microstructure can be acceptable. The current study shows that duplex 2205 steel affected by thermal cycles and containing about 10% of sigma phase still exhibit acceptable mechanical properties.
**Keywords:** Ductility; Duplex stainless steels; Heat treatment; Impact toughness

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## 1. Introduction

Duplex stainless steels are very attractive constructional materials for service in aggressive environments. Such steels offer several advantages over the common austenitic stainless steels. The duplex grades are highly resistant to chloride stress corrosion cracking, have excellent pitting and crevice corrosion resistance and are about twice as strong as the common austenitic steels. The term “duplex steel” has come to mean a grade of steel alloyed with 22 to 27 % chromium, 3 to 7 % nickel and up to 4.5% molybdenum which annealed structure consists of about equal parts of austenite and ferrite. These steels are usually selected where both corrosion resistance and high strength is required e.g. for chemical tankers, desalination plants, chemical and petrochemical processes, pipelines and oil and gas separators [1,2].

Mechanical properties of duplex stainless steels are excellent in the temperature range from -50°C to 300°C. When steel is subjected to elevated temperatures numerous different solid state reactions can take place. These lead to the formation of different precipitates resulting in detrimental changes in the properties of the material, especially in its toughness [3].

When duplex steels are exposed to temperatures over 300°C they are susceptible to ”475°C embrittlement”. During heat treatments in temperature range 500 - 900 °C, duplex stainless steels are prone to microstructure changes and precipitation of intermetallic phases. These precipitates are very harmful for the mechanical properties and corrosion resistance of the steel. Therefore, in certain temperature ranges the time when the first precipitate forms is rather short and this fact should be considered when using and designing procedures for duplex steels [4, 5].

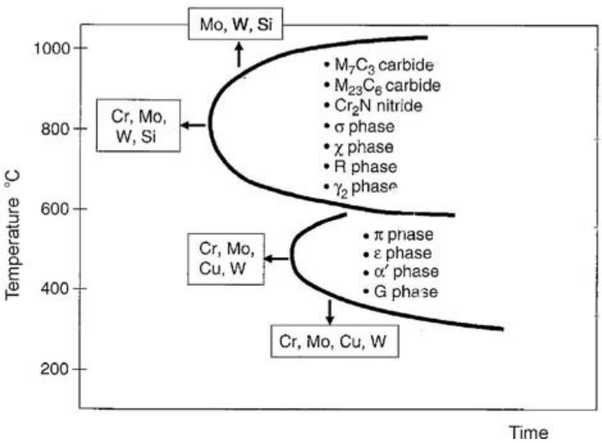


Fig. 1. Schematic TTP diagram for secondary phases in duplex stainless steels [6]

The precipitation tendency is strongly affected by the content of alloying elements and differs between various steel grades. Precipitation is pronounced in the superduplex stainless steels and weld metals because of their higher alloying content [7].

A schematic time-temperature-precipitation (TTP) diagram summarizes the approximate temperature ranges for precipitation of secondary phases in duplex stainless steels is shown in Fig. 1 [6]. This diagram also shows the influence of different alloying elements illustrating that the higher alloying content of superduplex stainless steels makes them more prone to precipitation. Higher alloying contents of Cr, Mo, W, and Si lead to extend the stability range of precipitates and move the nose of TTP curves to shorter times [7].

The most important secondary phases during manufacturing and welding are σ, χ, secondary austenite and chromium nitrides, all formed above 500°C. Decomposition of ferrite to α' is limiting the upper temperature during service. Below 500°C precipitation reactions are comparatively slow and of little importance for embrittlement. Secondary phases reported to form in duplex stainless steels and weld metals above 500°C are presented in Table 1 [7].

Table 1.
Secondary phases formed in duplex stainless steels at temperatures above 500°C [7]

| Type of precipitate | Nominal chemical formula | Temperature range (°C) |
|---|---|---|
| Ferrite (α) | - | - |
| Austenite (γ) | - | - |
| σ | Fe-Cr-Mo | 600 - 1000 |
| Chromium nitride | $Cr_2N$ | 700 - 900 |
| Chromium nitride | CrN | - 1000 |
| χ | $Fe_{36}Cr_{12}Mo_{10}$ | 700 - 900 |
| R | Fe-Cr-Mo | 550 - 800 |
| π | $Fe_7Mo_{13}N_4$ | 550 - 600 |
| τ | - | 550 - 650 |
| $M_7C_3$ | - | 550 - 650 |
| $M_{23}C_6$ | - | |

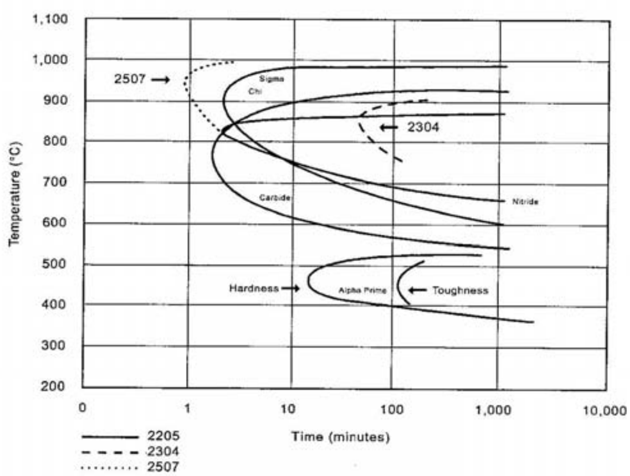


Fig. 2. Isothermal Precipitation Diagram for duplex stainless steels, annealed at 1050°C [8]

An isothermal precipitation diagram for 2304, 2205, and 2507 duplex stainless steels is shown in Figure 2. The chromium carbide and nitride precipitation begins at the relatively slow time of 1-2 minutes at temperature. This is slower than in the ferritic grades or the highly alloyed austenitic grades, and is due to high solubility of carbon and nitrogen in the low nickel austenite phase and possibly to a retardation effect of nitrogen on the carbide precipitation. The carbide and nitride formation kinetics are slightly affected by chromium, molybdenum, and nickel, so all the nitrogen-alloyed duplex stainless steel grades have kinetics similar to 2205 in regard to these precipitates. Sigma and chi precipitation occurs at higher temperatures. Superduplex grades that are more highly alloyed in chromium, molybdenum, and nickel will have more rapid sigma and chi kinetics than 2205 (Fig. 2) [8].

Although numerous research studies describing precipitation phenomena in Cr-Ni stainless steels have been already presented [4, 9 - 19] it is still not clear evidence as to the amount of secondary phases that are acceptable. The objective of the present work is to study the effect of heat treatments and the resulting changes in microstructure on the mechanical properties, mainly toughness, of commercial 2205 duplex stainless steel and high alloy superduplex 2507 grade.

## 2. Experimental

The chemical compositions of investigated 2205 duplex stainless steel (1.4462 acc. to PN-EN 10088-1:2007) and 2507 superduplex stainless steel (1.4507 acc. to PN-EN 10088-1:2007) are given in Table 2. The specimens were obtained from plates 12 mm thick. Both plates were delivered after solution annealing treatment at 1050°C.

Ageing treatments were performed by isothermal holding in the vacuum furnace for 6 minutes, one hour and 10 hours at temperatures between 500 and 900 °C before water quenching.

Table 2.
Chemical composition of tested 2205 and 2507 duplex stainless steels, acc. to control analysis

| duplex steel | Chemical composition, wt. % | | | | | |
|---|---|---|---|---|---|---|
| | C | Mn | Cr | Ni | Mo | N |
| 2205 1.4462 | 0.017 | 1.50 | 21.9 | 5.7 | 3.0 | 0.17 |
| 2507 1.4507 | 0.030 | 0.87 | 25.12 | 5.82 | 3.59 | 0.29 |

### 2.1. Microstructure

Microstructure of the specimens was examined after chemical etching in Murakami reagent. The Multi-Scan image analysis system and ferritscope magnetic tester were used to determine the amounts of various alloy phases.

Ageing in temperature range of 500-900 °C caused the formation of intermetallic phases, $Cr_2N$ and secondary austenite. Because of the large volume fraction of the σ-phase, this is the most important phase besides ferrite and austenite.

The structure of the steels when received is shown in Fig.3. The rolling of duplex steels forms a fine elongated lamellar structure of austenite and ferrite. Ferrite is the light etched phase. Ferrite content at duplex 2205 steel was estimated on 44 - 47 % and respectively 52 - 55 % at superduplex 2507 steel.

Samples of both steels aged at 500 °C even for the longest time do not exhibit significant phase transformations. The amount of ferrite slightly decreased due to nucleation of secondary austenite $\gamma_2$. Precipitations of α' phase was not recorded when light microscope was used for observations.

At temperature between 600 and 900 °C ferrite → austenite transformation and σ phase precipitation took place. Secondary austenite formation is independent of σ phase precipitation although these two transformations occur together and are both related to ferrite phase. Ferrite is unstable at elevated temperatures because the diffusion rates of alloying elements are 100 times faster than the corresponding values in the austenite. Moreover, the ferrite is enriched in chromium and molybdenum, which promote the formation of intermetallic phases.

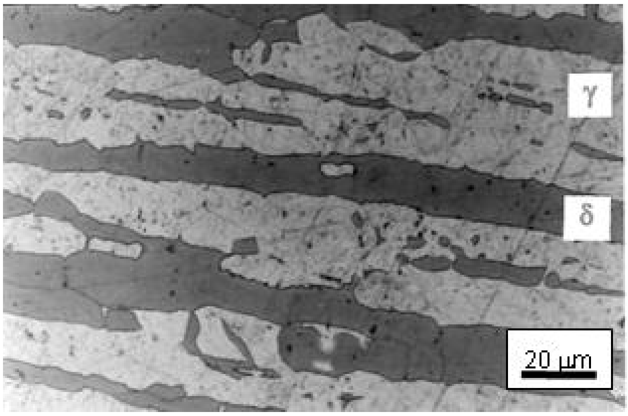


Fig. 3. Microstructure of 2205 duplex stainless steel in as received condition

Sigma phase appeared in duplex 2205 steel microstructure after 10 hours ageing at 600 °C or 700 °C. Ageing at 800 °C resulted in σ phase formation after less than 1 hour and less than 6 minutes during ageing at 900 °C. Superduplex 2507 steel was more prone to σ phase precipitation. Small amounts of this phase were recorded after 6 minutes ageing at 700 °C, but ageing in the same time at 800 °C resulted in advanced ferrite phase disintegration. Only samples aged at 500 °C and at 600 °C for shorter times (6, 60 min) were free of σ phase precipitates.

Sigma phase first forms inside ferrite grains and ferrite/ferrite or ferrite/austenite grain boundaries. After extended ageing times, σ phase creates according to the eutectoid reaction $\delta \rightarrow \sigma + \gamma_2$. The σ phase formation due to the eutectoid reaction is shown in Fig. 4.

At the temperature 900 °C and higher ferrite can transform into σ phase without accompanying austenite formation and the composition of sigma phase is very close to that of the ferrite. This kind of transformation is shown in Fig. 5.

The changes in ferrite content after various ageing treatment conditions for both investigated steels are shown in Figs. 6 and 7. The decrease in ferrite fraction is most emphasized at 800 °C where almost all ferrite content can be transformed into other phases, mainly σ and $\gamma_2$. Comparison of tendency to decomposition of ferrite structure for both tested steels show

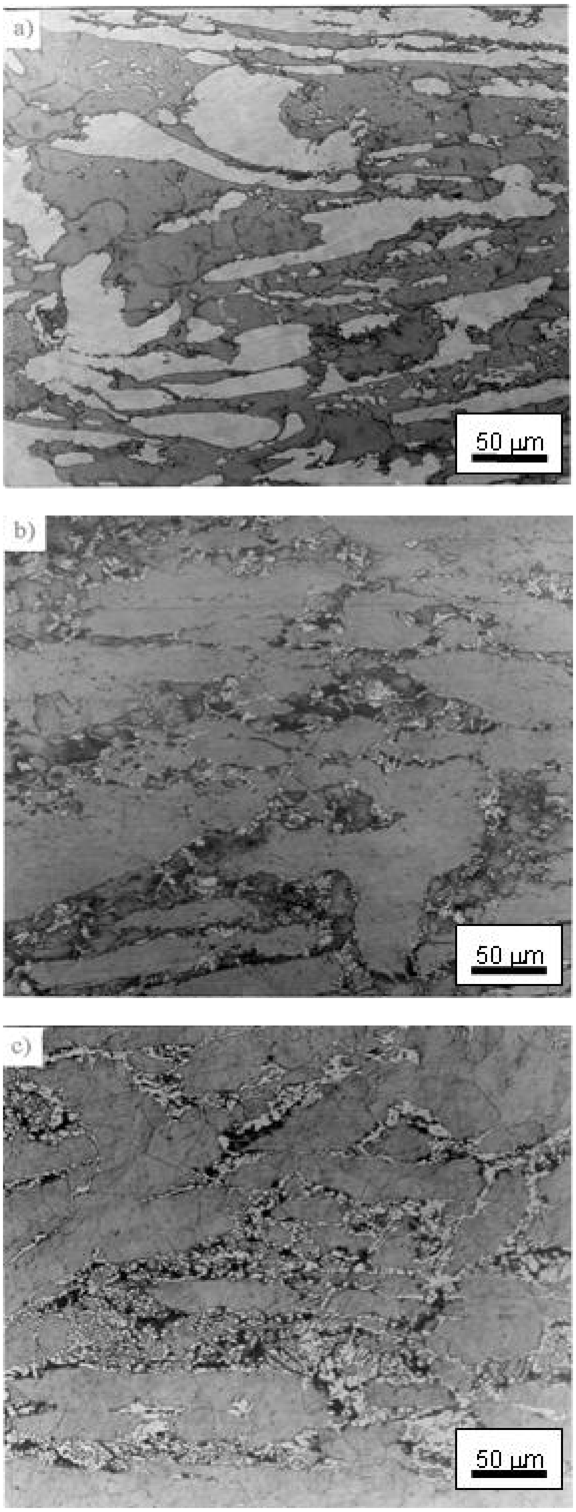


Fig. 4. σ-phase formation due to the eutectoid reaction $\delta \rightarrow \sigma + \gamma_2$. 2205 duplex steel. Ageing at 800°C. a) 6 min., b) 1 h., c) 10 h

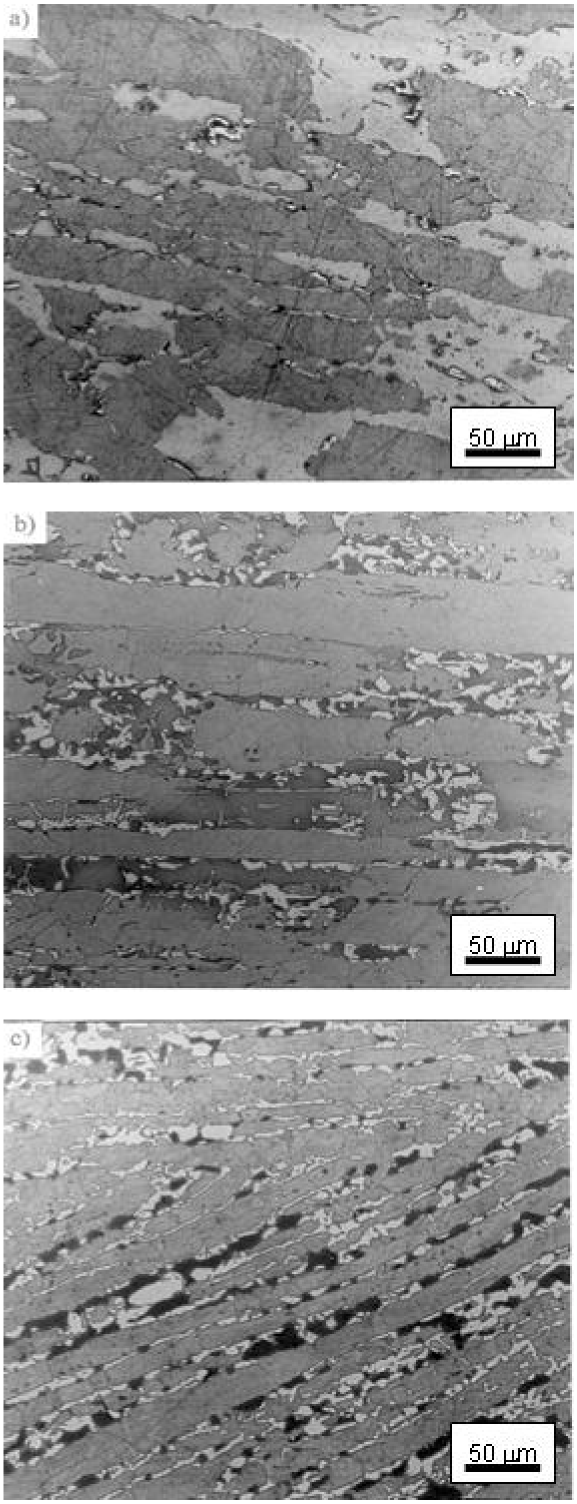


Fig. 5. σ-phase transformation directly from ferrite. 2205 duplex steel. Ageing at 900°C. a) 6 min., b) 1 h., c) 10 h

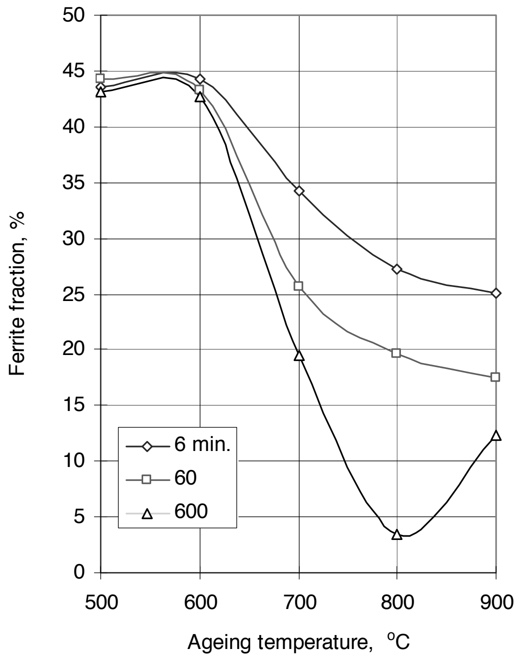


Fig. 6. Changes of ferrite fraction as a function of ageing conditions for 2205 duplex steel

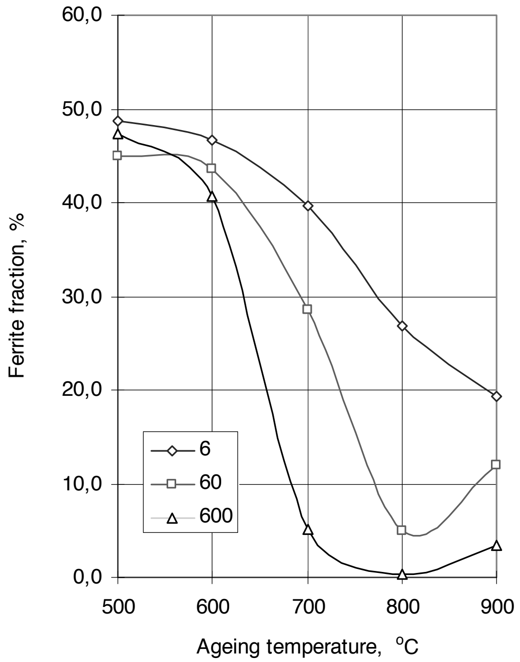


Fig. 7. Changes of ferrite fraction as a function of ageing conditions for 2507 superduplex steel

similar behaviour for short times (6 min.). Longer exposure time results in instability of ferrite and greater reduction of this phase in 2507 superduplex steel microstructure. The amount of ferrite is higher after ageing at 900 °C than at 800 °C due to direct transformation ferrite into σ phase at higher temperatures.

## 2.2. Mechanical properties

The hardness tests were performed with the use of the Vickers method according to the PN-EN 6507-1:2007 standard. The initial hardness of base materials was 254 HV10 for 2205 steel and 270 HV10 for 2507 steel. Ageing at 500 °C increased slightly hardness of both steels because of α' phase precipitations. Following ageing at 600 °C (and 700 °C for 2205 steel) resulted in little decrease of steels hardness (Figs. 8 and 9) probably due to creation of $\gamma_2$ phase. Further ageing at higher temperatures causes precipitation of σ phase and significant hardness increase. Maximum hardness of 348 HV10 was recorded for 2205 steel after 10 hour ageing at 800 °C. Superduplex 2507 steel show higher hardness reaching 438-441 HV10 after 10 hour ageing at 700 and 800 °C. The absolute value of hardness increase of 94 HV10 was obtained for 2205 steel, and 171 HV10 for 2507 steel. Ageing at 900 °C resulted in lesser hardness increase than ageing at 800 °C. It is related to the little amount of precipitates and higher ferrite content (Figs. 8, 9).

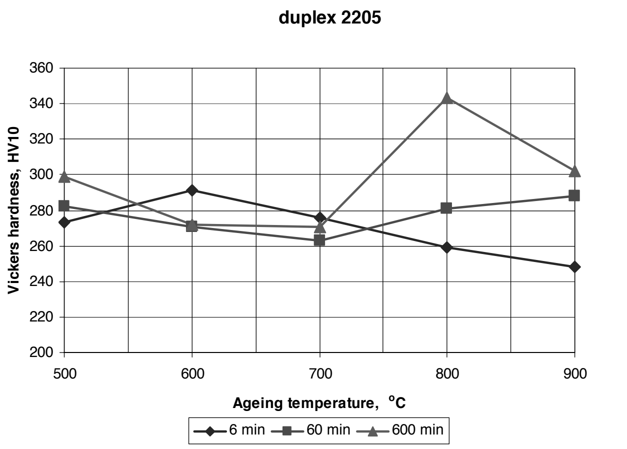


Fig. 8. The influence of ageing time and temperature on hardness of 2205 duplex stainless steel

Charpy V impact tests were performed at ambient temperature (+20 °C). The specimens were taken in the longitudinal direction of the plate. All samples in as received condition showed high fracture energy. These samples were broken but some of them absorbed almost all the energy of a swinging hammer (e.g. 300 J). Embrittlement behaviour of aged samples was different for duplex 2205 and superduplex 2507 steels (Figs.10, 11). Duplex 2205 is less sensitive to embrittlement. Ageing for short times (6 min.) at temperatures up to 800 °C did not remarkably decreased impact energy. Ageing at 500 °C for 10 hours and at 600 °C for 1 hour maintain high toughness on the level of 300 J, Fig. 10.

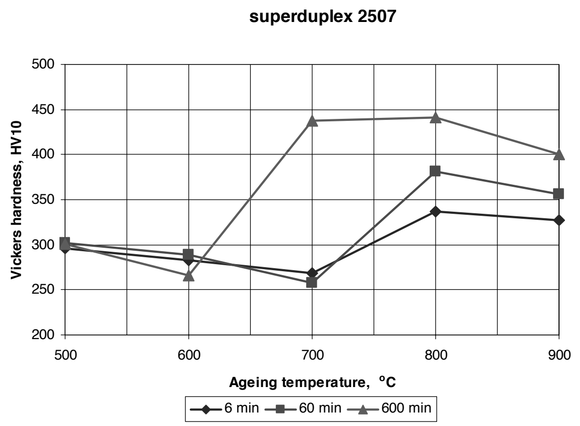


Fig. 9. The influence of ageing time and temperature on hardness of 2507 superduplex stainless steel

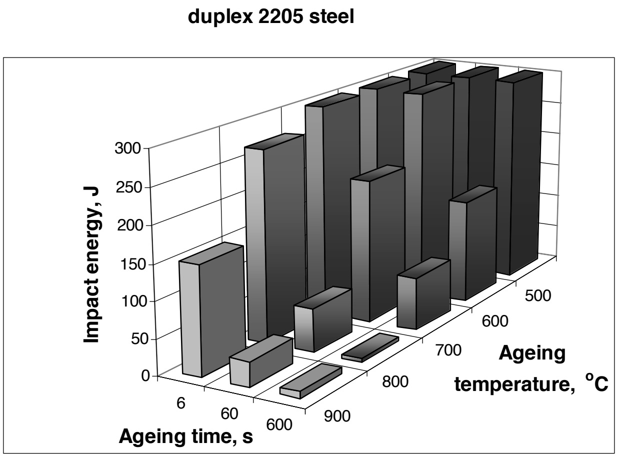


Fig. 10. The influence of ageing time and temperature on impact energy of 2205 duplex stainless steel

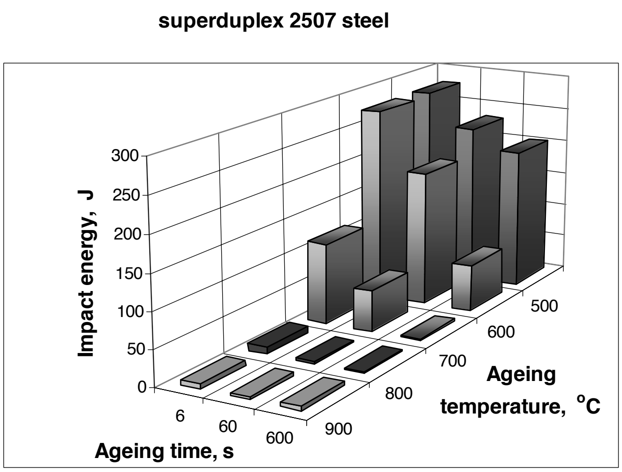


Fig. 11. The influence of ageing time and temperature on impact energy of 2507 superduplex stainless steel

Superduplex 2507 steel show high toughness only after ageing for 6 minutes at 500 and 600 °C. Other applied thermal cycles deteriorate remarkably steel plasticity. Ageing at 800 and 900 °C, regardless of exposure time, caused almost complete degeneration of toughness, Fig 11.

Next investigations were performed with the use of scanning electron microscopy in order to correlate impact toughness with fracture mode. All tests were conducted on Charpy V specimens of 2507 steel.

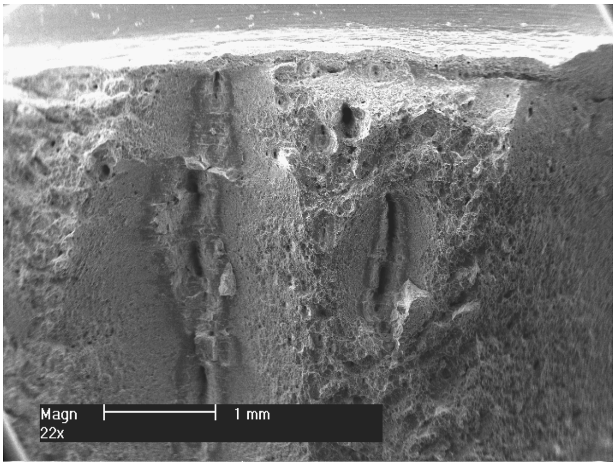


Fig. 12. Fracture surface of base material of 2507 steel

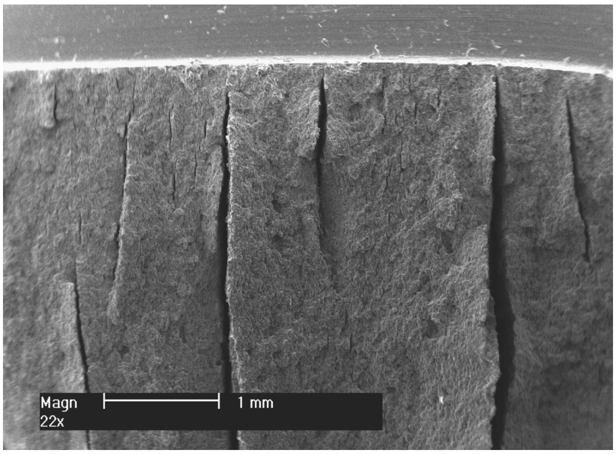


Fig. 13. Fracture surface of specimen treated at 800°C for 1 hour

The macroscopic appearance of fracture surface show a lot of secondary cracks (splits) or crackings grow perpendicular to surface area Figs. 12, 13. The splits propagate through the ferrite phase or ferrite-austenite boundaries [20].

The fracture area of base material and of samples treated at 600 and 700 °C for up to 1 hour is ductile and has a dimple rupture mode of fracture. The dimples are elongated in the stress direction, Fig. 14. Higher ageing temperatures and longer exposure times resulted in change of fracture mode. Mixed cleavage-like mode of fracture with small areas with dimples is characteristic for samples treated at 700 °C, Fig.15. Brittle fracture was recorded for samples treated at 800 and 900 °C, Fig. 16.

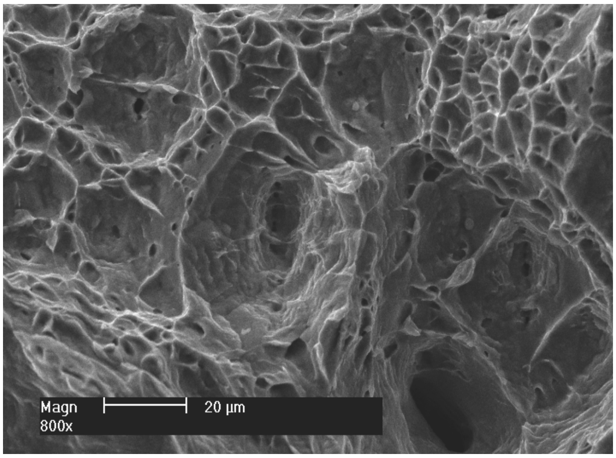


Fig. 14. Fracture surface of specimen treated at 500°C for 1 hour

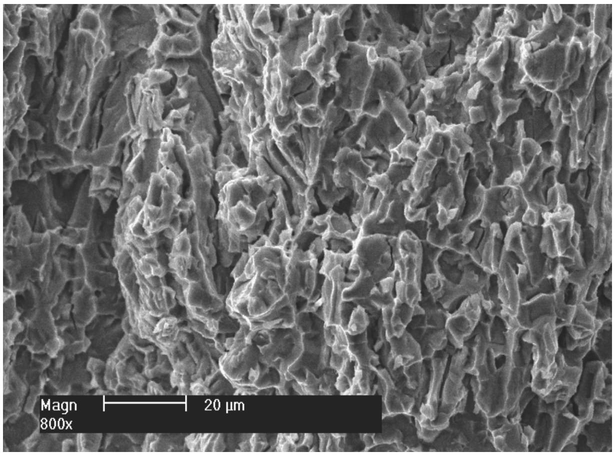


Fig. 15. Fracture surface of specimen treated at 700°C for 1 hour

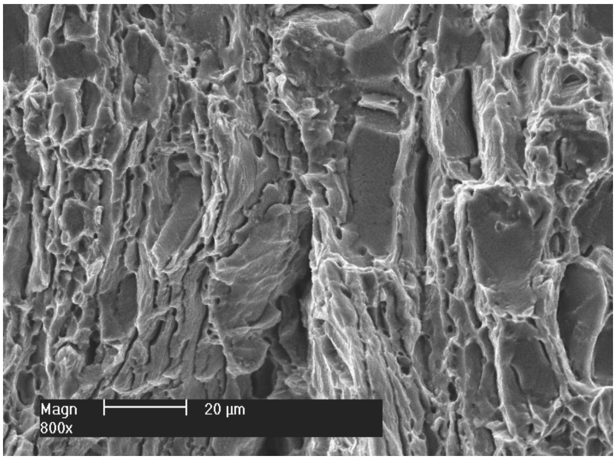


Fig. 16. Fracture surface of specimen treated at 900°C for 1 hour

## 3. Discussion

Performed tests have shown the negative effect of precipitates, mainly sigma phase, on the plasticity of duplex 2205 and superduplex 2507 stainless steels. Toughness of both steels decreased considerably when some amount of σ phase appeared in the steel microstructure. Sigma phase is tetragonally close-packed and is of a very brittle nature. So it is apparent that more σ phase in steel structure will provide a sharp decrease of plastic properties. The influence of σ phase content in microstructure of 2205 and 2507 duplex steels on impact energy is shown in Fig. 17 and Fig. 18.

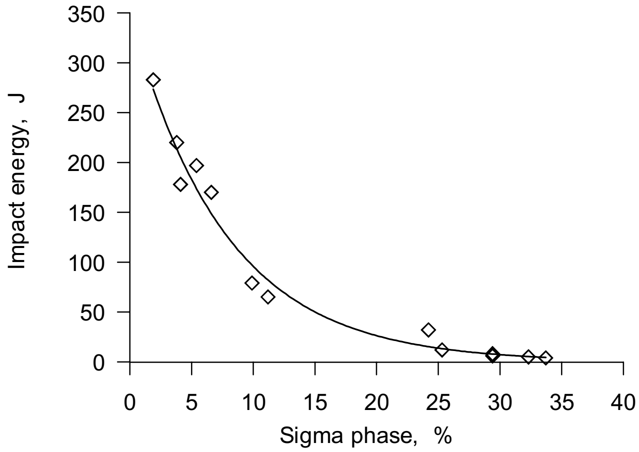


Fig. 17. The influence of σ-phase content on impact energy of 2205 duplex stainless steel

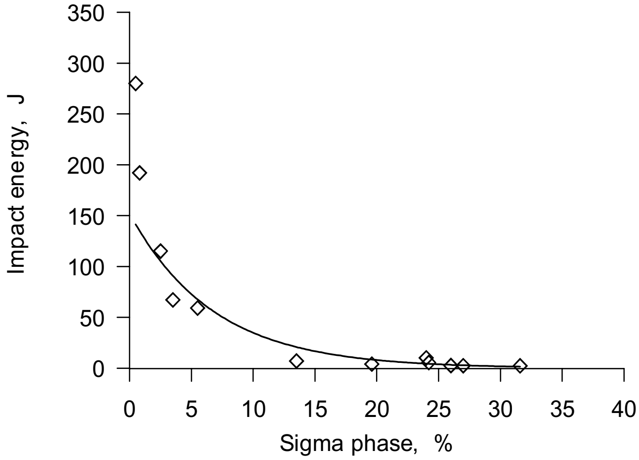


Fig. 18. The influence of σ-phase content on impact energy of 2507 superduplex stainless steel

A common design criterion according to requirements of Ship Classification Societies is that the impact energy obtained on Charpy V specimens should be higher than 27 J or 40 J depending on testing temperature and direction of specimens in wrought products. Figs. 17 and 18 show that examined 2205 steel could be used in room temperatures (+20 °C) with a content lower than 14% of σ phase (KV>40 J), and superduplex 2507 steel with maximum 8% of σ phase in the microstructure. According to the reported results [2] the acceptable amount of σ is about 4% but Charles and co-workers [6] claim that even small amounts of σ phase leads to a sharp decrease in toughness, which rapidly falls to a level which is inadequate for industrial applications.

The last opinion is adequate to results obtained in this work for 2507 steel. The sharp decrease of toughness was observed for very low amounts of σ phase in microstructure.

Duplex 2205 steel seems to be less prone to embrittlement. It is very important in exploitation and processing of this steel. The accidents during exploitation and errors in processing of duplex stainless steels can result in undesired temperature growth over 500 °C. Moreover some industrial processes, like hot straightening, require local heating for short times. Such operations or events brings question whether the steel can be still exploited or not.

The reported results confirm that the high temperature service of duplex stainless steels should be avoided. Precipitation of secondary phases strongly influences mechanical properties but some amounts of σ phase could be acceptable in 2205 grades depending upon the application of the steel.

## 4. Conclusions

- Isothermal heat treatment of 2205 and 2507 duplex stainless steels in the temperature range 500-900 °C cause the precipitation of the intermetallic phases (mainly s-phase) and generation of the secondary austenite.
- Sigma phase can form according to an eutectoid reaction $\delta \rightarrow \gamma + \sigma$ or directly from the ferrite phase.
- Both steels duplex 2205 and superduplex 2507 are prone to embrittlement when exposed to thermal cycles over 500 °C.
- Superduplex 2507 steel show great tendency to precipitation of intermetallic phases and in consequence sharp decrease of toughness after ageing at elevated temperatures, so high service temperature or incidental heating should be avoided.
- Duplex 2205 steel is less prone to embrittlement. Short time heating in the temperature range 500-700°C can be acceptable when mechanical properties are taken into consideration.

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