Influence of ageing treatment temperature and duration on \(\sigma\)-phase precipitation and mechanical properties of UNS S32750 SDSS alloy

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**GRAPHICAL ABSTRACT**

**ABSTRACT**

**Introduction:** Super-Duplex Stainless Steels (SDSS) proved an excellent potential for its use in many chemical and offshore applications due to their both high mechanical properties and a high corrosion resistance in chloride ion solutions.

**Objectives:** This study evaluates the influence of ageing treatment temperature and duration on \(\sigma\)-phase precipitation and mechanical properties of UNS S32750 SDSS alloy.

**Methods:** The influence of ageing treatment on microstructural features was analysed by SEM-EBSD (Scanning Electron Microscopy - Electron Backscatter Diffraction) technique, while on mechanical properties by tensile and impact testing techniques.

**Results:** The obtained results show that for the short duration ageing treatments the \(\sigma\)-phase nucleates, within the \(\delta\)-phase matrix, at the \(\delta/\gamma\) grains boundary by the \(\delta \rightarrow \sigma\) precipitation, while for long duration ageing treatments the \(\sigma\)-phase nucleates, within the \(\delta\)-phase matrix, at the \(\delta/\delta\) grains boundary, or in other favourable nucleation sites, due to the eutectoid decomposition \(\delta \rightarrow \sigma + \gamma_2\). Experimental data showed that at low temperatures, i.e. 780 °C, in order to induce the precipitation of \(\sigma\)-phase, the minimum incubation time is situated close to 20 min, and that increasing treatment temperature decreases the minimum incubation time, with at 850 °C and 920 °C the \(\sigma\)-phase being firstly detected at 5 min. The \(\sigma\)-phase precipitation shows the highest precipitation kinetics at 850 °C, when the maximum
weight-fraction is obtained for each treatment duration when compared with ageing treatments performed at 780 °C and 920 °C.

Conclusion: All presented data brings valuable insight into the σ-phase precipitation phenomena and its influence on UNS S32750 SDSS alloy’s exhibited mechanical properties and, also, can provide researchers and industrial steel processors a guide regarding the selection of optimal ageing treatment parameters to avoid/minimise the embrittlement induced by the precipitation of deleterious σ-phase.

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Introduction

During the last decades, the Super-Duplex Stainless Steeles (SDSS) proved an excellent potential for its use in many chemical and offshore applications, where both high mechanical properties and a high corrosion resistance in chloride ion (Cl\textsuperscript{-}) solutions are a must. The proper combination of mechanical properties and corrosion resistance resides in the SDSS alloys balanced microstructure, which contains approximately equal volume fractions of ferrite (δ-phase) and austenite (γ-phase) phases [1–4]. In the SDSS alloys, the austenite (γ-phase) ensures alloy’s ductility and weldability while the ferrite (δ-phase) raises alloy’s strength properties and corrosion (pitting, stress and crevice corrosion) resistance [5–9].

Considering the chemistry of SDSS alloys, especially the high weight-fraction of austenite-stabilising (C, Ni, N, Cu) and ferrite-stabilising (Cr, Mo, W) elements, in order to maintain both high mechanical properties and high corrosion resistance, the alloying elements should be kept dissolved in the parent ferrite (δ-phase) and austenite (γ-phase) phases. Also, the precipitation of various undesirable compounds, such as: carbo-nitrides (M\textsubscript{23}C\textsubscript{6}, M\textsubscript{7}C\textsubscript{3}) and intermetallic secondary phases, such as: sigma (σ-phase) and chi (χ-phase), must not be allowed [10–15]. It was showed that at temperatures above 600 °C, due to the high diffusion rates of Cr and Mo in the body-centred cubic (bcc) structure of δ-phase in comparison with the face-centred cubic (fcc) structure of γ-phase, nucleation and growth of compounds and secondary phases within the δ-phase occur, which act as stress-intensification sites and, therefore, drastically decrease the alloy’s strength properties (i.e. toughness) and corrosion resistance [16–20].

Research proved that one of the most important precipitated secondary phases is represented by the σ-phase, due to its fast formation kinetics over a wide range of temperatures, generally between 700 °C and 1000 °C. Also, it was showed that the σ-phase promotes strong embrittlement, drastically decreasing the toughness and corrosion resistance [21–27]. In this context, the aim of the present work is to study the influence of ageing treatment temperature and treatment duration on the σ-phase precipitation and mechanical properties of a commercially available UNS S32750 SDSS alloy.

Materials and methods

An UNS S32750 SDSS alloy, with the average chemical composition: Cr = 26.03%wt; Ni = 6.57%wt; Mo = 3.81%wt; Mn = 0.84%wt; Si = 0.43%wt; Cu = 0.14%wt, was thermomechanically (TM) processed by the processing route presented in Fig. 1. The as-received (AR) UNS S32750 alloy was hot-deformed, at a temperature of 1100 °C, by rolling (HR), in 6 rolling steps, with a total deformation degree (thickness reduction) of approx. ε\textsubscript{total} ≈ 65%. All HR samples were solution treated (ST) at 1050 °C for 30 min and water quenched (WQ), in order to obtain a defect-free microstructure. After ST, the TM processing continued with ageing treatments performed at 780 °C (A1), 850 °C (A2) and 920 °C (A3). Each ageing treatment (A) was performed using the following treatment durations: 5 min, 10 min, 20 min, 30 min, 60 min and 90 min. All ageing treated samples were water quenched to freeze the high-temperature microstructure. The aim of the performed ageing treatments was to assess the influence of ageing treatment temperature and treatment duration on σ-phase precipitation and exhibited mechanical properties. The selection procedure of the ageing treatment temperatures considered previously reported data for the necessary temperature and duration needed to initiate the precipitation of the σ-phase, which showed a necessary temperature above 780 °C and a necessary time in the order of minutes [27–30].

All TM processed specimens were microstructurally investigated to observe the microstructural constituent phases and occurred changes during TM processing, by SEM-EBSD (Scanning Electron Microscopy - Electron Backscatter Diffraction). Also, their mechanical properties were investigated, by tensile and impact testing techniques, in order to determine the absorbed energy, ultimate tensile strength, yield strength and elongation to fracture for each TM processed specimen.

A SEM-TESCAN Vega II-XMU microscope equipped with a BRUKER Quantax eflash EBSD detector was used to perform all SEM-EBSD measurements. The SEM-EBSD analysis procedure, applied to all investigated samples, is presented and detailed elsewhere [30]. All data was rounded to 0.1%. The chemical composition of all observed phases was quantified using the TESCAN Vega II-XMU microscope coupled with a BRUKER Quantax xFlash 6/30 EDS detector. In order to compute the average chemical composition of each phase, 5 random measurements were performed. All data was rounded to 0.01 wt%.

The mechanical properties were investigated using an INSTRON 3382 tensile testing equipment and an INSTRON 450 MPX-v2-J1 impact testing equipment. The applied investigation procedure (testing conditions and samples configuration) is presented and detailed elsewhere [30]. All data was rounded to 1 MPa, 0.5% and 0.1 J.

Results

Fig. 2 shows typical SEM-EBSD microstructure images corresponding to ageing-treated at 780 °C - A1 state (Fig. 2.a), ageing-treated at 850 °C samples - A2 state (Fig. 2.b) and ageing-treated at 920 °C - A3 state (Fig. 2.c), with treatment durations ranging from 5 min to 90 min. The following phases were identified: γ-phase (primary austenite) and γ\textsubscript{2}-phase (secondary austenite) - coloured in red, δ-phase (ferrite) - coloured in blue, σ-phase (FeCrMoNi) - coloured in yellow and χ-phase (FeCrMo) - coloured in green. The morphology analysis of the main δ-phase and γ-phase shows that in all cases the microstructure consists of an elongated γ-phase, along the rolling direction, within the δ-phase matrix. The prior deformed grains, due to intense hot deformation by rolling (HR), are fully recrystallized after applied solution treatment (ST). Besides primary γ-phase the secondary γ\textsubscript{2}-phase can be observed, mainly at δ / δ grain boundaries. The precipitation of γ\textsubscript{2}-phase is induced by heterogeneous nucleation, in sites where the precipita-
tion phenomena is enhanced due to the nitrogen supersaturation of δ-phase. The γ2-phase, also, can precipitate within the δ-phase grains in other preferential nucleation sites, i.e. next to microstructure inclusions [31,32]. The γ2-phase induced after ST treatment shows an average grain-size close to (2–3) μm (Fig. 2.a).

Analysing the case of ageing-treated at 780 °C/A1 state (Fig. 2.a), one can observe that the σ-phase is firstly observed for a treatment duration of 20 min, when the σ-phase is identified at the γ-phase/δ-phase interface, suggesting a δ → σ precipitation. The same behaviour is observed for a treatment duration of 30 min, the observed σ-phase showing the same morphology, but higher weight-fraction and grain-size. Starting with a treatment duration of 60 min, one can notice that beside the γ-phase/δ-phase interfaces, the σ-phase is nucleating within the δ-phase grains together/accompanied by the nucleation of adjacent γ2-phase, indicating a δ → σ + γ2 eutectoid decomposition [19,33,34]. The same behaviour is observed, also, for a treatment duration of 90 min, almost all δ-phase is transformed in σ-phase + γ2-phase and, the weight-fraction and average grain-size of both σ-phase and γ2-phase continuously increase along with the treatment duration. No ν-phase is observed/identified. Analysing the case of ageing treatments performed at 920 °C (Fig. 2.c), one can notice that firstly the σ-phase is identified for a treatment duration of 5 min, when the σ-phase is observed at the γ-phase/δ-phase interface. Increasing the ageing treatment duration leads to nucleation of both σ-phase and γ2-phase, within the δ-phase grains, by the δ → σ + γ2
eutectoid decomposition and the weight-fraction and average grain-size of both \(\sigma\)-phase and \(\gamma\_r\)-phase are continuously increasing with an increase in treatment duration. Comparing the case of ageing treatment performed at 920 °C (Fig. 2.c) with the case of ageing treatment performed at 850 °C (Fig. 2.b), one can notice the following: firstly, for each treatment duration smaller weight-fractions (almost half) of precipitated \(\sigma\)-phase and \(\gamma\_r\)-phase are recorded and, secondly, the weight-fraction of precipitated \(\gamma\_r\)-phase is much higher.

The following mechanical properties were determined during mechanical testing (see Table 1): absorbed energy during impact testing (\(E\)), ultimate tensile strength (\(\sigma_{ut}\)), 0.2 yield strength (\(\sigma_{0.2}\)) and elongation to fracture (\(\varepsilon_t\)).

**Discussion**

In depth microstructure and mechanical behaviour analysis of ageing treated sates A1 (treatment performed at 780 °C), A2 (treatment performed at 850 °C) and A3 (treatment performed at 920 °C) reveal that in each case a different behaviour is recorded. If one analyses the weight fraction evolution of all observed phases, one will identify different kinetics of \(\sigma\)-phase and \(\gamma\_r\)-phase precipitation from the parent \(\delta\)-phase. Considering that both \(\gamma\)-phase and \(\gamma\_r\)-phase belong to the same crystalline system, and both are characterized by very close lattice parameters, it can induce difficulties in distinguishing between them when using SEM-EBSD measurements and therefore, usually, both are quantified together. In order to discriminate between \(\gamma\)-phase and \(\gamma\_r\)-phase, the following assumption was considered: in all investigated cases the \(\gamma\_r\)-phase grains show an average grain-size bellow 10 \(\mu\)m. The limit value of 10 \(\mu\)m was selected after morphological and grain-size dispersion analysis of the \(\gamma\_r\)-phase.

Fig. 3.a shows the weight fraction variation of \(\gamma\)-phase, \(\delta\)-phase, \(\sigma\)-phase and \(\gamma\_r\)-phase, during ageing treatment performed at 780 °C, with treatment duration from 5 min to 90 min (A1 states). One can observe the following. Firstly, the weight fraction of primary \(\gamma\)-phase suffers almost no changes, remaining constant during ageing treatment. Secondly, for a treatment duration exceeding 20 min the weight fraction of the parent \(\delta\)-phase is continuously decreasing with the increase in treatment duration due to the precipitation of both \(\sigma\)-phase and \(\gamma\_r\)-phase; the half point, in which half of the initial weight fraction of \(\delta\)-phase is transformed, is placed at a treatment duration exceeding 90 min, indicating a much higher transformation speed in comparison with ageing treatments performed at 850 °C (A2 states). Thirdly, increasing treatment duration leads to an increase in weight fraction of both \(\sigma\)-phase and \(\gamma\_r\)-phase, after a treatment duration of 90 min an average weight fraction, close to 39.2%, is recorded for \(\sigma\)-phase and, close to 71.1%, for \(\gamma\_r\)-phase. Also, one can notice a much higher fraction of precipitated \(\sigma\)-phase in comparison with \(\gamma\_r\)-phase. Fig. 3.b shows the weight fraction variation of all phases during ageing treatment performed at 920 °C (A3 states). One can observe the following. Similarly to the case of ageing treatment performed at 780 °C or 850 °C, the weight fraction of primary \(\gamma\)-phase suffers almost no changes, remaining constant during treatment. Starting with a treatment duration exceeding 5 min, the weight fraction of the parent \(\delta\)-phase is, also, continuously decreasing, due to precipitation of \(\sigma\)-phase and \(\gamma\_r\)-phase, after 90 min the \(\delta\)-phase showing a weight fraction close to 2.2%, being almost fully transformed. The half point, in which half of the initial weight fraction of \(\delta\)-phase is transformed, is placed at a treatment duration exceeding 90 min, indicating a much lower transformation speed in comparison with the A1 and A2 states. Increasing treatment duration leads to an increase in the weight fraction of precipitated secondary phases (\(\sigma\)-phase and \(\gamma\_r\)-phase), after a treatment duration of 90 min an average weight fraction, close to 12.4%, is recorded for \(\sigma\)-phase.

![Table 1](image-url)

**Table 1**
Mechanical properties recorded for processed UNS S32750 SDSS alloy.

| Structural state | Mechanical properties | Ultimate tensile strength, \(\sigma_{ut}\) [MPa] | 0.2 yield strength, \(\sigma_{0.2}\) [MPa] | Elongation to fracture, \(\varepsilon_t\) [%] | Absorbed energy, \(E\) [J] |
|-----------------|----------------------|------------------------------------------|------------------------------------------|------------------------------------------|--------------------------|
| Solution treated ST |                       | 816 ± 13                                  | 527 ± 13                                  | 60.5 ± 3.5                               | 142.2 ± 2.5              |
| A1.1: T = 780 °C; t = 5 min; WQ |                       | 819 ± 12                                  | 528 ± 13                                  | 60.0 ± 3.5                               | 106.6 ± 2.2              |
| A1.2: T = 780 °C; t = 10 min; WQ |                       | 828 ± 10                                  | 528 ± 13                                  | 58.0 ± 3.0                               | 85.0 ± 2.3               |
| A1.3: T = 780 °C; t = 20 min; WQ |                       | 814 ± 11                                  | 525 ± 12                                  | 54.5 ± 3.5                               | 25.5 ± 1.6               |
| A1.4: T = 780 °C; t = 30 min; WQ |                       | 822 ± 13                                  | 537 ± 14                                  | 47.0 ± 3.0                               | 14.8 ± 0.9               |
| A1.5: T = 780 °C; t = 60 min; WQ |                       | 828 ± 12                                  | 534 ± 12                                  | 50.0 ± 1.0                               | 2.4 ± 0.5                |
| A1.6: T = 780 °C; t = 90 min; WQ |                       | 826 ± 13                                  | 535 ± 14                                  | 40.0 ± 1.0                               | 2.4 ± 0.5                |
| A2.1: T = 850 °C; t = 5 min; WQ |                       | 826 ± 13                                  | 534 ± 13                                  | 43.0 ± 3.0                               | 62.8 ± 2.5               |
| A2.2: T = 850 °C; t = 10 min; WQ |                       | 845 ± 14                                  | 549 ± 14                                  | 31.5 ± 2.5                               | 12.3 ± 1.7               |
| A2.3: T = 850 °C; t = 20 min; WQ |                       | 849 ± 11                                  | 546 ± 12                                  | 12.5 ± 1.5                               | 3.1 ± 0.6                |
| A2.4: T = 850 °C; t = 30 min; WQ |                       | 843 ± 10                                  | 544 ± 11                                  | 3.0 ± 1.0                                | 1.9 ± 0.7                |
| A2.5: T = 850 °C; t = 60 min; WQ |                       | 848 ± 12                                  | 548 ± 12                                  | 2.0 ± 1.0                                | 1.8 ± 0.6                |
| A2.6: T = 850 °C; t = 90 min; WQ |                       | 841 ± 11                                  | 554 ± 14                                  | 1.0 ± 0.5                                | 1.2 ± 0.5                |
| A3.1: T = 920 °C; t = 5 min; WQ |                       | 834 ± 13                                  | 554 ± 14                                  | 34.0 ± 3.0                               | 37.7 ± 2.2               |
| A3.2: T = 920 °C; t = 10 min; WQ |                       | 858 ± 14                                  | 562 ± 12                                  | 24.0 ± 2.5                               | 9.3 ± 1.8                |
| A3.3: T = 920 °C; t = 20 min; WQ |                       | 857 ± 12                                  | 564 ± 14                                  | 11.0 ± 1.5                               | 3.4 ± 0.8                |
| A3.4: T = 920 °C; t = 30 min; WQ |                       | 848 ± 14                                  | 565 ± 13                                  | 5.0 ± 1.0                                | 2.4 ± 0.7                |
| A3.5: T = 920 °C; t = 60 min; WQ |                       | 839 ± 11                                  | 559 ± 12                                  | 4.5 ± 1.0                                | 2.2 ± 0.8                |
| A3.6: T = 920 °C; t = 90 min; WQ |                       | 832 ± 13                                  | 556 ± 13                                  | 3.0 ± 1.0                                | 2.3 ± 0.5                |
and, close to 11.1%, for γ2-phase, being noticed the lowest fraction of precipitated σ-phase and the highest fraction of precipitated γ2-phase when comparing with the A1 and A2 states.

Considering that the applied ageing treatments induce changes in the weight fraction of all recorded phases, one can expect a high influence on the exhibited mechanical properties, mainly due to the precipitation of σ-phase. Fig. 4 shows the variation of mechanical properties as a function of applied temperature and treatment duration, for ultimate tensile strength σUTS (Fig. 4.a); 0.2 yield strength σ0.2 (Fig. 4.b); elongation to fracture εf (Fig. 4.c) and absorbed energy E (Fig. 4.d). Analysing the evolution of ultimate tensile strength σUTS (Fig. 4.a), one can notice a continuous increase from 816 MPa - for initial ST state, to 825 MPa for 780 °C and 90 min treatment duration and, to 840 MPa for 850 °C and 90 min treatment duration. In the case of the ageing treatment performed at 920 °C, a different behaviour is recorded, an increase is obtained up to 10 min treatment duration when a value of 858 MPa is recorded, followed by a decrease to 832 MPa for a treatment duration of 90 min. Analysing the case of 0.2 yield strength σ0.2 (Fig. 4.b) one can notice, for all ageing treatment temperatures, a slow increase, from 527 MPa - for initial ST state, to 535 MPa for 780 °C, to 543 MPa for 850 °C and, respectively, to 556 MPa for 920 °C. In the case of the elongation to fracture εf variation (Fig. 4.c), one can notice a continuous decrease with the increase of applied ageing treatment temperature and treatment duration, from 60.5% - for initial ST state, to 4.1% for 780 °C, to 1.1% for 850 °C and, respectively, to 3% for 920 °C. One can notice that the sharpest decrease in elongation to fracture is recorded in the case of ageing treatments performed at temperatures above 850 °C. Analysing the case of the absorbed energy E (Fig. 4.d) one can notice, also, the same behaviour with the ones recorded in the case of elongation to fracture. For all ageing treatment temperatures, a decrease is recorded, from 142.2 J - for the initial ST state, to 2.4 J for 780 °C, to 1.5 J for 850 °C and, respectively, to 2.3 J for 920 °C. Also, the sharpest decrease in absorbed energy is recorded in the case of ageing treatments performed at temperatures above 850 °C.

In order to explain the evolution of recorded properties (mechanical behaviour), one must consider the evolution of weight-fraction for all phases, due to the law of mixture, which states that a higher weight fraction of a certain phase induces a higher influence on the overall material’s exhibited properties. In the case of SDSS alloys, it was proved that the d-phase is responsible for strength properties while the γ-phase is responsible for plasticity/ductility [5–9]. The precipitation of secondary phases, such as σ-phase, inducing worsening effects on all exhibited mechanical properties [21–23,28–30]. Considering the recorded average weight-fraction of precipitated σ-phase, one can assess its influence on exhibited mechanical properties. The increase in weight-fraction of σ-phase shows a minor influence on strength proper-
ties, both ultimate tensile strength ($\sigma_{UTS}$) and 0.2 yield strength ($\sigma_{0.2}$) showing small increases in comparison with the initial ST state, and a major influence on ductility/plasticity properties, both elongation to fracture ($\epsilon_f$) and absorbed energy ($E$) showing high decreases in comparison with the initial ST state.

Considering that the highest weight fraction of $\sigma$-phase is recorded in the case of ageing treatments performed at 850 °C (A2 sates), the analysis of chemical composition variation during ageing treatment, for all phases, can be performed considering this case.

Fig. 5 shows the variation of the main alloying elements: Cr (Fig. 5.a), Ni (Fig. 5.b), Mo (Fig. 5.c) and Si (Fig. 5.d), in the case of $\gamma$-phase, $\delta$-phase, $\sigma$-phase and $\gamma_2$-phase, for ageing treated at 850 °C (A2 samples. Analysing the Cr variation (Fig. 5.a) one can notice, in the case of $\gamma$-phase, a constant content during treatment, close to 25.2 wt%. In the case of $\delta$-phase, one can notice a continuous decrease of Cr content, from approx. 28.9 wt% (for initial ST state) to approx. 25.6 wt% (for 90 min treatment duration). A sharper decrease in Cr content is observed for treatment durations exceeding 20 min. Analysing the case of $\gamma_2$-phase reveals, also, that the content of Cr is decreasing with increasing treatment duration, from 28.1 wt% (for 5 min treatment duration) to 25.2 wt% (for 90 min treatment duration). The decrease recorded in the case of $\delta$-phase and $\gamma_2$-phase, is compensated by the increased content recorded in the case of $\sigma$-phase, from 29.8 wt% (for 5 min treatment duration) to 31.5 wt% (for 90 min treatment duration). Analysing the Ni variation (Fig. 5.b), one can notice that the $\gamma$-phase shows, also, an almost constant content during ageing treatment, close to 8 wt%. In the case of $\delta$-phase, one can notice a slow decrease in Ni content, from approx. 4.9 wt% (for initial ST state) to approx. 4.4 wt% (for 90 min treatment duration). Also, a slow decrease in Ni content is observed in the $\sigma$-phase case, from 4.9 wt% (for 5 min treatment duration) to 4.0 wt% (for 90 min treatment duration). The decrease recorded in the case of $\delta$-phase and $\sigma$-phase is compensated by the increased content recorded in the case of $\gamma_2$-phase, from 4.5 wt% (for 5 min treatment duration) to 6.5 wt% (for 90 min treatment duration). The Mo variation (Fig. 5.c) shows, also, that the $\gamma$-phase possess constant content during treatment, close to 2.5 wt%. In the case of $\delta$-phase, one can notice a continuous decrease in Mo content, from approx. 3.9 wt% (for initial ST state and, also, for 5 min, 10 min and 20 min treatment durations) to approx. 1.6 wt% (for 90 min treatment duration). Also, a decrease in Mo content is observed in the $\gamma_2$-phase case, from approx. 3.3 wt% (for 5 min treatment duration) to approx. 2.5 wt% (for 90 min treatment duration). The decrease recorded in the case of $\delta$-phase and $\gamma_2$-phase, is compensated by the increased content recorded in the case of $\sigma$-phase, which increases from approx. 5.1 wt% (for 5 min treatment duration) to 5.5 wt% (for 90 min treatment duration). Analysing the Si variation (Fig. 5.d), one can notice a small decrease in the case of $\delta$-phase, from approx. 0.43 wt% (for 5 min treatment duration) to approx. 0.36 wt% (for 90 min treatment duration) and, $\gamma_2$-phase, from approx. 0.41 wt% (for 5 min treatment duration) to approx. 0.38 wt% (for 90 min treatment duration), compensated by a small increase in the case $\sigma$-phase, from approx. 0.46 wt% (for 5 min treatment duration) to approx. 0.48 wt% (for 90 min treatment duration). The $\gamma$-phase possess constant content during treatment, close to 0.38 wt%.

Overall, one can say the following. Firstly, $\gamma$-phase chemistry is not influenced by the applied ageing treatments, remaining almost constant during processing. Secondly, the precipitation of secondary phases induces depletion of parent $\delta$-phase in all alloying elements (Cr, Ni, Mo and Si). Thirdly, the chemistry of precipitated phases changes during processing, and that increasing treatment duration leads, mainly, to enriching in Cr and Mo and depletion in Ni – for $\sigma$-phase, and enriching in Ni and depletion in Cr and Mo – for $\gamma_2$-phase. Fourthly, if treatment duration is long enough, the $\gamma_2$-phase composition tends to reach the same composition with the ones of the primary $\gamma$-phase.

Considering, again, the case of ageing treatments performed at 850 °C (A2 sates) as the most relevant case, due to its highest recorded weight fraction of $\sigma$-phase, the fracture surfaces analysis
can be performed considering this case. The fracture surfaces analysis considered only Charpy tested specimens. In the following section, only the cases were noticeable differences occurred in fracture surfaces morphologies when comparing to the initial ST state (Fig. 6.a) are presented, namely the case of 10 min – A2.2 state (Fig. 6.b), 20 min – A2.3 state (Fig. 6.c) and 90 min – A2.6 state (Fig. 6.d).

The ST processed state (Fig. 6.a) shows a ductile behaviour, the fracture occurred after large plastic deformations (total elongation to fracture, close to 60.5 ± 3.5%) and high absorbed energies during impact testing (close to 142.2 ± 4.2 J). As observed, the fracture surfaces show fibrous morphologies, large voids/dimples and areas with signs of voids coalescence, all indicating a pronounced ductility. Also, one can observe the presence of fissures/crevices.

By analysing the fracture surfaces obtained after Charpy testing of 10 min ageing treated specimens – A2.2 state (Fig. 6.b), one can observe that the fracture surfaces still show signs of fibrous morphology but with a decreased voids/dimples density and smaller-size voids/dimples in comparison with the ST sate, indicating a decreased ductility. This observation is confirming obtained results related to ductility (elongation to fracture, close to 60.5 ± 3.5%) and high absorbed energies during impact testing (close to 142.2 ± 4.2 J). As observed, the fracture surfaces show fibrous morphologies, large voids/dimples and areas with signs of voids coalescence, all indicating a pronounced ductility. Also, one can observe the presence of fissures/crevices.

Conclusions

In this work, the influence of ageing treatment temperature and duration on γ-phase precipitation and mechanical properties of UNS S32750 SDSS alloy has been studied under strict TM processing conditions. Experimental data showed that at low temperatures, i.e. 780 °C, in order to induce the precipitation of γ-phase, the minimum incubation time is situated close to 20 min, and that increasing treatment temperature decreases the minimum incubation time, with at 850 °C the γ-phase being firstly detected at 5 min.

The γ-phase precipitation shows the highest precipitation kinetics at 850 °C, when the maximum weight-fraction is obtained for each treatment duration when compared with ageing treatments performed at 780 °C and 920 °C. Also, it was proved that the weight-fraction of γ-phase shows an important influence on ductility properties (elongation to fracture and absorbed energy) in comparison with strength properties (ultimate tensile strength and 0.2 yield strength), where a small increase in the weight-fraction of γ-phase induced a high decrease in the elongation to fracture and absorbed energy and a small increase in ultimate ten-
sile strength and 0.2 yield strength. The analysis of $\gamma_2$-phase showed that the $\gamma_2$-phase possess a continuous increase in kinetics with a rise in temperature, with maximum $\gamma_2$-phase weight-fractions being recorded in the case of the ageing treatment performed at 920 °C, in comparison with ageing treatments performed at 780 °C and 850 °C. The analysis of occurred changes in the chemical composition of all observed precipitated phases proved that increasing treatment duration leads to important changes in the chemical composition, to an enriching in Cr and Mo and a depletion in Ni in the case of $\sigma$-phase and to enriching in Ni and depletion in Cr and Mo in the case of $\gamma_2$-phase.

Overall, all presented data brings valuable insight into the $\sigma$-phase precipitation phenomena and its influence on UNS S32750 SDSS alloy’s exhibited mechanical properties and, also, can provide researchers and industrial steel processors a guide regarding the selection of optimal ageing treatment parameters (treatment temperature and treatment duration) in order to avoid/minimise the embrittlement induced by the precipitation of deleterious $\sigma$-phase.

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**Compliance with ethics requirements**

This article does not contain any studies with human or animal subjects.

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**Fig. 6.** Typical SEM images of fracture surfaces obtained after Charpy testing for ST (a) and ageing treated at 850 °C (A2) for 10 min – A2.2 (b), 20 min – A2.3 (c) and 90 min – A2.6 samples (d).
