Ageing Behavior and Restoration by Heat Treatments of 2205 Duplex Stainless Steel

N. Ouali[1], B. Belkessa[1] , M. Bouabdallah[2]

[1] Welding and NDT Research Center (CSC).
B.P 64, Cheraga, Algiers.

[2] Department of Metallurgy, National polytechnic school of Algiers.

Abstract:

Duplex stainless steels (DSS) are prone to intermetallic phase formation at high temperatures, which can drastically decrease the toughness property. This work investigates the microstructural evolution of σ phase in a SAF 2205 DSS after subsequent isothermal aging and annealing treatments and there influences on mechanical properties. We submit specimens to impact testing and hardness measurements before and after ageing and restore treatments. The results showed that considerable changes in term of precipitation of intermetallic particles were observed in microstructure. The formation of these intermetallic phases such as σ was recognized as the major reason for the observed changes. However aging at 850°C led to a significant decrease in toughness and slight increase in hardness and the high temperature treatment at 1080°C dissolves all secondary phases.

Key words: Duplex stainless steel, Ageing, Restore, Toughness, Sigma phase.

1. Introduction:

Duplex stainless steels are being widely used for a variety of applications such as oil and gas industries, chemical and power plants. The excellent combination of mechanical properties and corrosion resistance is the main reason for their increasing application in various industries. DSS have higher strength than austenitic grades as well as higher toughness than ferritic ones. In addition, high resistance to stress corrosion cracking, hydrogen embrittlement and intergranular corrosion are among their excellent properties [1]. Depending on chemical composition and applied thermomechanical processing, the microstructure of DSS mostly consists of ferrite and austenite which led to have optimized mechanical properties and corrosion resistance. On the other hand, the appropriate treatments are necessary to avoid formation of undesirable intermetallic phases such as σ and χ. Use of duplex stainless steels has to be restricted, especially in the temperature range over 500°C [2,3].
Therefore, the formation of dangerous phases and their influence on the toughness of duplex stainless steels have been extensively studied. Some authors suggest that the precipitation of σ phase is associated with the precipitation of χ particles and only few authors detect the carbides at the first time [6]. However, it is well known that the kinetics of intermetallic precipitation is markedly affected both by the plastic deformation and by the temperature of the solution treatment. Therefore, the critical time for σ, and other phase formation, seems to be dependent both on the solution treatment temperature and on the cooling rate of the subsequent quenching [6.7].
The purpose of the present work was therefore to find out the effect of ageing and restore treatment on microstructure evolution and mechanical properties of DSS grade 2205.

Particular attention was paid to establish a correlation between microstructure and toughness property of the investigated steel. In first time, the specimens were solution-treated at temperatures of 1080 °C and 1120 °C which were chosen, based on isotherms of Fe-Cr-Ni systems, then isothermal ageing was carried out at 850 °C for 30, 60, 90 and 120 min. Finally a solution treatment at 1080°C was applied to restore the δ/γ phases balance and to refine the structure of 2205 duplex stainless steel.

2. Experimental work:

The DSS used in this investigation was SAF 2205 alloy having a stable microstructure consisting of about 45% ferrite and 55% austenite at ambient temperature, with the composition mentioned in Table 1. It was received as tubes of 168mm diameter and 3mm thick from an industrial hot rolling line. All the specimens were submitted to solution annealing at 1080 and 1120°C for 60 min in order to achieve homogeneity Fig.1. Next isothermal heat treatment was performed at 850 °C for 30, 60, 90 and 120 min, followed by cooling in water see Fig.2 for all treatments performed.
To characterize the microstructures, samples were subjected to the standard grinding and polishing techniques before etching. The standard charpy specimens 12mm 3mm 55mm, were machined along the longitudinal rolling direction of the as received plate. The impact testing was carried out at the ambient temperature using a universal impact test machine with maximum capacity of 360 *J*±1 (*J*). A Vickers hardness tester was used to measure the Vickers hardness under 20 Kgf load. For optical microscopy investigations, the heat treated samples were prepared commonly and etched in. For quantitative metallographic studies, samples were electrolitically etched in appropriate solution of and the phase rate was also performed using image-analysis technique by Atlas soft imaging system.

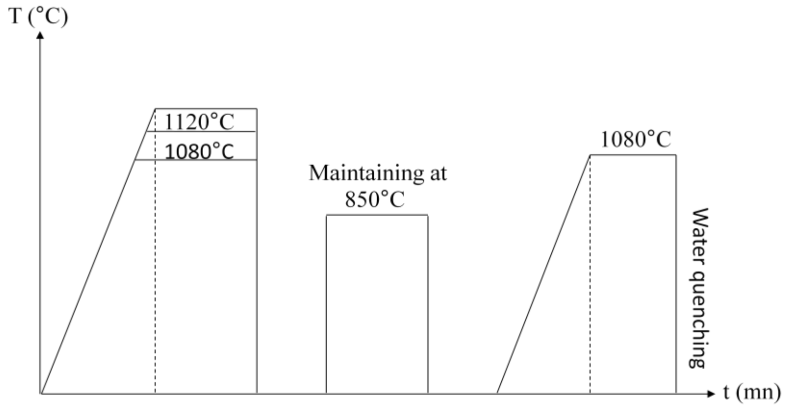


Fig.1. Diagram sequences of performed heat treatments.

Table 1: Composition of the studied steel (wt pct).

Elements	C	Si	Mn	Ni	Mo	Cr	P	S	Cu	N
%	0.03	0.36	1.77	5.70	2.258	22.05	0.018	0.015	0.2	0.14

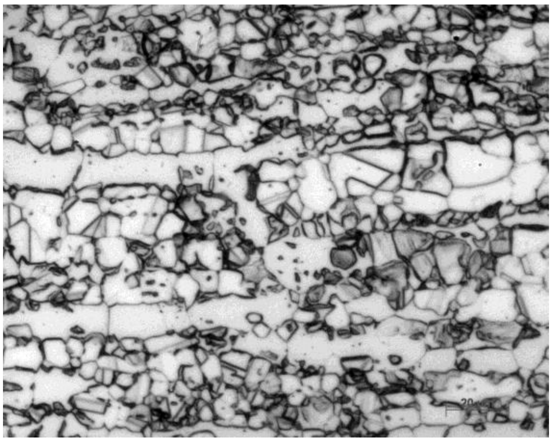


Fig.2: Microstructure of sample in the as received condition, consisting of austenite (light etched) and ferrite (dark etched).

As it is clear, in this case Fig.2, the structure mainly consists of austenite (γ) within a matrix of ferrite (δ). Fig. 3 shows the micrograph of the steel samples as-solution treated at 1080 °C and 1120°C followed by water quenching. No secondary phases were detected. It reveals a predominantly isotropic structure of ferrite and austenite grains displayed on transverse section.

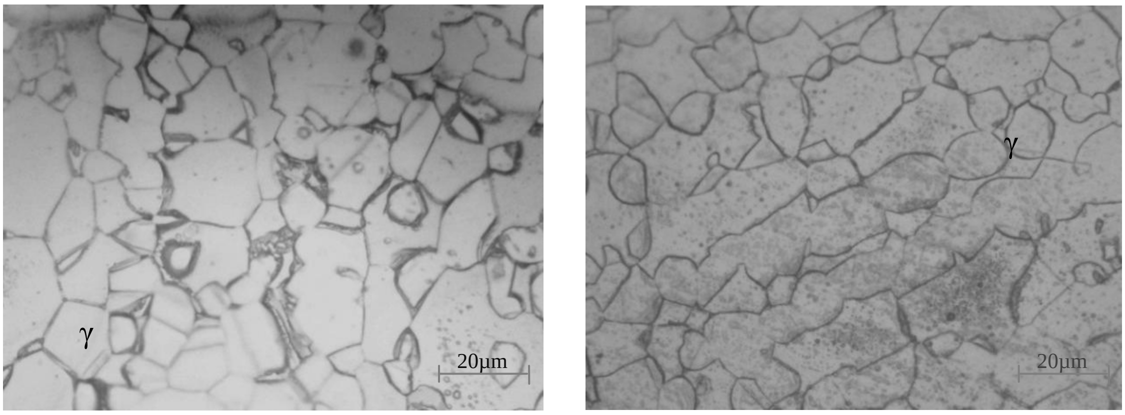


Fig.3: Optical micrographs of the specimens aged at 850 °C after being solution-treated at 1080 °C and 1120 °C.

3. Results and Discussion:

3.1 Toughness and hardness

Fig.4 shows the dependence of absorbed energy before fracture on aging time. As it is clearly illustrated, for a given annealing treatment, the absorbed energy decreases gradually with aging time. At 850°C a drastic drop in toughness occurs during all aging time. This abrupt decrease in toughness is likely pertaining to the formation of σ phase (Fig.7), which is known for its harmful effect on toughness and ductility. Not the same trend was observed when the hardness was considered *vs* aging time. The corresponding results are demonstrated in Fig.5.

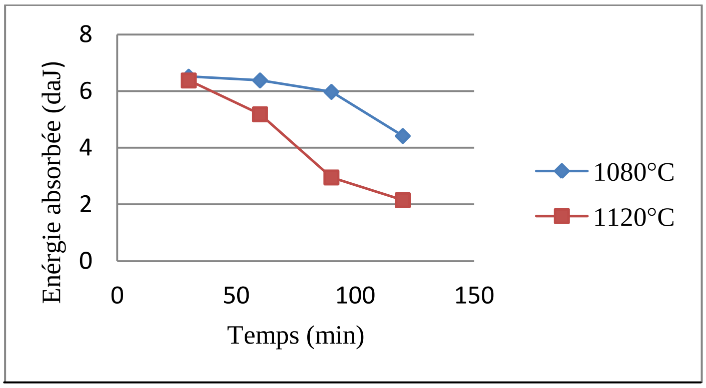


Fig. 4: Effect of aging times on absorbed energy of DSS 2205 at 850°C.

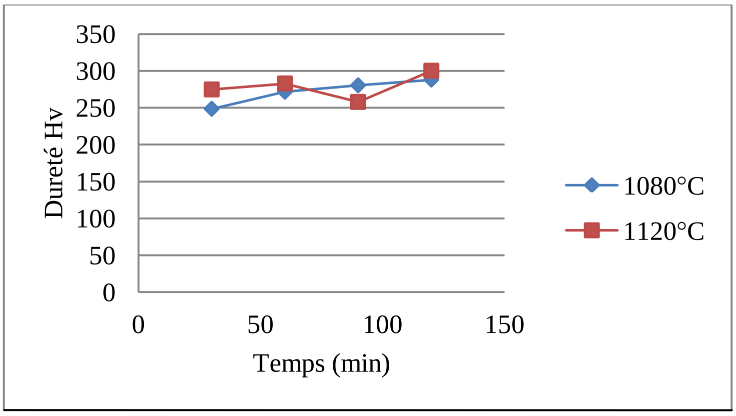


Fig. 5: Effect of aging times at 850°C on Vickers hardness.

As the aging temperature is increased, an increasing of austenite amount through the decomposition of ferrite should be also taken into account. The different strength of ferrite δ and γ leads to the difference in the impact behavior of these two phases. Consequently, the γ/δ interphases are the most prone area for the initiation of cracks. Therefore, for a ferritic-austenitic stainless steel, when the amount of γ is increased, not only the strength but the interfaces susceptible for grain boundary sliding and crack nucleation are also enhanced. This can readily result in decreasing of toughness or absorbed energy in impact testing. That is because during a rapid deformation such as the impact testing, the strain is concentrated mostly in the softer phase (δ) of duplex stainless steels rather than in the harder one (γ).

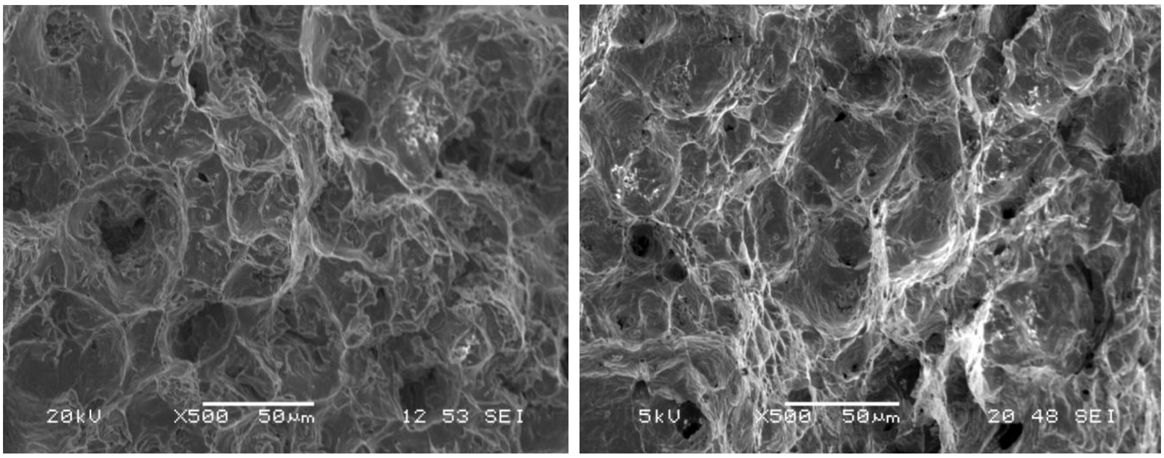
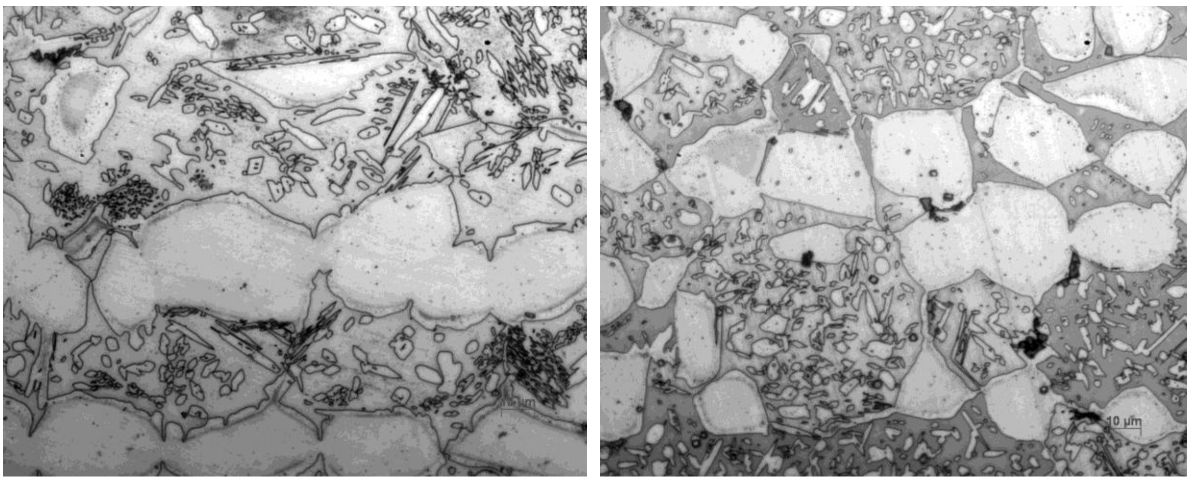


Fig.6: Rupture fractography of samples (1080 and 1120°C) after ageing at 850°C.

Fig.6 shows fractography of impact testing, which revel brittle fracture (cleavage). The microcavities show the high threshold of 850 ° C embrittlement. This brittleness is due to the presence of intermetallic phases such as sigma phase which is very hard and the most dominant phase, its field of plasticity is very small, so any stress causes stress concentration at the interfaces ferrite / sigma and austenite / sigma which are higher interfacial energy regions. Stress concentration causes the birth of microcavities, the interconnection of these microcavities is the origin of the crack causing the rupture of the material.

3.2 Microstructural evolution during aging

Fig.7 represents the optical micrographs of the specimens aged at 850°C for different time. As it is illustrated σ phase has formed along the δ/γ interfaces and grown into the adjacent ferrite region**,** it is well known that δ/γ interfaces can act as preferential nucleation sites for precipitation of σ phase [14]. As it is indicated by arrows, the precipitation of σ particles was observed even at the shortest aging time, i.e. 30 min. Since the formation mechanism of secondary phases is diffusion, the size and volume fraction of σ phase were basically increased with the aging time.



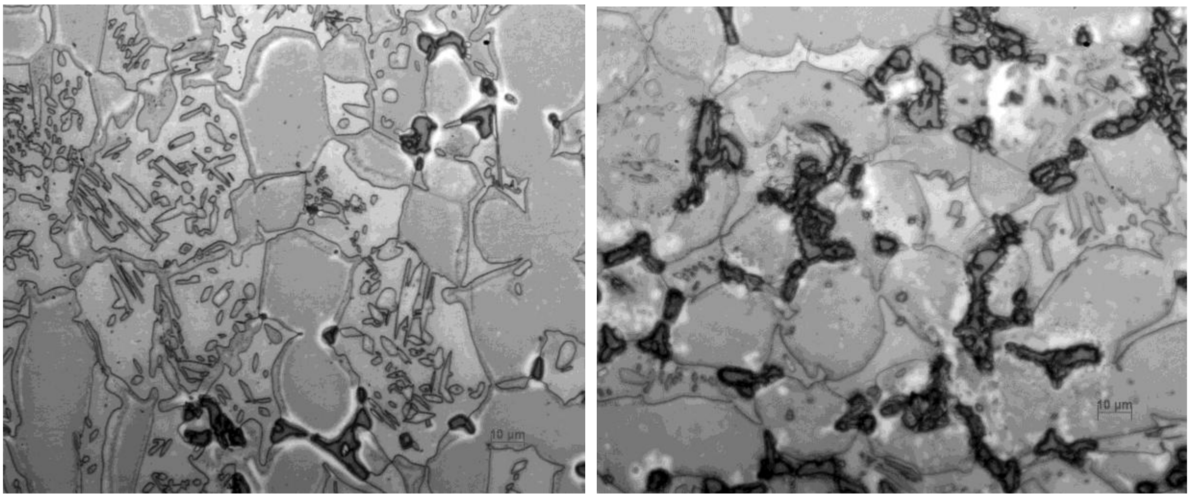


Fig.7: Optical micrographs obtained from DSS 2205 annealed at 1120°C and aged at 850°C for: (a) 30 min, (b) 60 min, (c) 90 min, (d) 120 min.

While the σ precipitates is normally coarser than the former. The precipitation of σ phase from ferrite takes place because the ferrite is rich in Cr and Mo. It is well established that σ is precipitated mainly through the eutectoid decomposition of $\delta \longrightarrow \sigma + \gamma^*$ (γ* refers to new-formed γ) [6,9]. As σ phase is rich in Cr and Mo [11], it is presumed that the ferrite region adjacent to the intermetallic phase could transform to the new austenite because of the depletion of these ferrite stabilizer.
However, prolonged aging time at this temperature resulted in averaging and coarsening of both σ phase and γ grains. The direct consequence of this is losing the coherency and reducing the pinning effect of σ precipitates. As a result, a coarse structure consisting of large σ particles as well as large γ grains are observed, as shown in Figs. 7(c) and (d). It is of significant importance that these changes in microstructure are in consistent with the changes in the toughness presented in Figs. 3 and 4, when coarsening the microstructure at high temperatures and prolonged aging resulted in a drop in toughness.

Microstructural evolution during restoration (Annealing treatment at 1080°C):

After aging treatments, all samples were annealed at 1080°C for 60 min and quenched in water; this treatment led to a complete dissolution of sigma phase and precipitates and thus restores the δ/γ balance of the duplex stainless steel.
Micrographs below Fig.8 show a new distribution of ferrite and austenite phases and a located refinement of structure. We also observe the presence of fine austenite grains within the bands of the δ-ferrite phase.

This new transformation follows the equation bellow:

Precipitates (carbures + sigma) $\longrightarrow$ ferrite δ + austenite γ

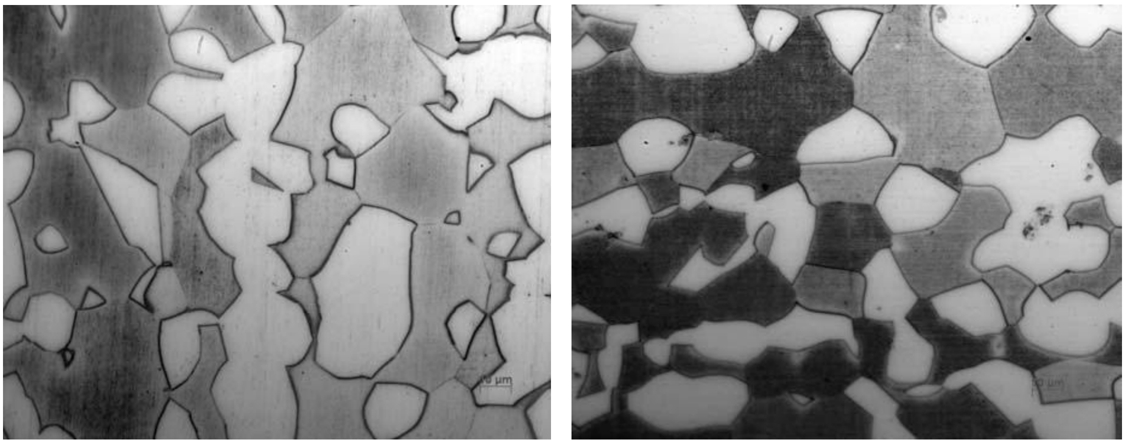


Fig.8: Optical micrographs obtained from DSS 2205 annealed at 1080°C for 60min. Respectively: (a) 1080/60-850/60. (b) 1080/60-850/120.

The solution treatment at 1080 ° C dissolved all intermetallic precipitates and restored the mechanical properties of the material for all samples regardless of the rate of the precipitates obtained after aging.

For temperatures studied, due to the dissolution of precipitates we can say that hardness has been slightly reduced but still acceptable after back into solution at 1080°C see Fig.10. So this property is not sensitive to low amounts of secondary phases. The hardness value has not decreased above 240 Hv which is the average value of the hardness in as received material.

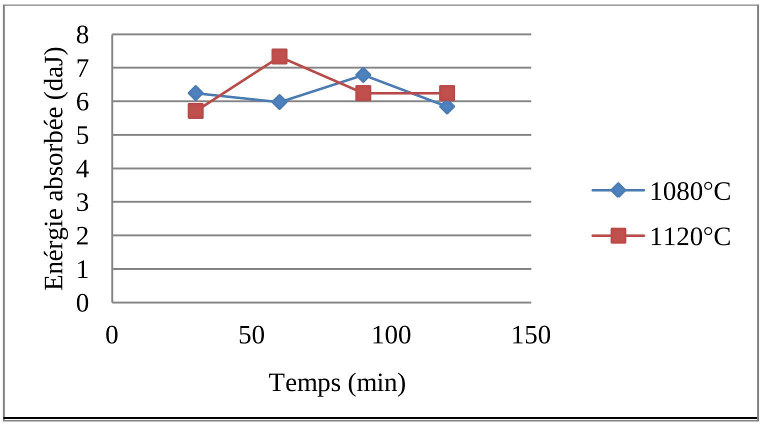


Fig.9: Evolution of the absorbed energy after restoring at 1080°C/60min.

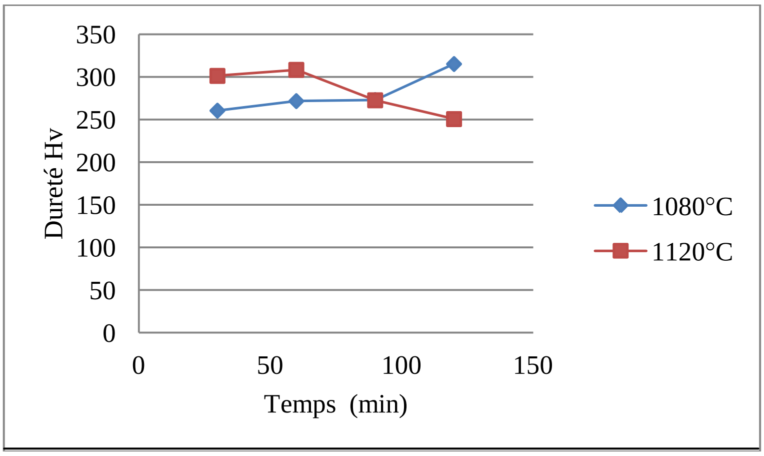


Fig.10: Hardness evolution after restoring at 1080°C/60min.

Fig.11 is a cupules fractographies; it is the character of ductile transgranular fracture accompanied by plastic deformation. Duplex stainless steels are composed of two phases, ferrite and austenite and the two phases have a wide range of plasticity.

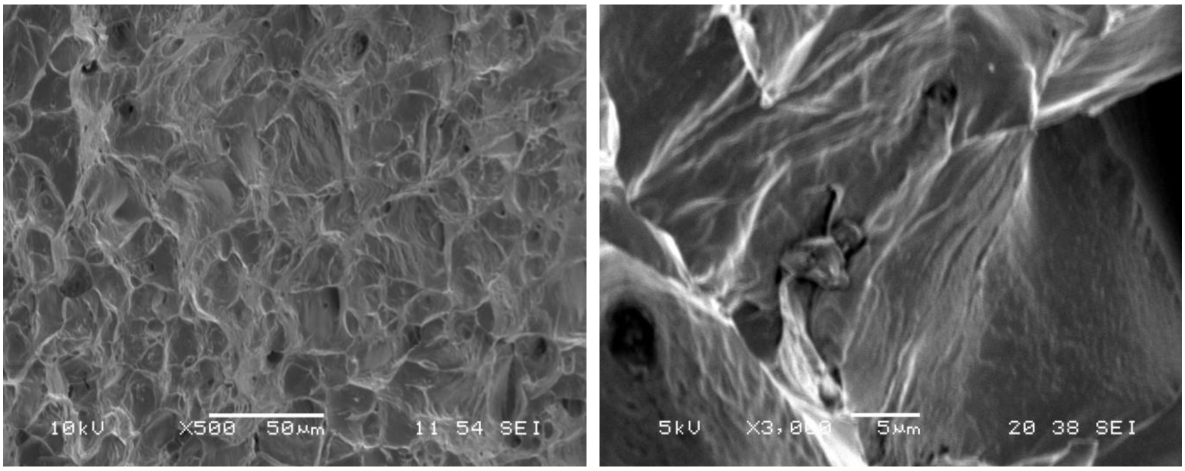


Fig.11: Rupture fractography of samples (1080 and 1120°C) after restoring at 1080°C.

T° d'hypertrempe	%δ	%γ	%δ/γ
1080°C	52.3	47.4	1.10
1120°C	43.4	56.6	0.76

Conclusion:

The effects of ageing and restore treatments on the microstructure and mechanical properties of SAF 2205 duplex stainless steel can be summarized in these points:

- The solution treatment at 1080°C and 1120°C of duplex stainless steel followed by water quenching, exhibited a coarse δ-ferrite structure with austenite.

- Microstructural evolution is extremely sensitive to sigma phase precipitation during ageing treatment.

- Absorbed energy to fracture was decreasing by aging temperature.

- The hardness pertaining to the aged specimens indicates slight increase with aging time at 850°C.

- The intermetallic sigma phase formed during aging was observed at the interface of boundaries after 30 min. It was clearly observed that the formation of this phase is the main reason for drop of impact toughness and increment of hardness.

- The volume fraction and particle size of σ increased with aging time.

- Sigma phase was completely dissolved by heat treatment at 1080 °C.

References:

1. Gunn, R. N. Duplex Stainless Steels. Microstructure, Properties And Applications. Abington Publishing, Cambridge England, (2003).
2. Zh.L. Jiang, X.Y. Chen, H. Huang, Xy. Liu. Grain Refinement Of Cr25ni5mo1.5 Duplex Stainless Steel by Heat Treatment. 2003.
3. R. Badji, M. Bouabdallah, B. Bacroix, C. Kahloun, B. Belkessa. Phase transformation and mechanical behaviour in annealed 2205 duplex stainless steel. Materials characterisation, 59 (2008) 447-453.
4. Chen T.H., Weng, K.L. And Yang, J.R., “The Effect Of High Temperature Exposure On The Microstructural Stability And Toughness Property In A 2205 Duplex Stainless Steel”, Materials Science And Engineering, A 338, (2002). 259 – 270.
5. Recent developments in stainless steels. H. Lo, C.H. Shek, J.K.L. Lai. Materials Science and Engineering R 65 (2009) 39–104.
6. Influence of isothermal aging on secondary phases precipitation and toughness of a duplex stainless steel SAF 2205. I. Calliari Æ M. Zanesco Æ E. Ramous.
7. High temperature ageing behavior of a duplex stainless steel. S.K. Ghosh, S. Mondal. Materials Characterization 59 (2008) 1776–1783.
8. Henrik Sieurin.Fracture Toughness Properties of Duplex Stainless Steels. Doctoral Thesis.
9. Joanna Michalska And Maria Soza´Nska. Qualitative and Quantitative Analysis of Σ and Χ Phases in 2205 Duplex Stainless Steel. Department Of Materials Science, Silesian University Technology, Katowice, Poland.
10. Martin, J. L. Et George, A. Caractérisation Expérimentale Des Matériaux II. Traité Des Matériaux, (1998).
11. I. Zucato, M. C. Moreira, I. F. Machado, S. M. Giampietri, Lebao. Microstructural Characterization and the Effect of Phase Transformations on Toughness Of The UNS S31803 Duplex Stainless Steel Aged Treated At 850 °C. Materials Research, Vol.5.