INVESTIGATION OF SECONDARY PHASES EFFECT ON 2205 DSS FRACTURE TOUGHNESS

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It is well known that the fracture toughness of DSS is strongly reduced by the precipitation of various intermetallic phases occurring in the temperature range 600-1000°C. A large decrease in impact fracture toughness occurs even at room temperature for volume fractions of intermetallic phases lower than 1%, when only small and rare particles are present.
In the present investigation, the influence of the intermetallic phases on the impact fracture behaviour of a 2205 grade DSS has been investigated. Samples containing different amounts of the intermetallic phases have been obtained by isothermal aging treatments in the range 800-950°C. The results of the impact tests confirm that the dangerous phase content determine both the toughness and the fracture behaviour of the DSS examined. At content lower than 1%, when precipitates are rare and small, their effect is a reduction of the absorbed energy for the ductile fracture. But the 1% appears as the critical content, when some particles became large enough to operate the nucleation of the brittle fracture. Indeed, at higher content, a number of large particles are present, well sufficient to induce a general brittle fracture. The obtained results allow correlating the absorbed energy values with the intermetallic phases content and dimensions.

KEYWORDS: duplex stainless steels, intermetallics, fracture

INTRODUCTION

Duplex stainless steels (DSS) have an austenitic-ferritic microstructure that gives them a very good combination of mechanical and corrosion properties, at a competitive cost. A typical property of DSS is the high pitting resistance that makes them suitable for structural applications in very aggressive environments. However the use of DSS is limited by their susceptibility to the formation of dangerous intermetallic phases, such as σ-phase and χ-phase, resulting in detrimental effects on impact toughness and corrosion resistance [1-12]. Therefore, many standards, relating to manufacturing of DSS, require "no intermetallic phases" in the microstructure [13].
To this end, DSS are submitted to a solution treatment, followed by water quenching, if and where it is possible. The aim of this treatment is not only to redissolve the dangerous phases and to avoid their precipitation in the 600-900 °C temperature range, but also to restore the ferrite/austenite ratio to approximately equal amounts, corresponding to the best mechanical and corrosion properties for the DSS.

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In a previous paper [14], the formation of secondary phases in the most popular DSS steel, the 2205 grade, has been examined, during both continuous cooling and isothermal treatments, to compare and define times and sequences of precipitation in the two different conditions. Our results indicate that the sequence of precipitation during continuous cooling can be different from that obtained by isothermal ageing. In the latter the χ-phase is always the first precipitating phase, as metastable "precursor" of the stable σ-phase. On the contrary, in the continuous cooling the same sequence occurs only at lower cooling rates, but at the highest cooling rate the χ-phase formation is no longer possible and the σ remain as the first and only precipitating phase.
Generally, the deterioration of the toughness of DSS is attributed to sigma phase, but this statement would not seem to be always correct. It is true that if the sigma phase is present, the toughness is lowered. However some results [13] indicate that the toughness of 2205 steel is already lowered before significant sigma content appears. The main drop in toughness occurs at the early stages of precipitation, also when the only phase detected are small and rare χ-phase particles. Surely, the σ is a dangerous phase for the toughness, but it does not seem to be the only phase which determines the embrittlement of the DDS steels, especially at very low intermetallic phase's content, when the σ is still virtually absent.
This conclusion agrees with Nilsson [2] who underlined the role of the χ-phase in the drop of toughness and with Gunn [4], who

C	Si	Mn	Cr	Ni	Mo	P	S	N
0.030	0.56	1.46	22.75	5.04	3.19	0.025	0.002	0.16

Tab. 1 ***Chemical composition of DSS 2205 (wt-%).***
Composizione chimica del 2205 DSS (% ponderale).

observed that the drop could occur also before any intermetallic phase could be detected by ordinary metallographic techniques. Nevertheless, it is not easy to understand and to justify how an extremely low intermetallic phase content, about 0, 5 %, that is few and very small intermetallic particles, could induce such an abrupt embrittlement even at room temperature Therefore it seems interesting to study in more detail the drop of the toughness of the DSS deriving from the first stages of precipitation of intermetallic phases.

EXPERIMENTAL

The as received material was a wrought SAF 2205 DSS rod (30mm), with chemical composition reported in Tab. 1. Isothermal ageing treatments of specimens, previously solubilised at 1050 °C for 30 minutes, were carried out in the temperature range 780-900 °C. Relatively short ageing times were chosen, on the basis of previous results [14-15], to produce low amounts of secondary phases and to investigate its influence on the impact toughness of the alloy.

Different phases have been identified by SEM-BSE examination of unetched samples. The ferrite appears slightly darker than austenite, while the secondary phases are lighter. Owing to the higher content of molybdenum, in combination with the large atomic scattering factor of molybdenum χ-phase appears in brighter contrast than σ-phase. The amount of secondary phases has been determined using image analysis software on SEM-BSE micrographs (10 fields, 1000x) [17]. The contribution of each phase to the total volume fractions was determined. The volume fractions of ferrite and austenite in a solution treated sample have been measured on 3 longitudinal and 3 transversal sections (20 fields for each section) by image analysis on light micrographs at 200x, after etching with the Beraha's reagent (R.T., 10s).

The impact fracture toughness of the materials under study has been investigated by means of instrumented impact testing. The tests were carried out at room temperature using Charpy-V notched specimens (10 x 10 x 55 mm) and using an available energy of 300 J (impact velocity of 5.52 m/s). The load deflection curves were partially smoothed using the moving averages method.

RESULTS AND DISCUSSION

Microstructure

In the SEM-BSE images of samples after isothermal aging ferrite and austenite appear in the background: with the ferrite darker than austenite. The secondary phases appear as small bright regions, with the χ-phase brighter than sigma.

Two examples of secondary phase's morphology and distribution, at the first stages of precipitation, are shown in the Fig. 1a and 1b. The morphology and localization of precipitating phases, χ and σ appears very similar.

As already reported [15], in all the range of the temperatures of isothermal aging considered, the first precipitating phase is the χ, generally decorating the grain boundaries. By increasing the holding time, the amount of χ increases and also the σ-phase appears, in the form of coarser precipitates at the γ/α boundary, but growing into the ferrite. Although σ particles are, at the beginning, less numerous than χ-phase particles, they are coarser, and grow more rapidly, quickly arriving almost to the same volume fraction. By increasing the holding time, σ grows to large particles, moving from the boundaries into the ferrite, embedding some small χ particles. This seems to show the progressive transformation of χ to σ, occurring mainly at 900°C. In the samples at lowest soaking times, we can observe that the preferential sites of formation of the first small particles of χ-phase are the triple points connections of γ/α boundaries.

The localization of the new phases at the ferrite/austenite boundaries is well known [1]. Generally the secondary phase's formation at grain boundaries and the growth into ferrite is justified by diffusion behaviour of the elements involved in the transformation: Mo diffusion coefficient is higher than Cr and Ni and is also higher in ferrite than in austenite.

a

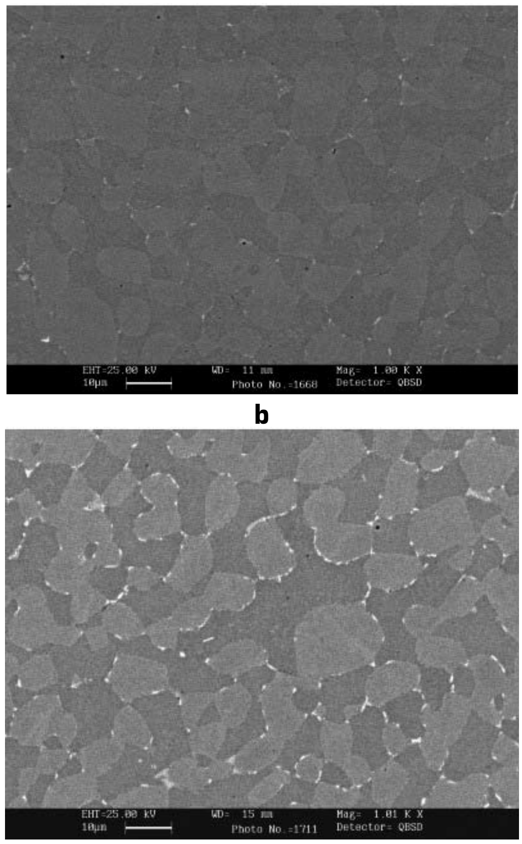

Fig. 1 ***SEM backscattered electron images, left: Sample 20'at 850°C with χ-phase; right: Sample 40' at 850°C, with σ and χ-phase.***
Immagine SEM con elettroni retrodiffusi, sinistra: campione trattato per 20' a 850°C con fase χ; destra: campione trattato 40' a 850°C.

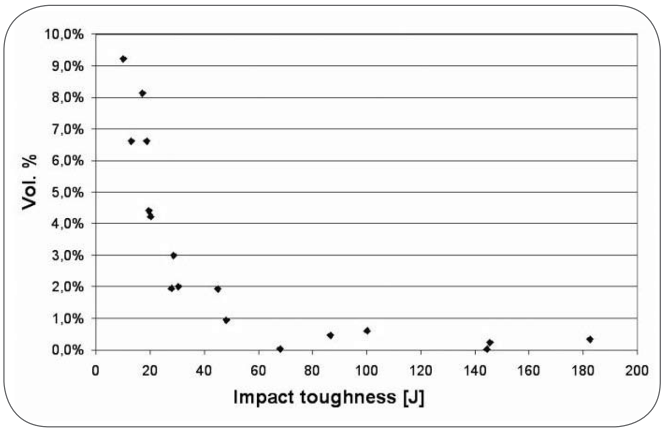

Fig. 2
Charpy toughness versus vol. % of secondary phases detected with SEM-BSE.
Tenacità a frattura ottenuta con prova Charpy in funzione della frazione volumetrica percentuale delle fasi secondarie quantificata al SEM-BSE.

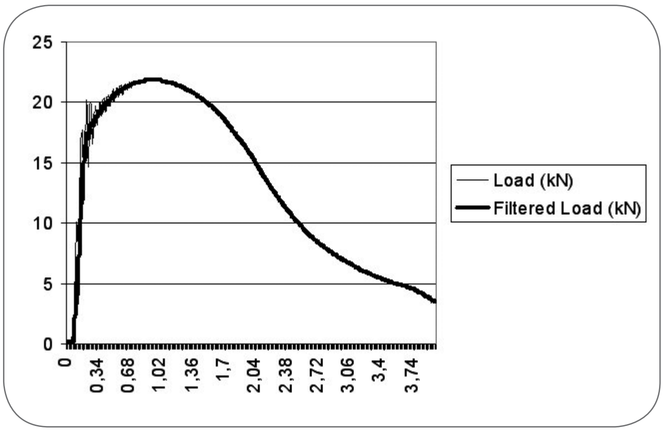

Fig. 3
Impact curve of the sample aged at 850°C, 15'.
Curva di impatto del campione trattato per 15' a 850°C.

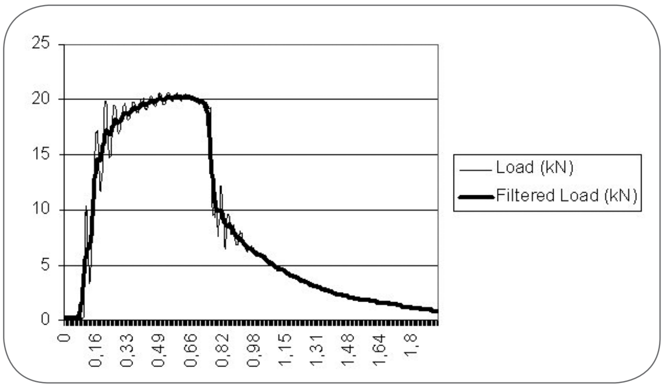

Fig. 4
Impact curve of the sample aged at 850°C, 30'
Curva di impatto del campione trattato per 30' a 850°C.

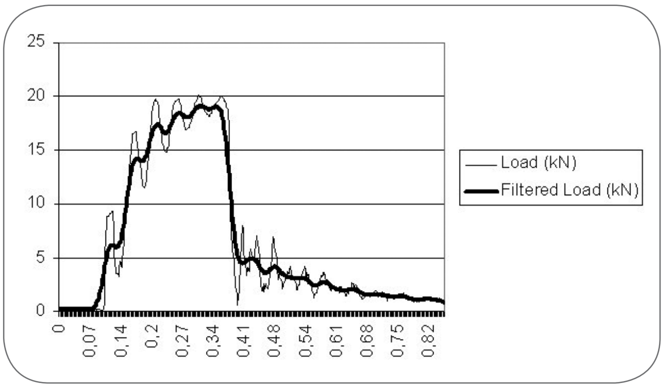

Fig. 5
Impact curve of the sample aged at 850°C, 40'
Curva di impatto del campione trattato per 40' a 850°C.

Impact fracture toughness

In Fig. 2 the impact energy of the materials under study is shown as a function of their content of intermetallic phases. The effect of the secondary phases is evident starting from 0.5% volume fractions, this value giving impact energy about 100 J. A drastic drop is evident at 1%, when the impact energy is about 50J, with a more severe deterioration of toughness induced by higher values (volume fractions > 1.5-2%). This statement agrees with the generally accepted specification for the DDS, asking for an intermetallic phase's content of less than 1%, or lower, to maintain the toughness value of 40-50 J. It is also evident the weak of correlation between impact toughness and volume fraction at the lowest secondary phase content. If the volume fraction is about 0, 5%, the toughness lies between 200 and 50 J: clearly the toughness do not depend on the volume fraction but probably on the morphology and distribution of the secondary phase's particles.

The analysis of the data in Fig. 2 and of the impact curves, allows us to subdivide the impact behaviour of the materials under study in three regions.

The first region pertains to the materials with impact energy higher than 150 J. The content of intermetallic phases in these materials is lower than 0.5% and the fracture behaviour is completely ductile. This is demonstrated by the impact curve shown in Fig. 3 , relevant to the material aged at 850°C for 15 ' (with a content of intermetallic phases not quantifiable, only small chi phase particles have been detected). It can be clearly noted that after general yielding (at about 15 kN), the applied load initially increases, because of strain hardening, and then continuously decreases, because of the ductile propagation of the crack nucleated at the maximum load. It can be therefore stated that the intermetallic phases simply act as nuclei for ductile damage.

The second region pertains to the materials with impact energy between 50 and 150 J, characterized by a content of intermetallic phases lower than 1.5%. A typical impact curve of the materials of this region is displayed in Fig. 4. It is relevant to the material aged at 850°C for 30' (with a content of 0.6% of intermetallic phases). In this case, after general yielding (at 15 kN), the materials undergo strain hardening and ductile crack nucleation (at the maximum load). After some stable propagation, however, brittle fracture takes place and there is an abrupt drop in the applied load. Brittle fracture can be clearly attributed to the breaking of a hard intermetallic phase and to the successive propagation of the crack in the neighbour ferrite phase.

The third region pertains to the materials with impact energy lower than 50 J, characterized by a content of intermetallic phases larger than 1.5%. In this case, fracture is completely brittle, as demonstrated, as an example, by the impact curve in Fig 5. It is relevant to the material aged at 850°C for 40' (with a content of

2.5% of intermetallic phases). After yielding at the notch root (at a load of about 15 kN), brittle fracture suddenly take place as long as the local plastic radius reaches a critical value.
In all the material, dynamic yielding takes place at a load of about 15 kN, showing that the amount of the intermetallic phases does not influence the yielding behaviour of the materials under study.
This allows estimating for the materials under study, using the Server [18] equation, the impact yield stress (σYd) and with the well-known Griffith relation, the critical brittle fracture stress, which depend by both the matrix properties and by the size of the embrittling particles.
From the analysis of the size distribution of the intermetallic particles, using above simple calculations, it is possible to evaluate that for contents of the intermetallic phases larger than 1% (with the coarsest particles lager than 1.1 μm), the critical conditions for room temperature brittle fracture at the notch root of the Charpy-V specimen can easily reached. On the contrary, if the content is lower than 1% (and the coarsest intermetallic particles smaller than 1.1 mm), brittle fracture cannot occur.
These evaluations agree and justify the effect of intermetallic phase's precipitation on the impact behaviour of the steel under examination.

CONCLUSIONS

The effects of isothermal treatments in the temperature range 780-900 °C on the microstructure and fracture toughness of SAF 2205 duplex stainless steel can be so summarized:
1 During the isothermal heat treatments, χ-phase is the first intermetallic phase to precipitate, always at the α/γ boundaries: σ-phase appears later on, and gradually substitutes χ-phase;
2 The effect of the secondary phases on DSS fracture toughness is evident starting from 0.5% volume fractions, this value giving impact energy about 100 J.
3 A drastic drop is evident at 1%, when the impact energy is about 50J, with a more severe deterioration of toughness induced by higher values (volume fractions > 1.5-2%).
4 It is also evident the weak of correlation between impact toughness and volume fraction at the lowest secondary phase content.
5 At volume fraction about 0, 5% the toughness lie between 200 and 50 J: clearly the toughness do not depend on the volume fraction but probably on the morphology and distribution of the secondary phases particles.
6 At contents of the intermetallic phases larger than 1% (with the coarsest particles lager than 1.1 μm), the critical conditions for room temperature brittle fracture at the notch root of the Charpy-V specimen can easily reached, while if the content is lower than 1% (and the coarsest intermetallic particles smaller than 1.1 mm), brittle fracture cannot occur.

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ABSTRACT

STUDIO DELL'EFFETTO DELLE FASI SECONDARIE SELLA TENACITÀ A FRATTURA IN UN 2205 DSS

Parole chiave: acciai inossidabili duplex, intermetallici,fratture

È ben noto che la tenacità a frattura degli acciai duplexduplex è fortemente ridotta dalla precipitazione di fasi intermetalliche che avviene nell'intervallo di temperatura compreso tra 600°C e 1000°C.
La frazione di volume critica di fasi intermetalliche che comporta il crollo della tenacità a frattura nella prova di impatto, anche a temperatura ambiente, è di 1%, sotto la quale i precipitati si presentano piccoli ed finemente dispersi. Nel presente studio è stata investigata l'influenza della precipitazione di fasi intermetalliche sulla tenacità a frattura nel 2205 DSS (tabella 1). Sono stati condotti trattamenti termici isotermi a temperature comprese tra i 800-950°C per ottenere campioni contenenti differenti quantitativi di fasi intermetalliche (Fig. 1). Il risultato delle prove di impatto ha confermato l'influenza sfavorevole delle fasi intermetalliche sulla tenacità e sul comportamento a frattura dell'acciaio esaminato (Fig. 2). Quando il contenuto di fasi intermetalliche è inferiore a 1% i precipitati sono piccoli e finemente dispersi e diminuiscono l'energia assorbita nel meccanismo di frattura duttile (Fig. 3). Mentre, se il contenuto supera il valore critico di 1%, i precipitati sono grandi abbastanza da agire come nucleanti di fratture fragili (Fig. 4). Quando il contenuto cresce ulteriormente, le particelle di fasi intermetalliche sono sufficientemente grandi e ravvicinate da indurre un meccanismo di frattura complessivamente fragile (Fig. 5). In accordo con questi risultati l'energia assorbita durante la frattura può essere correlata sia con il contenuto che con la dimensione delle fasi intermetalliche.