HOT DUCTILITY OF MICROALLOYED STEELS

M. Vedani, D. Ripamonti, A. Mannucci, D. Dellasega

The loss in ductility experienced by microalloyed steels at temperatures generally ranging from 700 to 1100°C is a widely studied subject in steel research. The hot ductility behaviour of steels is usually measured through the reduction of area of the samples after hot tensile tests performed up to fracture. The so-called standard hot ductility curves are then obtained by testing the steel at different temperatures. With the aim of achieving more precise information about the hot deformability behaviour of microalloyed steels, interrupted hot-tensile tests followed by rapid quenching (i.e. to "freeze" the microstructure) were carried out. Several steels were characterized by this method, followed by metallurgical investigations of the strained samples in order to identify the damage mechanisms and the precipitates affecting hot ductility.
The paper presents a summary of the results achieved about the effects of chemical composition on hot cracking, as affected by precipitation of secondary phases at austenite grain boundaries.

KEYWORDS: hot ductility, microalloyed steels, plastic deformation

INTRODUCTION

The loss in ductility at high temperature experienced by steels is a widely studied subject with large implications for industrial issues. For instance, transverse cracking occurring during continuous casting can occur when cracks are formed on the solidified surface of the steel due to ductility loss when the billets are straightened. A second field of concern for hot ductility refers to high-temperature plastic deformation of wrought products or of forging billets. Here, deformation in the critical temperature range may give rise to defects in sensitive steel grades even at moderate strain levels.
Hot ductility curves are generally produced by hot tensile testing, showing the reduction of area at fracture versus temperature. A typical curve is reported in Fig. 1. It shows three distinct regions, namely: the high ductility - low temperature region (region I), the ductility trough or embrittlement region (region II) and the high ductility - high temperature region (region III).
The fracture of specimens broken in the brittle temperature range is always intergranular and it can be caused by one or a combination of the following mechanisms. (i) Failure within a thin film of ferrite located at austenite grain boundaries since ferrite is softer than austenite at high temperatures. (ii) Formation of precipitate free zones (PFZ) adjacent to austenite grain boundaries, featuring a weaker resistance to deformation than the rest of the matrix structure. (iii) Grain boundary sliding governed by the limited

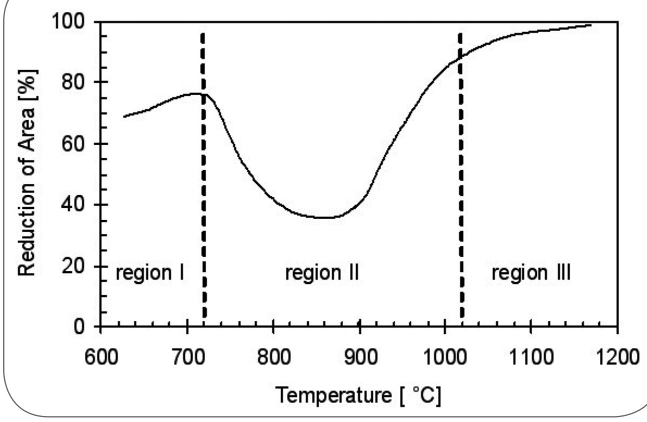

Fig. 1 **Typical hot ductility curve recorded for a steel showing the embrittlement region at an intermediate temperature range.**
Tipica curva di duttilità a caldo di un acciaio con evidenza del calo di duttiltà alle temperature intermedie.

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dynamic recovery of austenite and by the occurrence of workhardening build up at triple joints of grains.
At the high-temperature end of region II, an abrupt improvement in ductility occurs due to the onset of dynamic recrystallization. Any crack that has formed by intergranular failure in austenite is thus isolated as a consequence of grain boundary migration away from the crack. The isolated cracks can then grow only into large voids and failure ultimately takes place by void linking, at a much larger strain.
At the low-temperature end of region II, a relatively large fraction of ferrite is formed and the strain localization effect is suppressed. The highest recovery rate of ferrite at high temperature and the finer grain size than that of the austenite from which it has formed also contribute to improve steel ductility in the presence of a significant amount of ferrite.
Grain boundary precipitates are generally detrimental to ductility because they encourage void formation either at ferrite films or during grain boundary sliding. Precipitates are also directly responsible of the formation of the precipitate free zones adjacent to grain boundaries. Generally the finer the precipitates, the lower the ductility of the steel owing to a more effective pinning action of grain boundaries, allowing more time for crack nucleation and linking [1].
Compositions and precipitation temperatures for many of the possible secondary phases in microalloyed steels have been well defined in literature (see for instance: [1-4]) for many steel grades. However, care should be taken to the actual microstructural condition and thermal history of the steels when drawing comparisons from published works. Indeed, a large number of papers on hot ductility refers to as cast structures tested on cooling at various rates from the melting temperature or on reheating the cast structure from room temperature. Under these conditions, the coarse grain structure and the heavy segregation of alloying elements at grain boundaries strongly affects precipitation. Moreover, when studying wrought steels subjected to hot deformation, the combined effects given by the straining and by heating should not be neglected. The imparted strain mainly acts by accelerating the transformation kinetics, possibly lowering the precipitation temperatures.
In C-Mn-Al steels, precipitation of AlN phase can have a large influence on hot ductility. In cast steels, the formation of the AlN phase is very sluggish and hence the solubility product [Al]x[N] should exceed values of the order of $2{\cdot}10^{-4}$ for detrimental precipitation to occur during solidification and cooling. Precipitation of the AlN phase in the austenitic field of a plain carbon steel occurs at the fastest rate at about 1150°C, but even at this temperature the rate of precipitation is very slow. The onset of the $\gamma-\alpha$ phase transformation promotes a much faster precipitation kinetics due to more favourable solubility and diffusivity factors, especially for Al. Within this range, the time for the fastest precipitation of AlN becomes of the order of tenths of minutes at 800-850°C [2].
It is to note that AlN itself is not always directly responsible for steel embrittlement since many other residual elements (e.g. S, Cu, Sn, Sb) can segregate at austenite grain boundaries and reduce their cohesive strength at high temperatures. AlN can also act indirectly by pinning the grain boundaries, allowing more time for grain boundary segregation and encouraging intergranular cracks formed by other mechanisms to link up [1,2].
In C-Mn-Nb-Al steels, the formation of AlN interacts with the precipitation of the Nb(CN) phase, giving important implications. It was demonstrated that the temperature for maximum rate of Nb(CN) precipitation is 950°C while that for AlN precipitation is 815°C, so that the former phase would be expected to precipitate much before on cooling, being unaffected by the latter phase. However, it was also proposed that Al would slow down the diffusion of N atoms so that the formation of the Nb(C,N) phase would occur at lower temperatures and hence in a finer form. Experimental results are consistent in showing that the hot ductility in Nb-bearing steels is significantly deteriorated by Al owing to a finer precipitation of Nb(C,N) particles rather than by the formation of AlN precipitates [1].
Nb(C,N) precipitates are known to be some of the most detrimental phases for hot ductility. As for the other precipitates, when formed at austenite grain boundaries with a size of a few tenth of nanometers or less, they delay the onset of dynamic recrystallization and grain boundary mobility, that can otherwise isolate occasional cracks formed by other mechanisms. Concurrently, when grain boundary sliding is active as a process of plastic deformation, Nb(C,N) particles will encourage the development of cracks [1,4-8].
Titanium is added to structural microalloyed steels to improve toughness of welds, especially in the heat affected zone, by restricting grain growth. It is also recognized to have a great influence on hot ductility, with the possibility of improving or depleting it in cast steels, mainly depending on steel composition, cooling rate and testing temperature. A further important parameter for the definition of Ti effects is represented by the thermal history of the steel. In C-Mn-Al steels tested on cooling after solidification to simulate continuous casting operation, TiN has little opportunities to precipitate, especially if relatively high cooling rates are applied (larger than 25 K/min). On the contrary, investigations carried out on reheated samples showed that, on heating from room temperature, the TiN particles that are not completely re-dissolved are able to refine the grain structure and hence improve hot ductility. When this effect does not happen, adding Ti to C-Mn-Al steels generally causes the ductility to deteriorate because TiN precipitates can be finer and more detrimental than AlN particles [1,4,9,10].
In C-Mn-Nb-Al steels, either reheated or directly cast, small addition of Ti have been found to systematically improve hot ductility since the TiN particles can act as preferential nucleation sites for Nb precipitates. The Nb(C,N) phase is thus able to form at higher temperatures and becomes too coarse to deplete significantly hot ductility [1,4,5,6,10]. An additional effect that is often referred as of secondary importance is that the precipitation of TiN at high temperature depletes N from the austenite and hence reduces the volume fraction of the AlN particles.

MATERIALS AND EXPERIMENTAL PROCEDURES

Within the framework of a research programme focussed on hot ductility of several C-Mn and microalloyed steels, a few examples of experimental results will be selected in this paper with the aim of discussing general microstructural features affecting hot ductility of wrought steel grades.

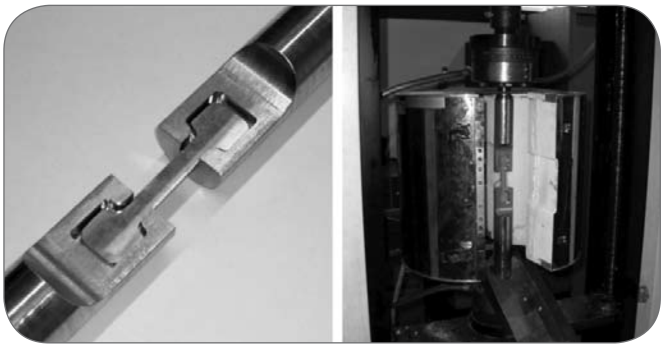

Fig. 2

Tensile specimen installed in the fixturing device (left) and experimental setup for the interrupted hot tensile straining tests (right).

Provino per trazione a caldo installato nel dispositivo di afferraggio (a sinistra) e vista generale dell'apparecchiatura per le prove di trazione a caldo interrotte (a destra).

Hot ductility of the steels was mainly investigated at two temperatures of 850 and 950°C, three plastic strain levels of 10%, 16,7% and 30% and two strain rate values of $2{,}8.10^{-3}s^{-1}$ and $6{,}7.10^{-4}s^{-1}$. Analysis of possible microstructural damage during plastic straining of the steels was investigated by performing interrupted hot tensile tests. For this purpose, an experimental setup was developed that allowed rapidly quenching the specimens right after straining so as to "freeze" the steel structure for further microstructural analyses. The fixturing device for the specimens and load train depicted in Fig. 2 were thus designed to pull in tension flat specimens having a typical gauge length of 50 mm and a rectangular section of 3x10 mm^2 in a tubular resistance furnace. After having reached the expected strain level, the specimens could be rapidly separated from the fixtures and quenched in a water tank positioned below the furnace within a couple of seconds. The described experimental device was installed in a universal testing machine, as also shown in Fig. 2.

The specimens strained at high temperature and quenched into cold water were then subjected to different types of analyses. The grain structure and the defects formed at high temperature were examined by optical and SEM microstructural analyses on metallographic samples longitudinally cut form the specimen gauge length. The samples were polished by standard techniques and chemically etched for 30-60 seconds in a solution of 8 g of $K_2S_2O_5$ in 100 ml of H_2O.

Fractographic analyses were also carried out on other hot-strained specimens by first fracturing them by an impact at the liquid nitrogen temperature. By this method, the brittle cleavage fracture produced at low temperature could be easily distinguished from the pre-existing damaged regions formed during high-temperature straining. In particular, fractographic analyses performed on the specimens, allowed to analyse the morphology of the clean, non oxidized facets of the high-temperature cracks and the possible precipitates located on their surfaces. For this purpose careful measurements of precipitate composition were performed by EDS microprobe systems linked to both a conventional SEM and a Field Emission SEM. This latter was especially used to improve morphological resolution during high-magnification analyses of the submicrometer precipitates located at the fracture surfaces.

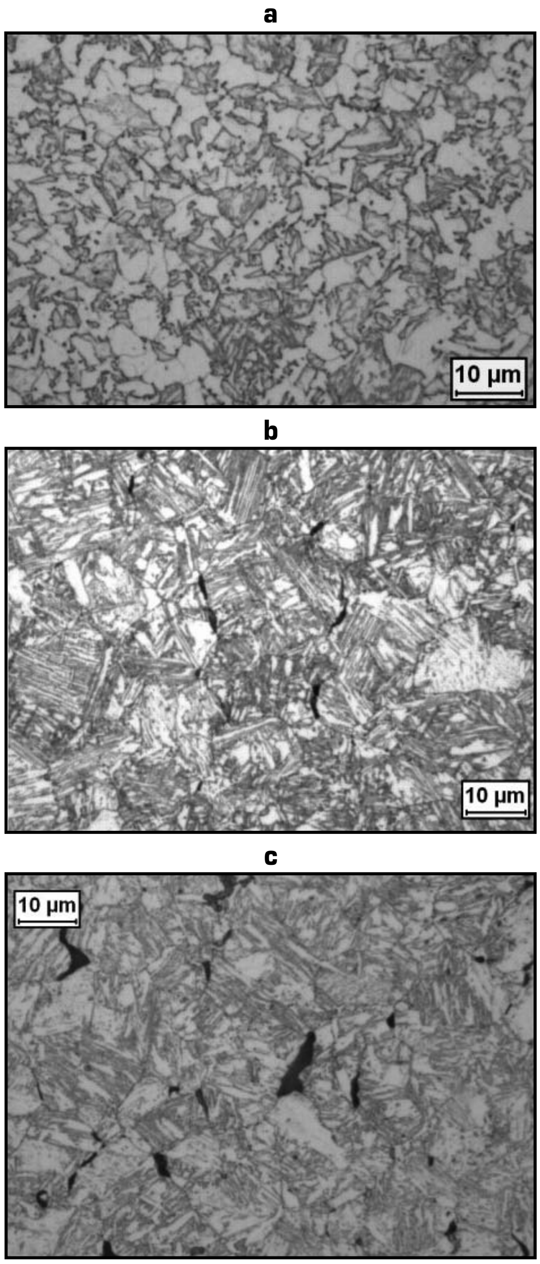

Fig. 3

Representative optical micrographs of a C-Mn microalloyed steel strained at (a) 850°C to 16,7%, (b) 950°C to 16,7% and (c) 950°C to 30%.

Micrografie ottiche di un acciaio microlegato sottoposto a deformazione a caldo alle seguenti condizioni: (a) 850°C al 16,7%, (b) 950°C al 16,7% e (c) 950°C al 30%.

RESULTS

Fig. 3 depicts the microstructure of the hot strained specimens of a C-Mn steel microalloyed with Nb-V after hot straining at different conditions. The optical micrographs were taken from longitudinally sectioned specimens at

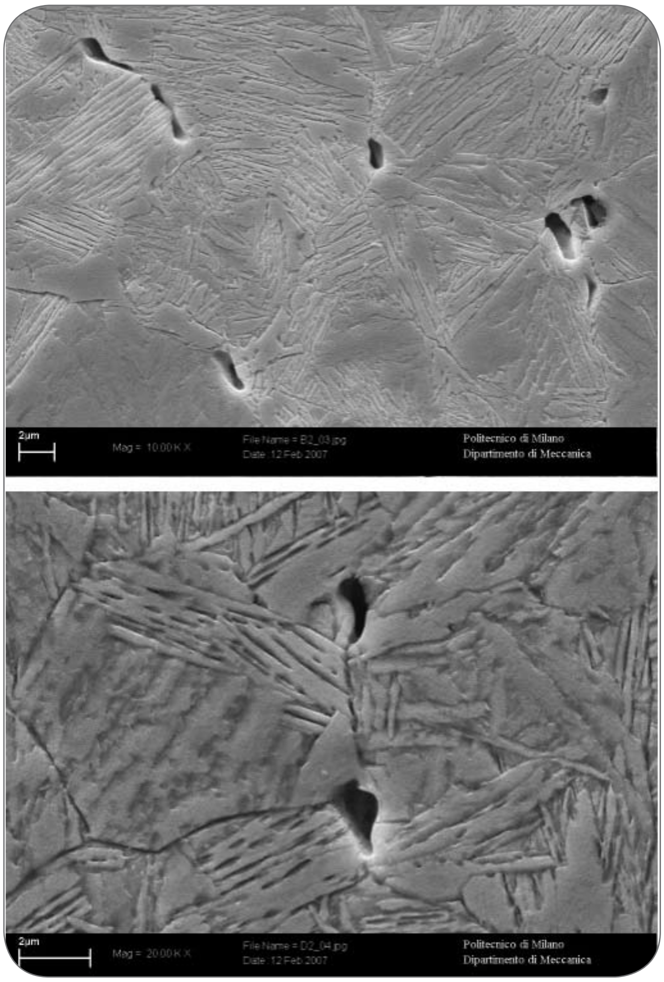

Fig. 4
SEM micrographs of hot strained steels pulled to 16,7% strain at 950°C.
Micrografie SEM di acciai deformati in trazione al 16,7%, alla temperatura di 950°C.

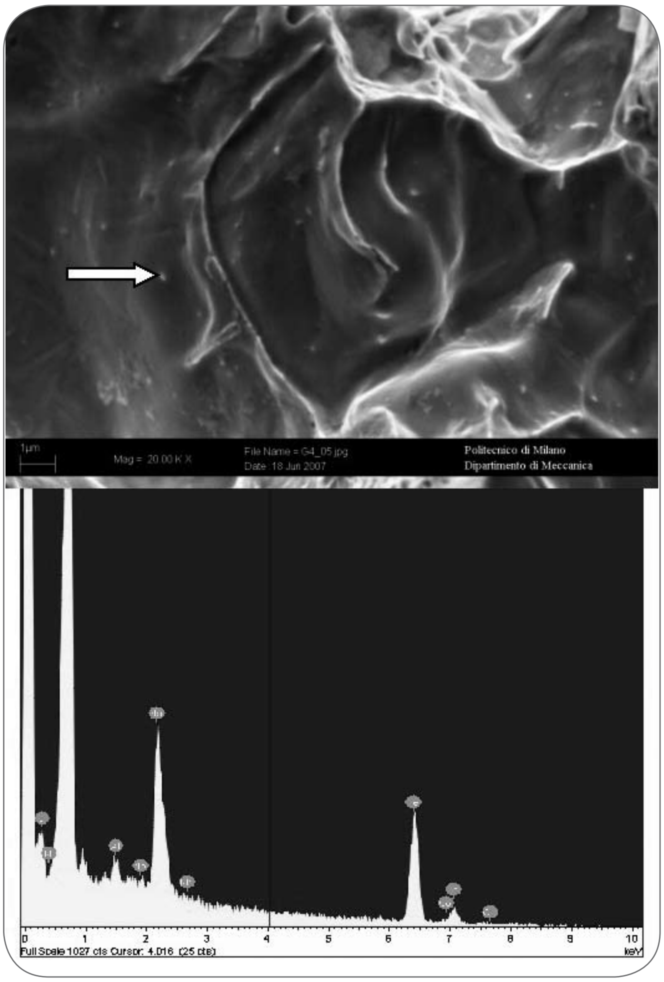

Fig. 5
SEM image of the surface of cracks generated during hot straining at 950°C and EDS spectrum of the arrowed precipitate exposed on the surface.
Immagine SEM della superficie una cricca prodotta durante la deformazione a 950°C ed esempio di spettro EDS del precipitato evidenziato sulla superficie di frattura.

mid length of the gauge portion (the loading direction is always horizontal in the micrographs). Further selected images taken at higher magnification by SEM are given in Fig. 4, showing in more details the intergranular character of the hot cracks.
The collection of the above micrographs suggests that straining at 850°C to 16,7% generally produces only small cracks, hardly detectable on the optical micrographs. At the highest temperature level here examined of 950°C, the damage induced by 16,7% straining is significantly increased. From the micrographs it is inferable that the cracks nucleated at prior-austenite grain boundaries, preferably along those facets that are located perpendicularly to the loading direction (the horizontal direction) and at triple joints of the grains.
To have an improved view of the intergranular crack surfaces, a set of samples pulled in tension at 950°C up to the onset of necking was fractured into two halves after having been cooled into a liquid-nitrogen bath. Information on chemistry of the submicrometer precipitates lying at austenite grain boundaries (exposed on the pre-existing hot cracks) were collected by careful EDS analyses. It is to remark that analyses on the fractured samples were also performed on the materials hot strained at 850°C but the limited amount of damage detected, prevented any analysis on precipitates to be performed.
In Fig. 5, a typical SEM image is displayed together with an EDS spectrum of the exposed precipitates. It can be inferred from the fractographs that the damage mechanism operating at 950°C in the steels investigated consisted of grain boundary sliding, as suggested by the smooth almost featureless grain facets exposed on the free surfaces of the cracks. The EDS spectrum depicted in the figure represents and example of a much larger population of particles analyzed by the microprobe. It was revealed that the main phases existing at grain boundaries are very often rich in Al, presumably combined with N to form the AlN phase. The AlN precipitates can be found in isolated form or they can be combined with Nb(C,N) and/or TiN, depending on steel composition and on specific particle analyzed.
Further FE-SEM observations on the same samples were carried out in order to collect views at higher magnification of the fracture surfaces, with improved resolution

on the tiny precipitates. The images reported in Fig. 6 clearly support the hypothesis that the above described precipitates were the only secondary phases that decorate the austenite grain boundaries of the investigated steels at high temperature. The absence of other, even smaller particles that could have not been resolved by the conventional SEM was therefore verified. FE-SEM analyses also allowed revealing with improved quality some shallow ridges and depressions on the fractured grain boundaries. It is believed that these features could be the traces of the slip bands accommodating plastic deformation inside of the grains and/or groves related to relative movements of adjacent grains during sliding.

DISCUSSION AND CONCLUDING REMARKS

Considering the hot tensile behaviour of the materials in the as-received state, it is to recall that the appearance of moderate or even large amounts of cracks after 16,7% straining at 950°C was the rule, irrespective of steel grade and of its specific composition. Analyses on surfaces of the cracks formed at high temperature showed that the damage mechanism operating in the wrought steels investigated consisted of grain boundary sliding, as suggested by the smooth grain facets exposed on the free surfaces of the cracks and by the presence of shallow ridges and depressions on the fractured grain boundaries. These features were interpreted as traces of the slip bands accommodating plastic deformation inside of the grains and/or groves related to relative movements of adjacent grains during sliding. It is of importance to emphasize that the presence of fine grain-boundary precipitates can enhance the hot-cracking damage by stimulating crack nucleation. This concept was already highlighted during literature review and it was clearly confirmed by the present experimental analyses.
The combined use of both a conventional SEM and a FE-SEM during the fractographic investigation allowed analysing with good accuracy the surfaces of the prior-austenite grain boundaries. It could be stated that a population of tiny precipitates having a size smaller than 100 nm decorate the grain boundaries of the hot-strained steels. EDS spectra suggested that the particles affecting grain-boundary cohesion were rich in microalloying elements. Typically AlN and Nb(C,N) were found in combined or isolated form. Depending on steel composition and on specific particle analyzed, NbV(C,N) and TiN were also detected, even if with a lower frequency.
A comparison of the chemical composition (not reported in detail in this paper) of the steel samples and the qualitative assessment of hot-cracking showed that a simple and unique correlation between composition and hot-cracking damage does not exist for all of the steels investigated. However, from the compositional data, it might be confirmed that Nb would generally have a role on hot ductility, depleting the steel resistance to hot cracking. Ti generally brings an improvement of steel behaviour but, among Ti-bearing steels, no defined trend could be established on the basis of the specific amount of Ti and N of the steels investigated.
Concerning the effect of Al, it is clear from the information collected that Al-containing precipitates do have a strong role on hot ductility behaviour. There is clear evidence from observations of fracture surfaces that any sample

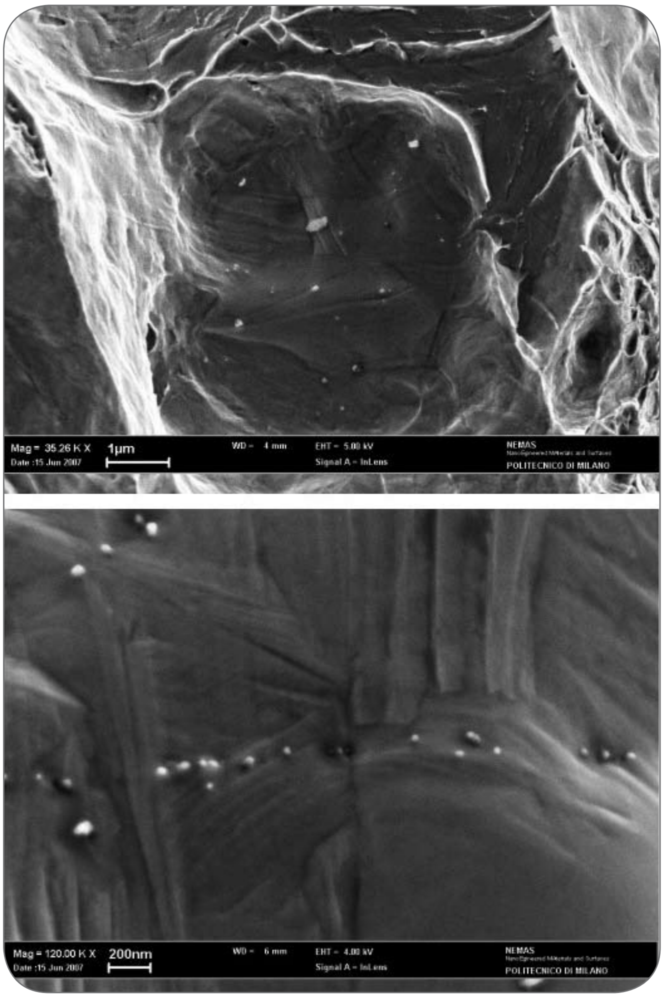

Fig. 6
FE-SEM images of the surfaces of cracks generated during hot straining at 950°C.
Immagini FE-SEM delle superfici di cricche prodotte con la deformazione a 950°C.

that showed poor hot-ductility properties also featured the presence of Al and/or Al-Nb precipitates at austenite grain boundaries.
One of the microstructural parameters that was not possible to assess in this study was the distribution and amount (volume fraction) of the precipitates at austenite grain boundaries. Such information is barely considered also in other literature works owing to difficulties in quantitatively measuring the fine precipitates decorating grain boundaries on a reliable statistical basis. However, from a qualitative visual analysis of the fracture surfaces produced at high temperature, it can be inferred that those steels featuring reduced or negligible tendency toward cracking always showed large dimples on the fracture surface combined with the presence of few particles, often with relatively large size. On the contrary, the steel grades that revealed to be more prone to cracking during hot straining generally showed a fine distribution of precipitates at grain boundaries. It is therefore supposed that hot ductility of the steels is strongly affected also by modifications of the distribution and size of precipitates, the development of cracks increasing with decreasing the size of the grain-boundary precipitates.

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ABSTRACT

MICROALLIGAZIONE E DUTTILITÀ A CALDO DI ACCIAI

Parole chiave: acciaio, frattura, lav. plastiche a caldo

Il calo di duttilità riscontrabile negli acciai, specialmente nelle qualità microlegate, in un intervallo di temperatura variabile tra i 700 ed i 1100°C è un tema molto studiato nel campo della siderurgia. La duttilità a caldo degli acciai viene generalmente valutata attraverso semplici prove di trazione alle diverse temperature, misurando la riduzione percentuale nella sezione di rottura del provino.

Nell'intento di raccogliere informazioni più dettagliate sulla deformabilità e la duttilità a caldo di acciai microlegati, nella presente indagine sono state svolte prove di trazione a caldo fino a livelli di deformazione prefissati, seguite dal rapido raffreddamento del provino in modo da conservare traccia della struttura esistente al momento dell'interruzione della prova. Dopo tale serie di prove a caldo "interrotte", eseguite in varie condizioni di temperatura, deformazione e velocità di deformazione, sono state svolte analisi microstrutturali per identificare i meccanismi di danneggiamento operanti nelle varie condizioni di prova e per i diversi tipi di acciaio.