Reverse ageing of pearlitic carbon steel wire rod

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Abstract

High carbon steels are employed for the production of critical components, such as ropes, cables and springs. After hot rolling, their microstructure must be carefully controlled by Stelmor cooling, since they are often subjected to strong drawing operations. The as-rolled deformability is a basic property to prevent damages in the following plastic deformation. In the industrial world, it is known that their deformability improves significantly after a storage of a certain time, ranging from some days up to 15-20 days. In this experimental work, the deformability of a high carbon steel was investigated after different storage times varying the wire rod diameter. The percentage reduction of area increased after some days, until a stable value reached after about 7-15 days depending on the considered diameter. In the technical literature, this phenomenon is called "reverse aging" and it is addressed to the reduction of the hydrogen content in the material as a consequence of the diffusional mechanisms. Since it is thermally activated, the recovery of deformability was studied also at higher temperatures (from 100°C to 200°C). As expected, the steady values of the reduction of area were obtained very quickly: instead of some days, only few hours were necessary to get a full recovery of the deformability. The UTS values, on the contrary, didn't show any appreciable variation neither during the room nor during the higher temperature aging treatments. The fracture surfaces of the aged specimens were analyzed by SEM.

Introduction

Drawn pearlitic carbon steel wire is commonly used for many structural applications because of its high mechanical strength. Particularly, high carbon wire rod is widely employed for the production of ropes, cables and pre-stressed concrete. The drawnability of such material is a basic property, but it is well known in the industrial world, that the deformability in the as-rolled condition can be critical. In [1, 2], the spheroidizing annealing was employed for increasing the deformability and the influence of the as rolled microstructure was considered and analyzed. It is also well known in the practical experience that for pearlitic carbon steel rods, after storage for 10-15 days at ambient temperature, the deformability shows a huge increase, making it suitable for the following cold plastic deformation. The technical literature [4, 5, 6] addresses such recovery to the release of hydrogen from the wire rod thanks to its high diffusion coefficient [6,7]. Nevertheless, the hydrogen release rate can change a lot on the base of the number of traps such as grain boundaries, precipitates, non-metallic inclusions, lattice defects, inter-space among the pearlite lamellae [6, 7]. The described recovery of ductility is known as reverse aging and can be classified as internal reversible hydrogen embrittlement [8]. This damage manifests itself as a loss of tensile ductility, especially in high strength steels. Such damage is considered reversible if no micro-crack nucleates before a sufficient amount of hydrogen left the material by diffusion [8]. The need of a minimum storage time before being drawn, reduces the productivity significantly. This justifies a wide investigation about the influence of the wire rod diameter and the aging temperature. Since the reverse aging is a diffusion driven phenomenon, it is expected that the recovery time decreases for smaller diameters and for aging temperatures higher than ambient one. The determination of the minimum time necessary to reach the maximum deformability varying the wire diameter can be used to optimize the material storage after the production. The reverse aging kinetics varying the temperature has a great importance too, because it allows the producer to know the wire maximum deformability in a much shorter time in respect to the room temperature aging. The investigation reported in this paper considers three diameters, 8.5mm, 10mm and 12mm, and two kind of aging, natural and artificial.

Material and methods

The material studied in this paper is a high carbon steel subjected to hot rolling and Stelmor cooling by forced air. The obtained microstructure is fully pearlitic.

Wire rod diameter (mm)	**%C**	**%Mn**	**%Si**	**%P**	**%S**	**%Cr**	**%V**
8.5	0.79÷0.81	0.60÷0.70	0,15÷0.25	0÷0.02	0÷0.025	0÷0.10	0÷0.02
10, 12	0.80÷0.82	0.70÷0.80	0.18÷0.28	0÷0.02	0÷0.015	0.25÷0.30	0÷0.02

Table 1. Chemical composition of the analyzed steel.

Three wire rod diameters were considered, 8.5mm, 10mm and 12mm. Their mechanical properties were determined by tensile tests on 300mm long samples according to ISO 6892-1:2019 in the as-produced condition and after natural and artificial aging as reported in Table 2.

Diameter	As-produced	Ambient	100°C	150°C	165°C	200°C
8.5 mm	V	V	V		V	V
10 mm	V	V	V	V		V
12 mm	V	V	V	V		

Table 2. Experimental plan.

The evolution of the material deformability was measured by the reduction of area, because this parameter is generally considered more representative of the steel behavior during the industrial cold plastic deformations. Finally, the tensile specimens fracture surfaces were studied by scanning electron microscope (SEM), in order to associate the microscopic features with the ductility recovery.

Results and discussion

The microstructure of the considered wire rods consisted of fine pearlite with no traces of martensite or secondary cementite network. After being polished by the standard metallographic technique, some specimens were etched by Nital 1% reagent for revealing the microstructure, whereas others were etched by alkaline sodium picrate to reveal the possible presence of secondary cementite network. As an example, the metallographic analysis performed on 8.5 mm and 12 mm diameters wire rod is reported in Figure 1.

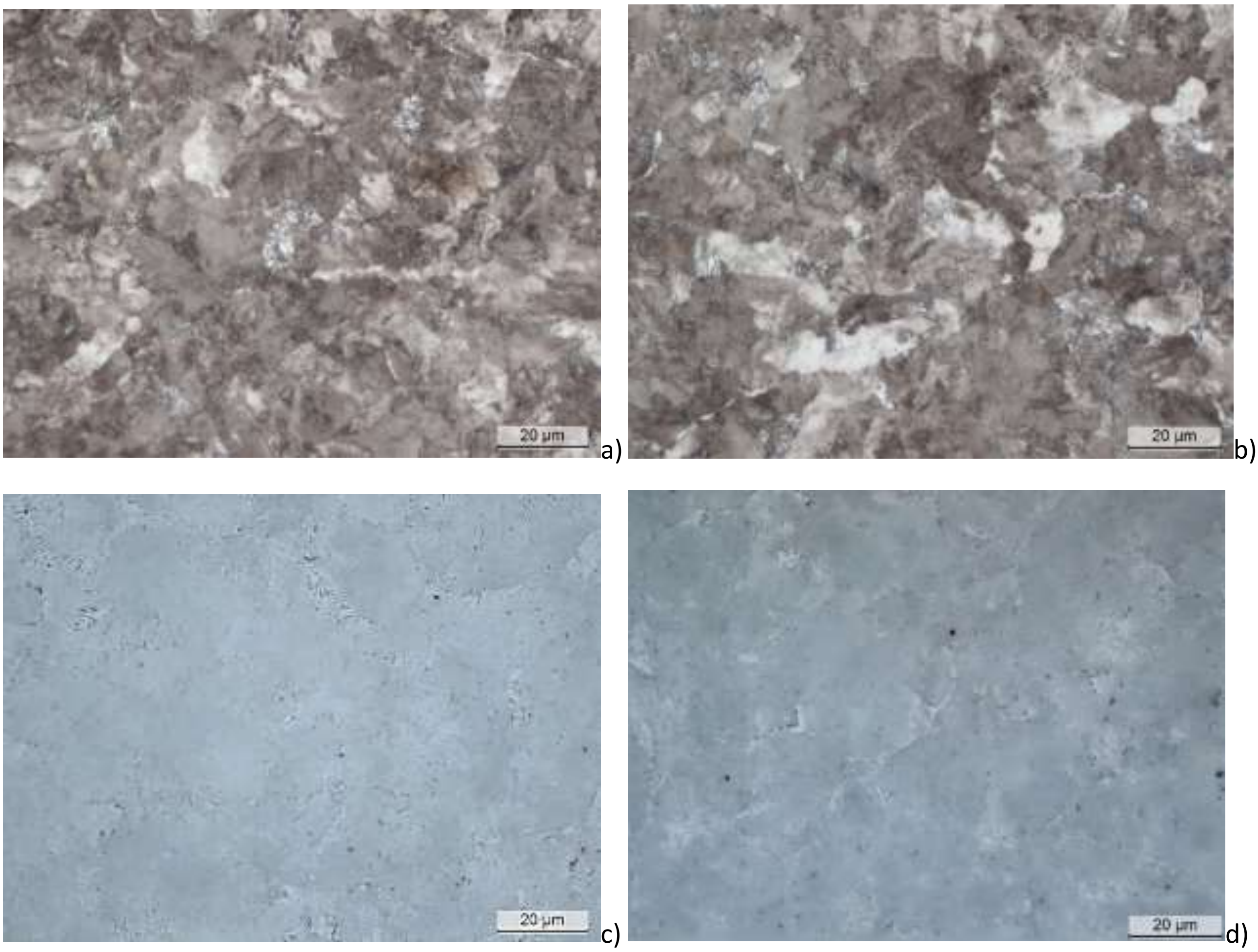


Figure 1. Pearlitic microstructure revealed by Nital 1% etching – (a) 12 mm, (b) 8.5mm. Absence of secondary cementite network, alkaline sodium picrate etching – (c) 12 mm, (d) 8.5 mm.

The deformability evolution was studied by the reduction of area (ROA) coefficient. Figure 2 shows a comparison among the considered diameters.

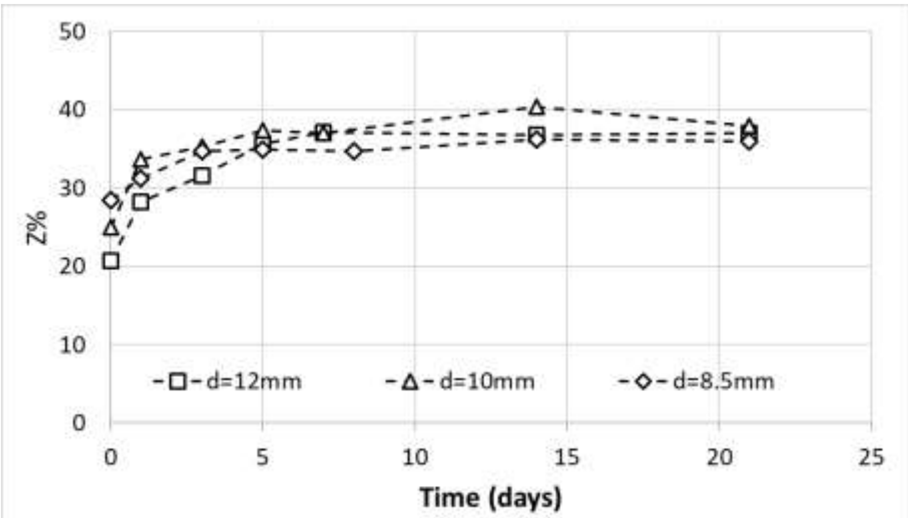


Figure 2. Reduction of area coefficient varying the storage time at ambient temperature.

The data in Figure 2 point out that the as-produced reduction of area is function of the wire rod diameter and that the regime values are very similar for all the diameters. Figure 3 displays a comparison among such values.

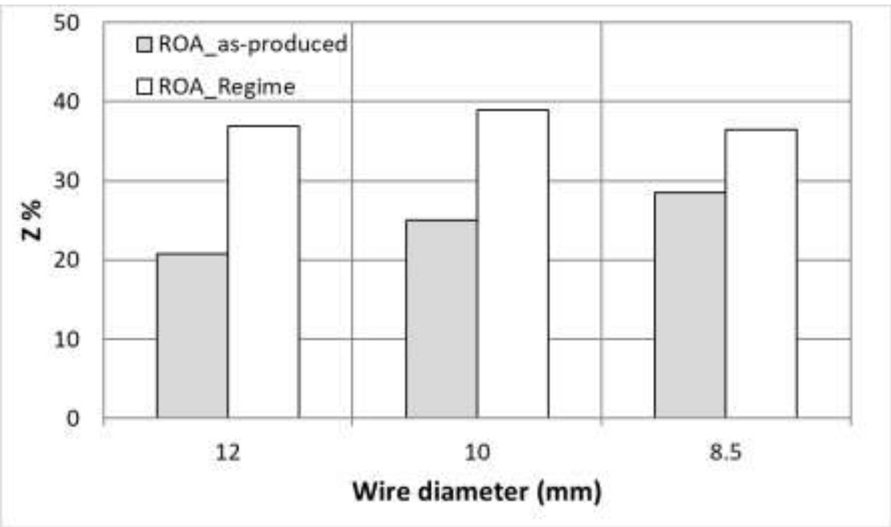


Figure 3. Reduction of area in the as-produced and the regime conditions.

The deformability in the as-produced condition increases as the wire rod diameter decreases. This can be explained considering that the hydrogen release occurs along the whole manufacturing process, especially after the phase transformation since its solubility in the α phase decreases a lot. After the Stelmor conveyor, the wire temperature is about 300°C. The coil moves on specific hooks to the sampling zone and then to the warehouse. The standard quality control procedure requires that the specimens taken from the coil are tested in tension recording the values of the UTS and the reduction of area. For the considered diameters, the elapsed time from the end of the cooling stage and the tensile tests is about 60 minutes. The diffusion of hydrogen occurs all along this time, but the amount that is released is more, when the diameter gets smaller, since the distance it must cover to exit from the metal is shorter.

The regime time is also very important, because it determine when the product can be sent to the customers. As expected, it is function of the wire rod diameter as shown in Figure 4.

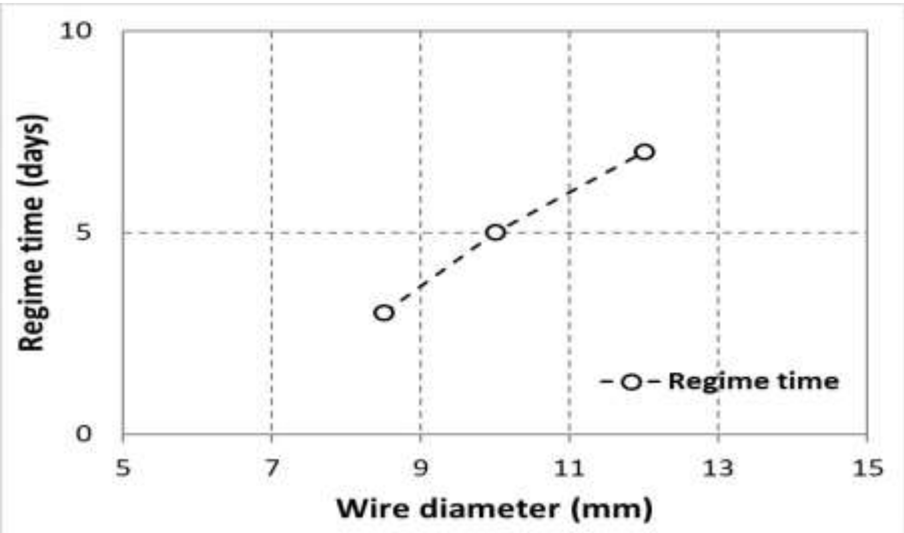


Figure 4. Regime time varying the wire diameter.

Being the reverse aging a diffusion driven phenomenon, the temperature is a key factor. The recovery of the deformability can hence be accelerated by an artificial aging at temperature higher than room one. The artificial aging is often preferred in the standard industrial practice, because it allows the determination of the maximum deformability in a much shorter time, i.e 1-3 hours at temperature included among 100°C and 200°C. Since the regime value of the reduction of area is a basic parameter to determine the quality of the product, the authors investigated the artificial aging too, in order to compare the regime reduction of area values with the ones obtained at ambient temperature. The results are reported in Figure 5.

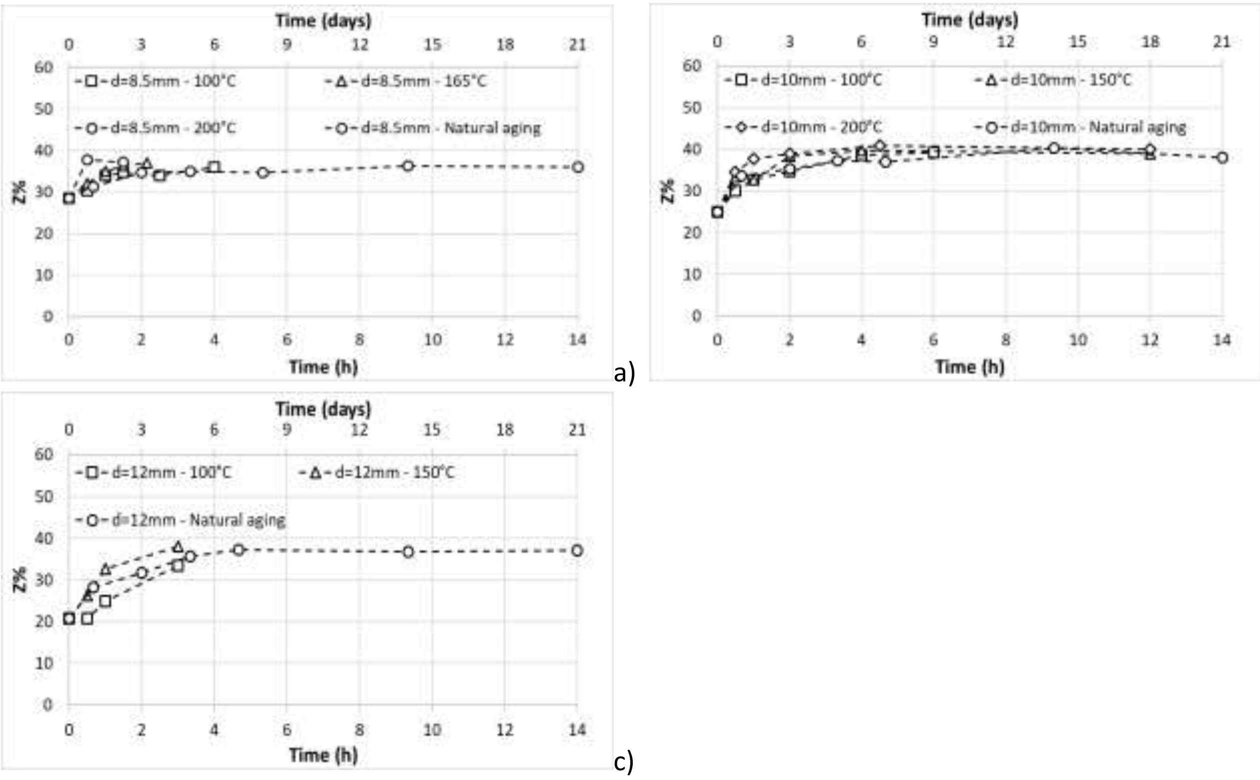
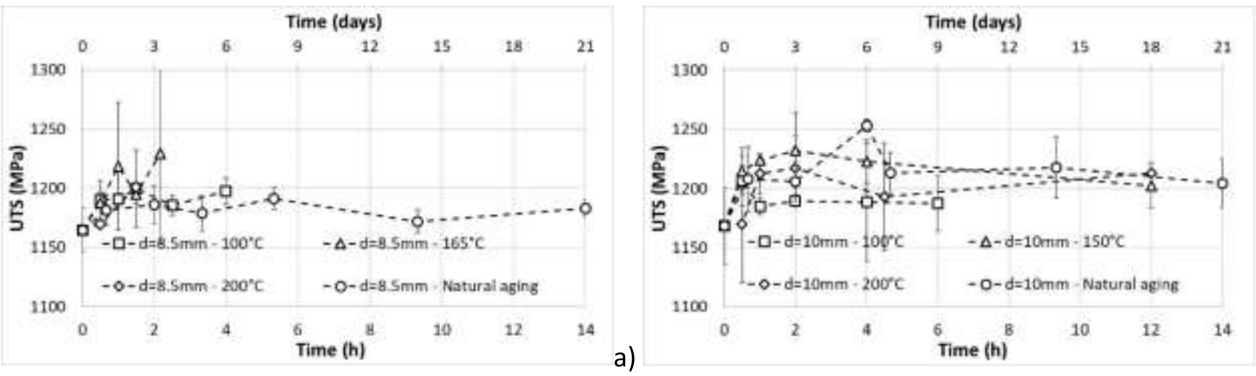
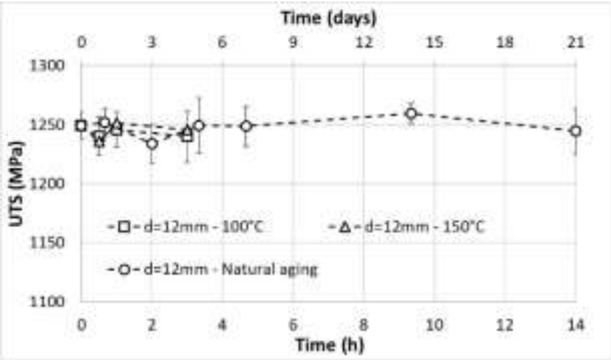


Figure 5. Comparison among the natural and the artificial aging. The standard deviation was lower than 2%, 2.5% and 2.5% for the wire rod diameters 8.5mm, 10mm and 12mm respectively.

The data shown in Figure 5 confirm that the regime reduction of area tend to the same values after both the natural and the artificial aging. The last parameter necessary for the product characterization is the UTS. Before concluding that the artificial aging can be used as standard practice to assess the material properties, it is necessary to study if the artificial aging temperature can modify the steel UTS. Figure 6 shows the obtained results.



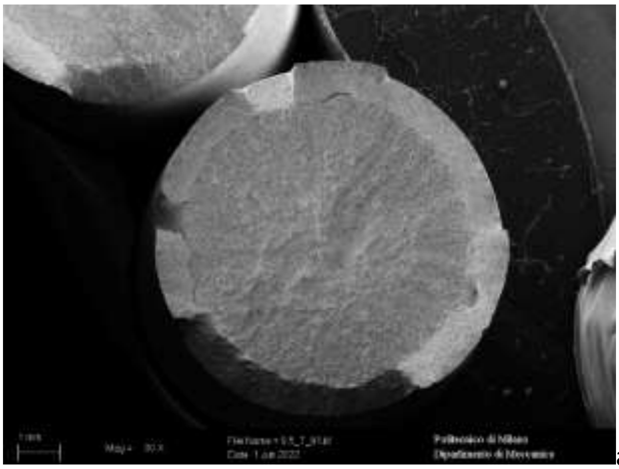


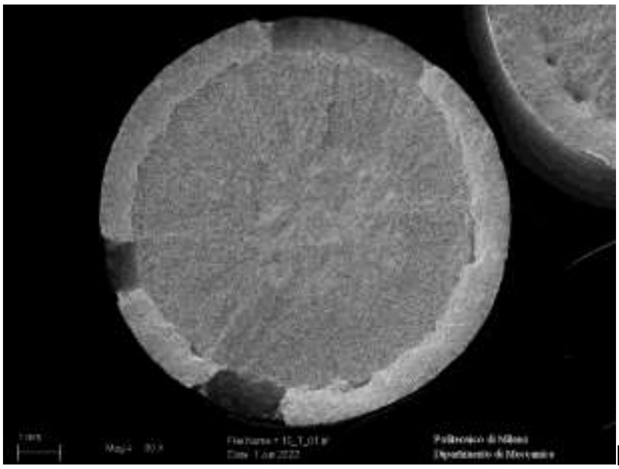
c)

Figure 6. Comparison among the natural and the artificial aging.

The data in Figure 6 confirm that there's no significant influence of the aging temperature on the UTS values.

In order to understand the micro-mechanisms associated to the deformability recovery, a wide SEM investigation of the tensile samples fracture surfaces was carried out. In Figure 7, the fracture surfaces are compared at low magnification: the fracture nucleate in the center zone, propagates radially leaving the well-known radial marks and finally a slant surface is visible [7]. Focusing the attention on the propagation zone at about midway among the center of the sample and the beginning of the slant surface, the microscopic aspects of the fractures were observed at higher magnification. Figures 8-10 show a comparison among the fracture surfaces varying the natural aging time for the investigated wire rod diameters.


a)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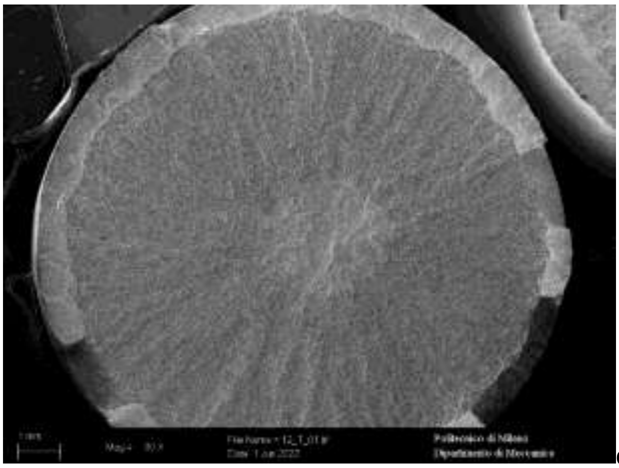

c)

Figure 7. Comparison among the fracture surfaces of the as-produced samples. 8.5 mm (a), 10 mm (b), 12 mm (c).

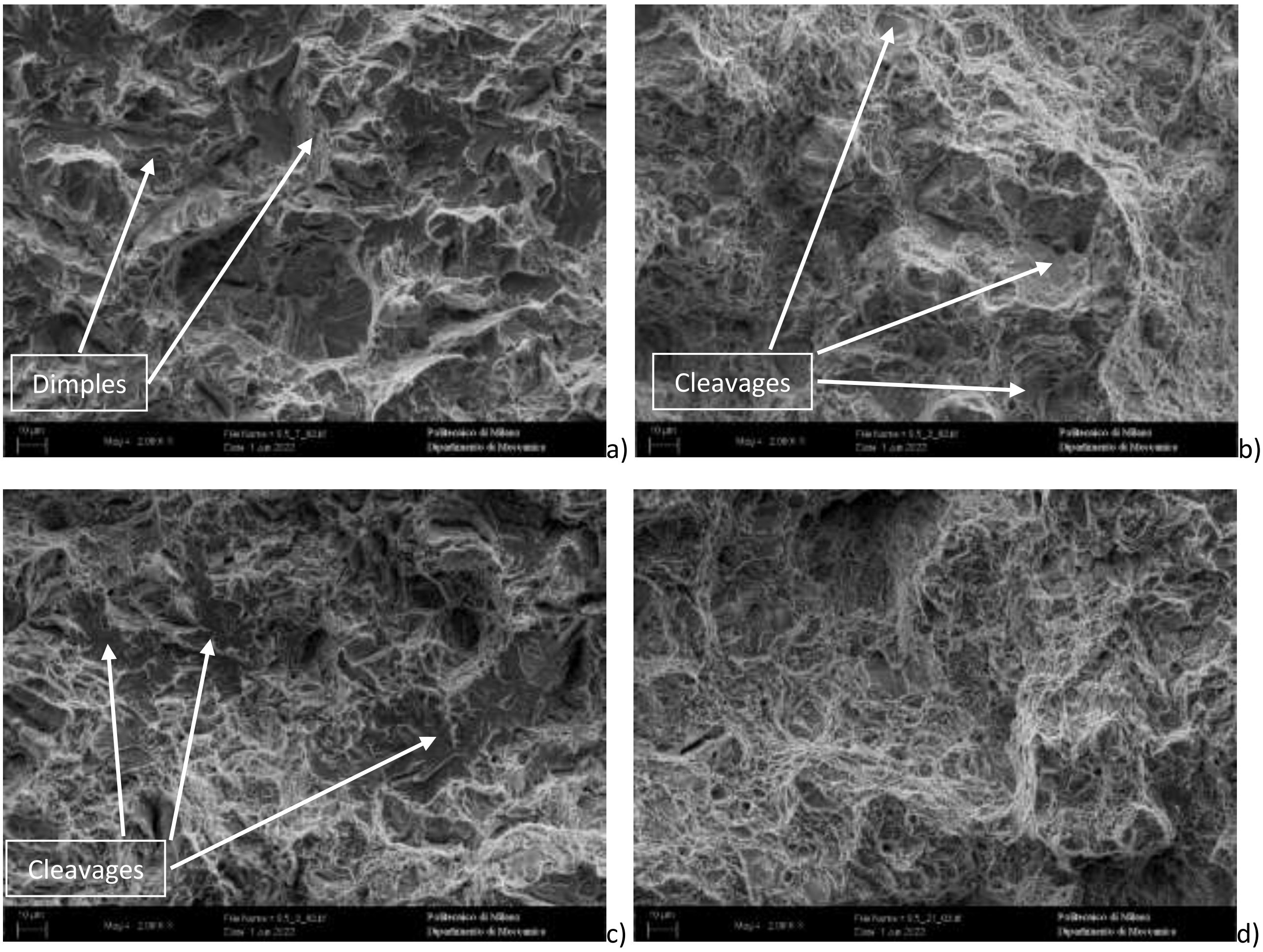
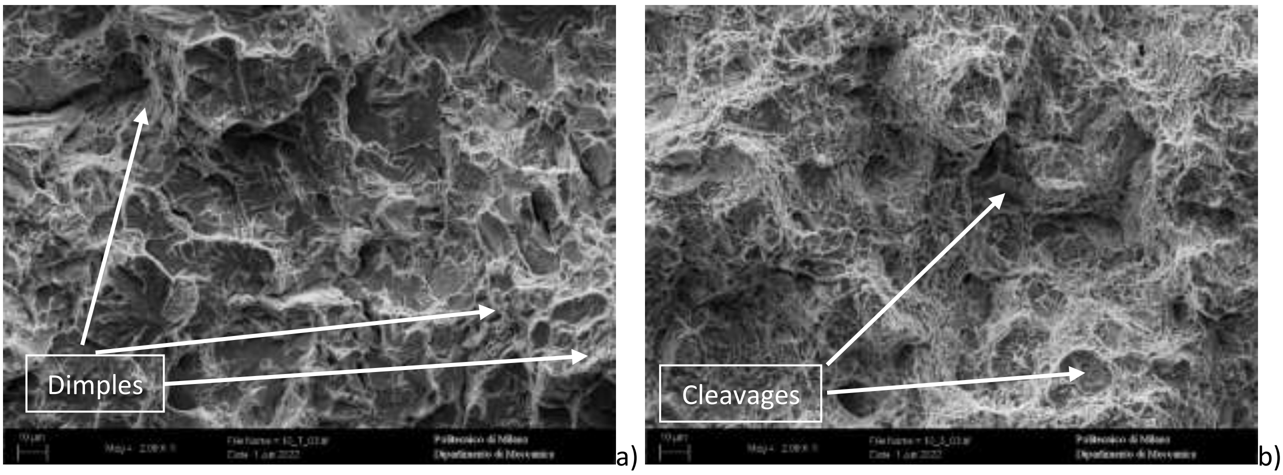


Figure 8. Fracture surfaces of 8.5 mm diameter samples. As-produced – Prevalently brittle fracture surface with small number of ductile zones (a), 3 days natural aging – Prevalently ductile fracture surface, with some cleavage areas (b), 8 days natural aging - Prevalently ductile fracture surface, with some cleavage areas (c), 21 days natural aging – The fracture surface is almost completely ductile (d).



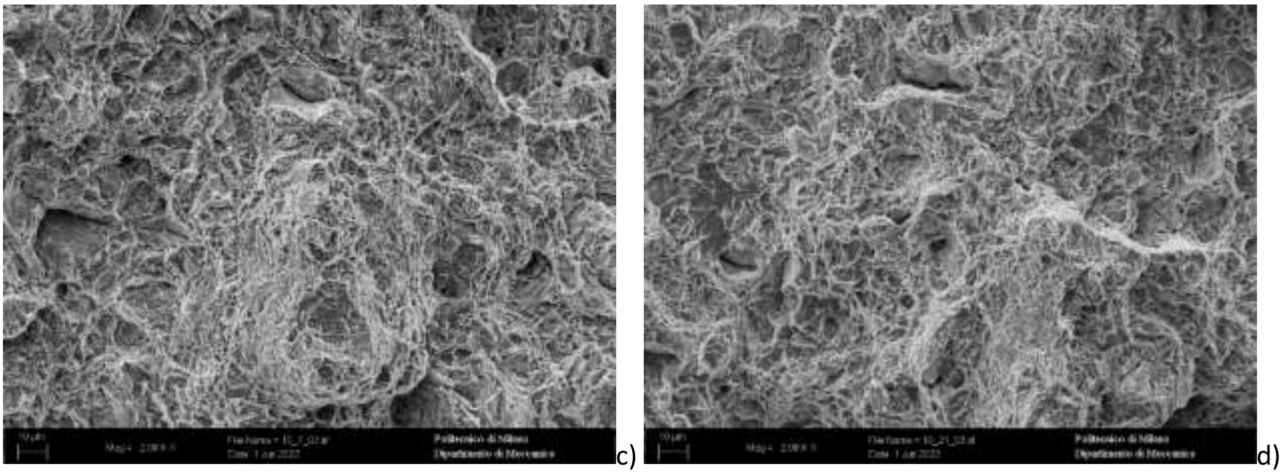


Figure 9. Fracture surfaces of 10 mm diameter samples. As-produced – Prevalently brittle fracture surface with small number of ductile zones (a), 3 days natural aging – Prevalently ductile fracture surface, with some cleavage areas (b), 7 days natural aging – The fracture surface is almost completely ductile (c), 21 days natural aging – The fracture surface is almost completely ductile (d).

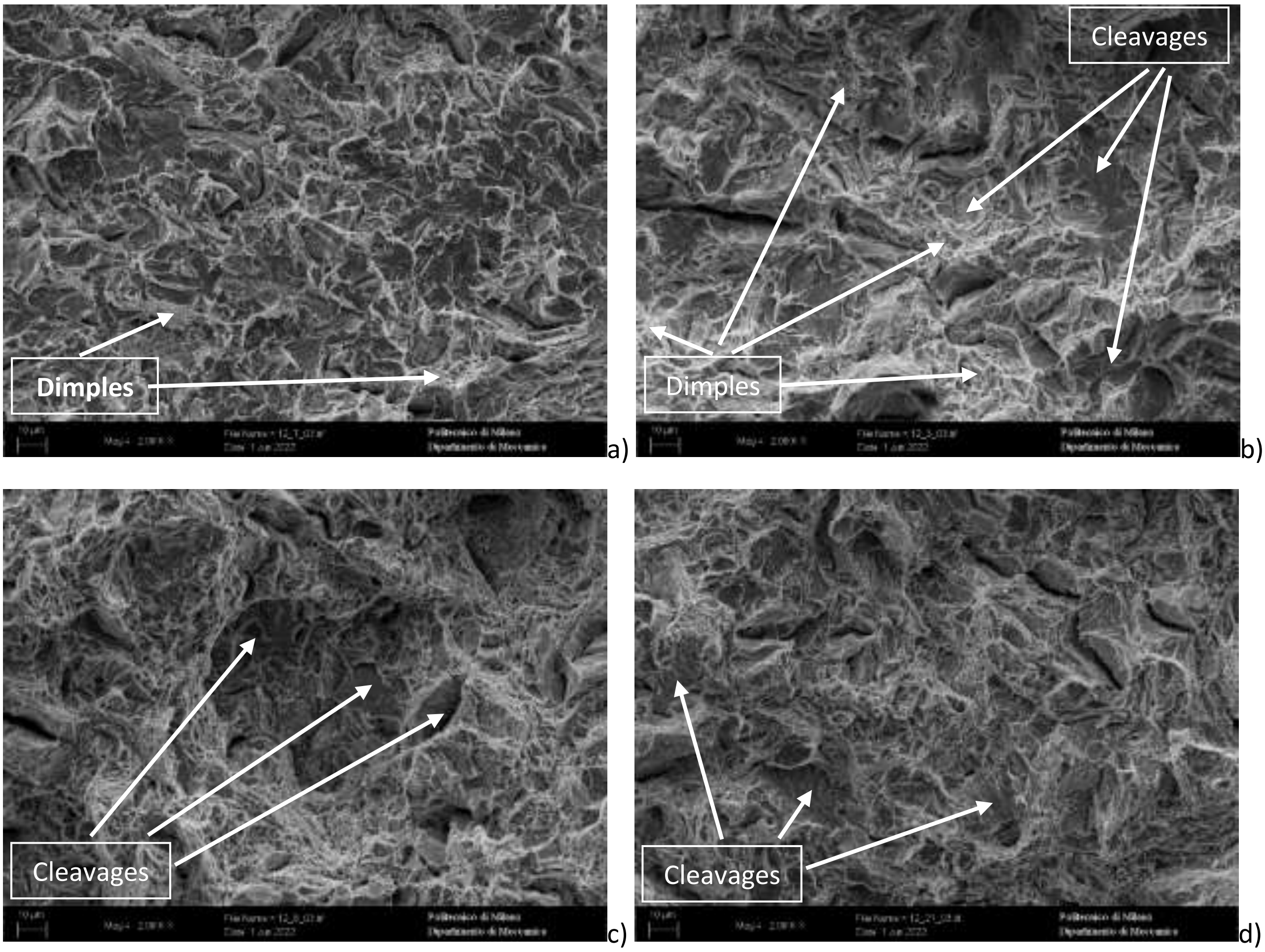


Figure 10. Fracture surfaces of 12 mm diameter samples. As-produced – Prevalently brittle fracture surface with very small number of ductile zones (a), 3 days natural aging – Both brittle and ductile zones are visible (b), 8 days natural aging - Prevalently ductile fracture surface, with some cleavage areas (c), 21 days natural aging - Prevalently ductile fracture surface, with some cleavage areas (d).

The as-produced fracture surfaces are characterized by prevalently brittle features as confirmed by the large amount of cleavage zones. Some micro-dimple areas are visible on the fracture surfaces, even if they decrease passing from 8.5 mm to 12 mm diameters. This justifies the decrease of the as-produced deformability when the rod diameter increases. When the aging time increases, the areas characterized by micro-dimples get larger. This explains the increase of deformability observed in Figure 2.

Since the artificial aging is able to accelerate the deformability recovery greatly, large percentages of ductile regions are expected on the fracture surfaces also for short soaking times. This was investigated by SEM in figures 11-13.

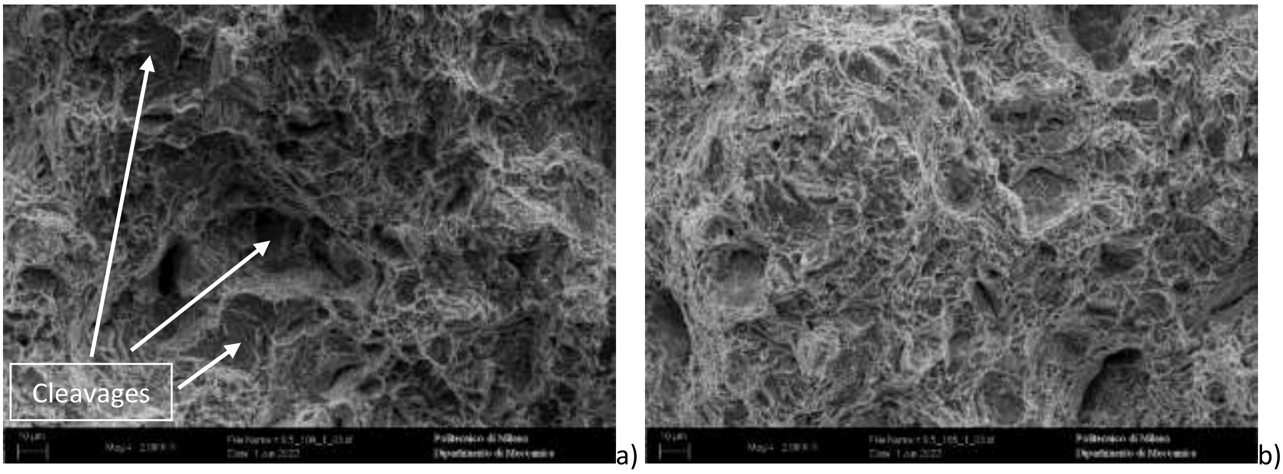


Figure 11. Fracture surfaces of 8.5 mm diameter samples. 100°C, 1h - Prevalently ductile fracture surface with some cleavage areas (a), 165°C, 1h – The fracture surface is almost completely ductile (b).

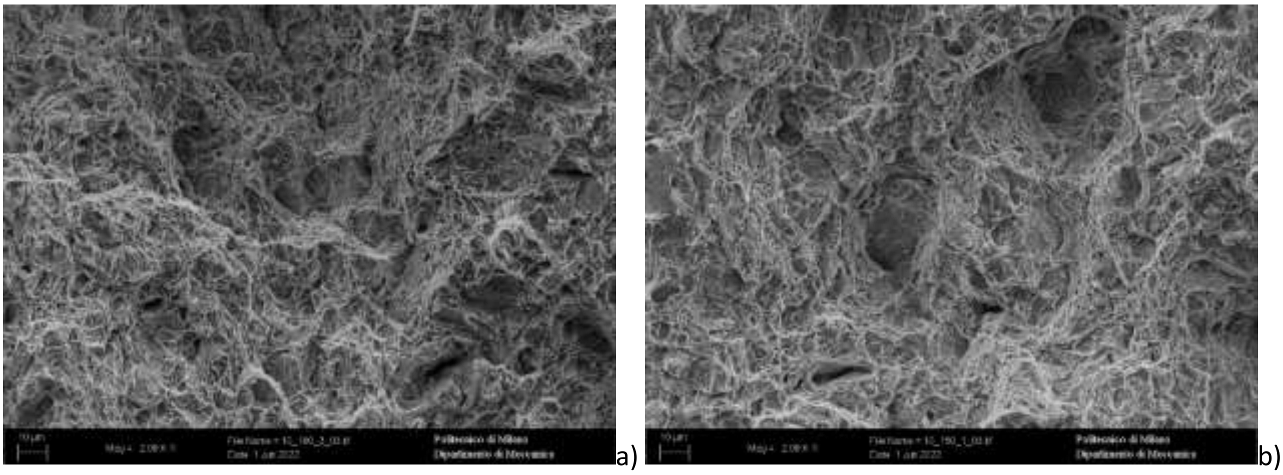


Figure 12. Fracture surfaces of 10 mm diameter samples. 100°C, 3h – The fracture surface is almost completely ductile (a), 150°C, 1h – The fracture surface is almost completely ductile (b).

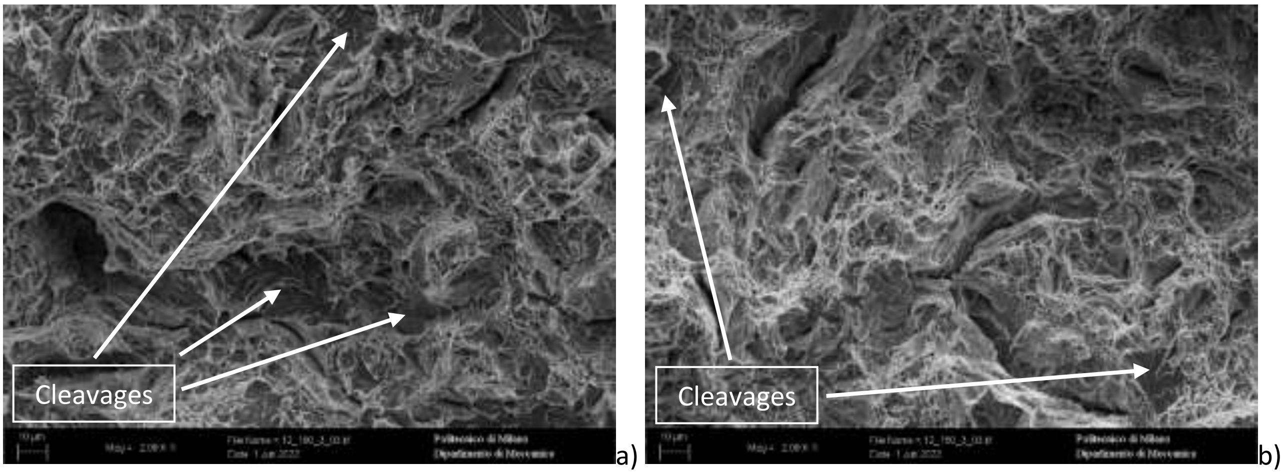


Figure 13. Fracture surfaces of 12 mm diameter samples. 100°C, 3h - Prevalently ductile fracture surface with some cleavage areas. Some secondary cracks are visible too (a), 150°C, 3h - Prevalently ductile fracture surface with some cleavage areas. Some secondary cracks are visible too (b).

Conclusions

This paper investigates one phenomenon known in the technical literature as reverse aging: the deformability of hot rolled high carbon steels increases significantly when stored at room temperature for some days. The reason of such improvement is generally addressed to the release of hydrogen because of its low solubility and its high diffusion coefficient in the α phase. Artificial aging was studied too: being the reverse aging a diffusion driven phenomenon, the temperature is a key factor to increase the deformability recovery rate. By tensile tests, the percentage reduction of area and the UTS were determined for the considered wire rod diameters after both natural and artificial aging. The results confirmed the significant increase of deformability after both kind of aging: considering the regime conditions, an increase of 28%, 55% and 78% were observed for diameters 8.5 mm, 10 mm and 12 mm respectively. UTS values, instead, didn't show appreciable differences neither after the natural nor after the artificial aging. The deformability enhancement was related to the microscopic features observed by SEM on the tensile specimens fracture surfaces. Focusing the attention on the crack propagation zones, all the as-produced wire rods showed a prevalently brittle failure, with presence of small ductile areas whose number increase when the diameter get smaller. The regions characterized by micro-dimples enlarged increasing the soaking time at ambient or at higher temperatures. Such increment of the ductile mechanisms justifies the huge enhancement of the material deformability. Since the hydrogen release is also function of the microstructural features, future developments of this work will consider a reduction of the pearlite colony size by thermo-mechanical rolling after optimization of the process parameters. Moreover, the authors will study the influence of a strong degassing stage on the material deformability in the as-produced and in the aged conditions.

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