Degenerated graphite nodules influence on fatigue crack paths in a ferritic ductile cast iron

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ABSTRACT. Focusing on ferritic-pearlitic DCIs, these alloys are characterized by a microstructure that ranges from a fully ferritic to a completely pearlitic matrix, and they are widely used for many applications (e.g. wheels, gears, crankshafts in cars, exhaust manifolds, valves, flywheels, boxes bearings, hubs, shafts, valves, flanges, pipelines ...). Considering the graphite elements, their morphology can be considered as degenerated when its nodularity is too low and this can be due to different causes (e.g., a partially failed nodularization process or a wrong inoculant). In this work, a ferritic DCI with degenerated nodules was obtained by means of an annealing treatment and the fatigue crack propagation resistance was investigated by means of fatigue crack propagation tests performed according to ASTM E647, focusing on the influence of degenerated graphite nodules on the fatigue crack paths. This analysis was performed both analysing the crack path profile by means of a scanning electron microscope (SEM) and by means of a SEM fracture surfaces analysis.

KEYWORDS. Ferritic-pearlitic ductile cast irons; Fatigue damaging mechanism; Degenerated graphite elements.

INTRODUCTION

Due to their interesting mechanical properties and good castability, DCIs are widely used in the automotive parts (e.g., crankshafts, truck axles, etc.), and in many other application, like pumps, pipes or turbine components [1, 2]. DCIs can be considered as natural composites, with graphite nodules embedded in a metal matrix. The DCIs performances are strongly affected by the graphite elements morphological peculiarities (e.g., graphite elements nodularity, volume fraction, density, distribution, dimension). The most common metallurgical defects in DCIs can be classified as follows [3]:

- Exploded graphite, mainly due to an excess of rare earth additions, Fig. 1;
- Chunky graphite, due to an excess of rare earth additions, Fig. 2;
- Compacted graphite, mainly due to low residual magnesium and/or rare earth (high temperatures or long holding time), Fig. 3;
- Spiky graphite, due to very small amounts of lead which have not been neutralised by rare earth;
- Graphite flotation, which potential causes can be high carbon equivalent, excess of pouring temperature, slow cooling rate in thicker sections or an insufficient inoculation;
- Surface structure, due to a sulphur excess in moulding sand;
- Nodule alignment, due to the presence of large dendrites, with nodules aligned between arms of dendrite;

- Carbides, due to different causes, with a key role played by the presence of carbide promoting elements such as Mn, Cr, V, Mo, and by a rapid cooling rate, Fig. 4;
- Irregular graphite, due to high holding and/or long holding temperature or to a poor inoculation;
- Slag inclusions, that can be due to different causes (e.g., inadequate slag control from pouring system);
- Shrinkage, due to inadequate feed of available metal, excess of magnesium, under or over inoculation;
- Gas holes, that can be due to many causes (e.g., melting procedures).

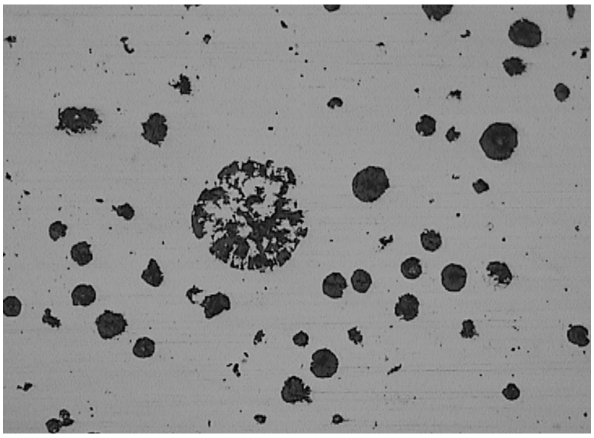

Figure 1: Defects in DCIs. Exploded graphite (x200). Courtesy of Zanardi Fonderie S.p.A.

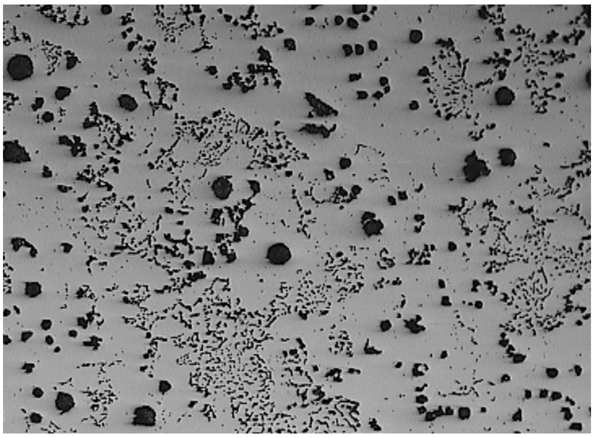

Figure 2: Defects in DCIs. Chunky graphite (x200). Courtesy of Zanardi Fonderie S.p.A.

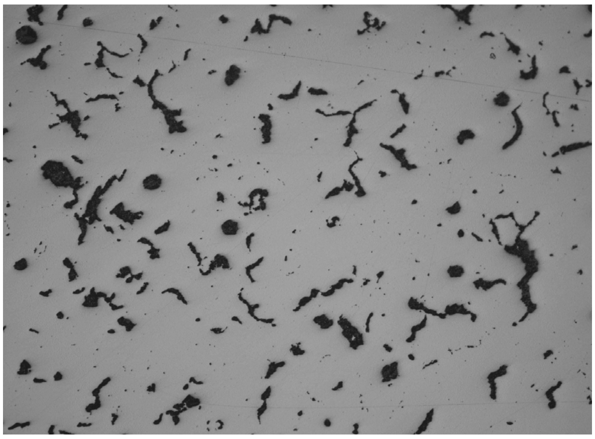

Figure 3: Defects in DCIs. Compacted graphite (x100). Courtesy of Zanardi Fonderie S.p.A.

Figure 4: Defects in DCIs. Carbides (x200). Courtesy of Zanardi Fonderie S.p.A.

All these defects can strongly affect the DCI mechanical behaviour, considering both static or quasi static or cyclic loading conditions [4].
Considering the ferritic DCI, corresponding to lower ΔK, very short secondary cracks are observed (Fig. 5) and the main damaging micromechanism due to the graphite nodules presence is the graphite elements – ferritic matrix debonding, with or without residual graphite (Fig. 5 and 6), respectively). This residual graphite seems to be due to a mechanical properties gradient inside the graphite nodules, with the nodule core (obtained directly from the melted DCI) that is characterized by lower wearing properties and lower hardness values with respect to the outer graphite nodule shield, obtained by means of a carbon atoms solid diffusion during the cooling process [5, 6]. This internal gradient implies the nucleation and propagation of secondary cracks inside the graphite nodules: this mechanism depends on the matrix microstructure and is more evident with static or quasi-static loadings, but can be also observed during fatigue crack propagation (as in Fig. 4 and 6).

In order to investigate the influence of the graphite nodules mechanical properties gradient on the DCI mechanical properties and damaging micromechanisms, in this work, a long annealing heat treatment was performed on a pearlitic DCI in order to activate the carbon atom solid diffusion process and increase the thickness of the outer graphite shield. Fatigue crack propagation tests were performed on a long term annealed DCI and crack paths were investigated by means of a scanning electron microscope. In addition, overloads effects on crack tip were also investigated.

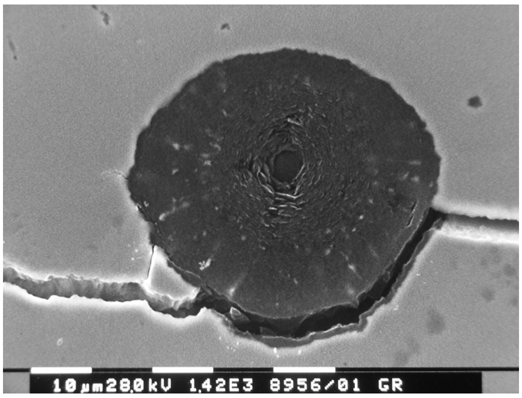

Figure 5: Graphite core-graphite-shield debonding (with residual graphite).

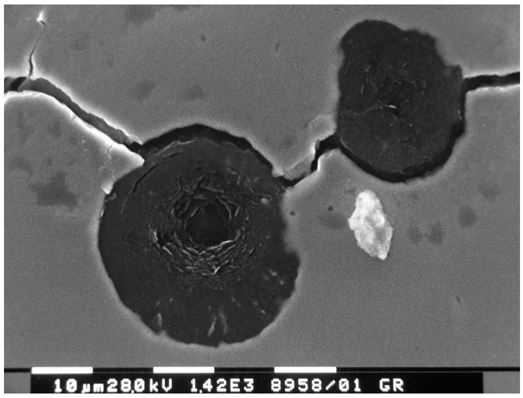

Figure 6: Graphite nodule – matrix debonding and graphite nodule internal damaging.

INVESTIGATED MATERIAL AND EXPERIMENTAL PROCEDURE

A fully pearlitic DCI with a good graphite elements nodularization (chemical composition is shown in Tab.1; microstructure is shown in Fig. 7) was submitted to a long annealing heat treatment, according to the following procedure:
- 170 hours at 850°C;
- Cooling in furnace to lab temperature.

C	Si	Mn	S	P	Cu	Mo	Ni	Cr	Mg	Sn
3.59	2.65	0.19	0.012	0.028	0.04	0.004	0.029	0.061	0.060	0.098

Table 1: Investigated pearlitic DCI chemical composition (5%F + 95%P; before heat treatment).

Heat treatment was performed on 10 mm thick Compact Type (CT) specimens. As a result of this heat treatment, a ferritic matrix with degenerated graphite nodules embedded was obtained (Fig. 8). Nodules were characterized by a surface with a higher roughness (“degenerated nodules”), if compared to the nodules embedded in the DCI before the heat treatment: the long term annealing activated a carbon solid diffusion process, with a consequent increase of the nodules diameters and an evident modification of their shape.
After the heat treatment, CT specimens were submitted to a metallographic preparation procedure. Fatigue crack propagation tests were performed according to ASTM E647 standard [7], with a stress ratio of $R = P_{min}/P_{max} = 0.1$. Tests were performed using a computer controlled servohydraulic machine in constant load controlled conditions, considering a 20 Hz loading frequency, a sinusoidal loading waveform and laboratory conditions. Crack length measurements were performed by means of a compliance method using a double cantilever mouth gage and controlled using an optical microscope (x40). During the fatigue crack propagation tests, SEM crack path observations of the specimens lateral surfaces were performed with a step by step procedure. Furthermore, fracture surfaces were analysed by means of a scanning electron microscope, focusing both the graphite elements and the metal matrix (crack propagates always from left to right). Results were compared with the behaviour of a ferritic DCI with “normal” graphite nodules [8, 9] (chemical composition is shown in Tab. 2).

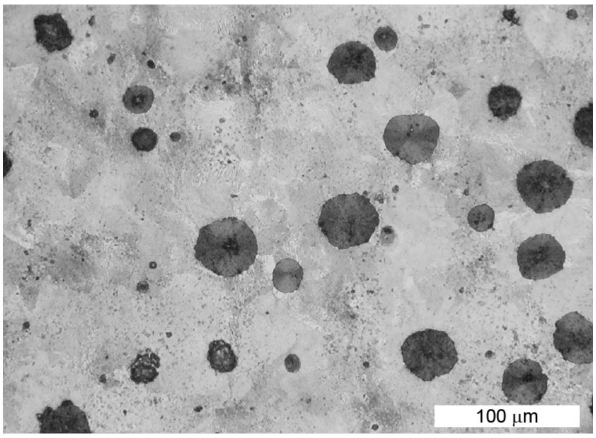

Figure 7: Pearlitic DCI before long annealing heat treatment.

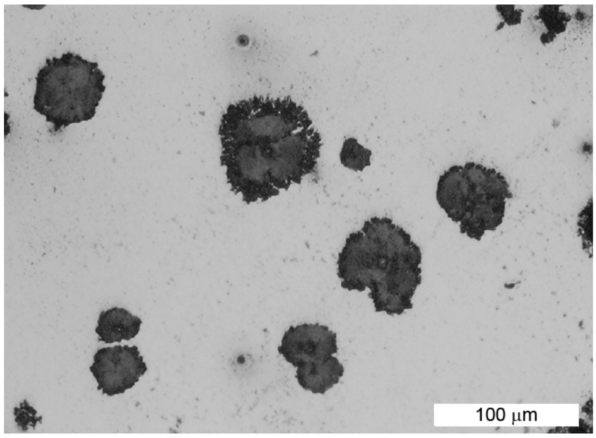

Figure 8: Pearlitic DCI after long annealing heat treatment (ferritic matrix and degenerated graphite elements).

C	Si	Mn	S	P	Cu	Cr	Mg	Sn
3.66	2.72	0.18	0.013	0.021	0.022	0.028	0.043	0.010

Table 2: Ferritic DCI chemical composition (100%F).

In addition, a 3D fracture surface reconstruction procedure was performed. Corresponding to the same specimen position, a stereoscopic image was obtained performing an eucentric tilting around the vertical axis and capturing two different images, with a tilting angle equal to 6° (tilting results in a static center point in the image), Fig. 9. 3D surface reconstruction was performed using the Alicona MeX software and profile evolution was analysed corresponding to graphite nodules.

Finally, static overloads were applied according to the following step-by-step procedure:

1) Applied K_I increase was obtained by means of a servohydraulic machine under load control conditions. Corresponding to each overload, COD was measured. Applied K_I values were: 10, 20, 30, 40 MPa√m, respectively.

2) The load was decreased to zero and the specimen was removed from the grips. Using the "screw loading machine" in Fig. 10, the specimen was loaded again up to the same COD value obtained in step 1. This "screw loading machine" allowed to observe the specimen lateral surface by means of a SEM under overloading conditions.

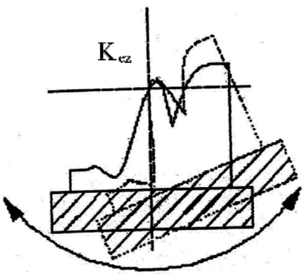

Figure 9: Eucentric tilting.

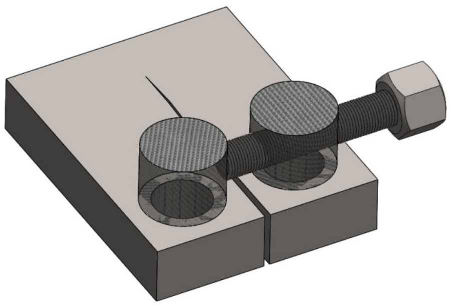

Figure 10: "Screw loading machine".

Results and discussion

Fatigue crack propagation

The result of the long annealing heat treatment of the pearlitic DCI is a ferritic matrix with degenerated graphite nodules. These are characterized by a larger radius and by a higher surface roughness, if compared to the "starting" pearlitic DCI (Fig. 7 and 8, respectively): the outer shield obtained during the long annealing treatment

is characterized by the presence of some small metal particles embedded in the matrix.
Considering that fatigue crack propagation results in DCIs is usually characterized by a good repeatability [10] and that, for lower R values, the matrix influence is almost negligible [9], Fig. 11, fatigue crack propagation resistance of the ferritized DCI seems to be influenced by the presence of the degenerated graphite nodules (Fig. 12).

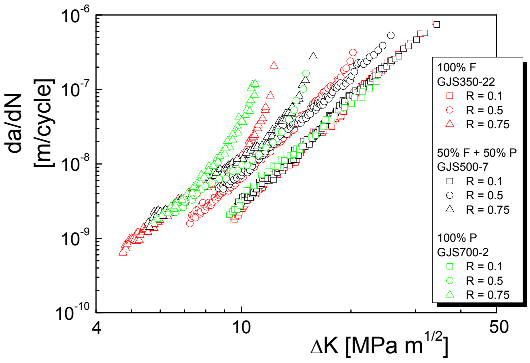

Figure 11: Matrix and loading conditions influence on DCIs ferritic-pearlitic fatigue crack propagation resistance [6].

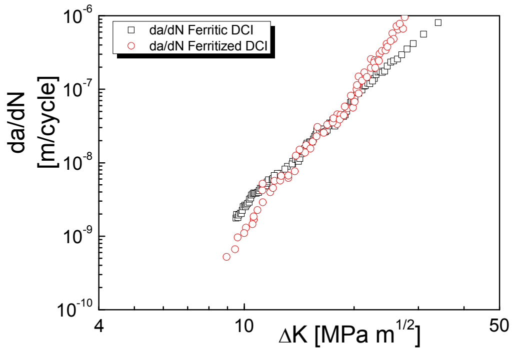

Figure 12: Degenerated graphite elements influence on fatigue crack propagation in ferritic DCI.

Considering that the long annealing treatment modified both the dimension and the shape of the graphite nodules, and considering the hypothesis that the graphite obtained directly from the melt during solidification (nodule core) is characterized by a different behaviour with respect to the graphite of the outer shield (obtained during the cooling process by means of a solid diffusion process), it is hard to define to contribution of all the geometrical and material parameters (graphite nodules shape and dimension and graphite mechanical properties gradient) on the da/dN-ΔK results.
Analysing the fatigue crack path (Fig. 13), it is possible to observe that the path tortuosity is analogous to the one observed with the ferritic DCI [9, 10]. Secondary crack are frequent but short. The main difference concern the interaction between the graphite nodules and the crack: the debonding between the graphite nodules and the matrix or the "internal" debonding between the nodules core and the nodules shield are difficult to observe: more frequently, fatigue crack propagates inside the graphite nodule.

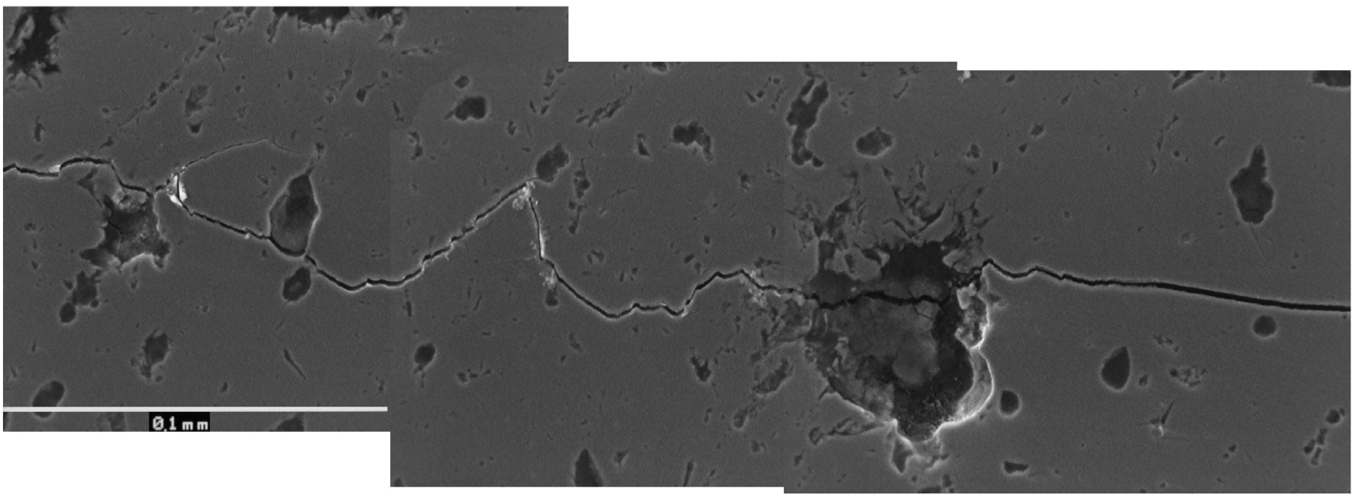

Figure 13: Ferritized DCI. Crack path.

Fracture surface SEM observations allows to identify the interaction mechanism between the fatigue crack and the "degenerated" graphite nodules (Fig. 14, 15). Fatigue crack propagates inside the graphite shield obtained during the long annealing treatment, probably following the interfaces between graphite and metal particles. Secondary cracks nucleate and propagate corresponding to the interface between the shield obtained during the long annealing treatment and the "original" graphite nodule. The main crack propagates along this interface, with an apparent graphite nodule disaggregation. This micromechanisms is more evident considering 3D images obtained according to the "3D reconstruction" procedure described in the *Investigated material and experimental procedure* paragraph.

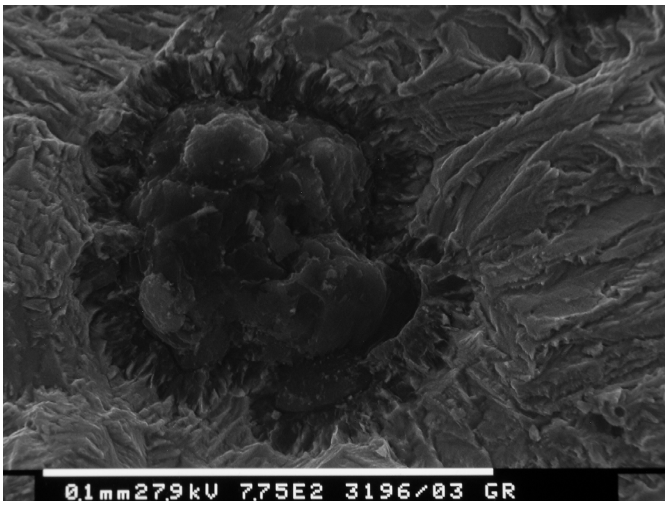

Figure 14: Ferritized DCI. SEM fracture surface analysis (ΔK = 12 MPa√m).

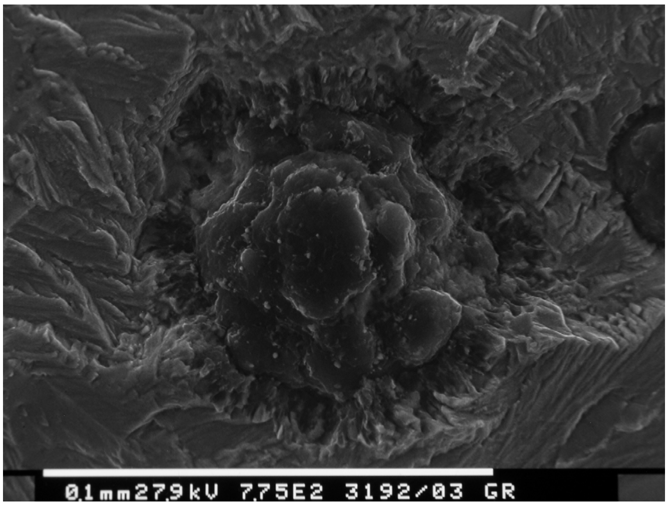

Figure 15: Ferritized DCI. SEM fracture surface analysis (ΔK = 16 MPa√m).

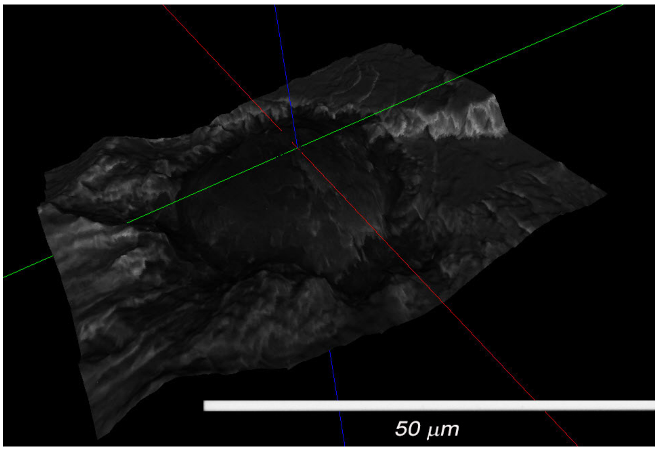

Figure 16: Ferritized DCI. SEM fracture surface analysis: 3D reconstruction (ΔK = 14 MPa√m).

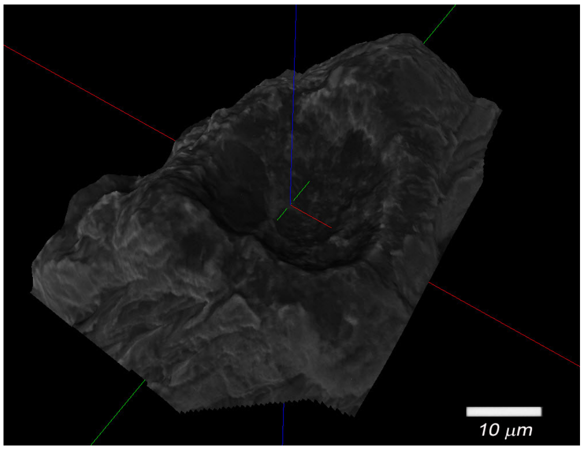

Figure 17: Ferritized DCI. SEM fracture surface analysis: 3D reconstruction (ΔK = 15 MPa√m).

3D reconstructed images confirm the micromechanisms formerly described: crack propagates in the matrix and in the shield obtained during the long annealing treatment, but avoids to propagate inside the graphite nodule: the result on the fracture surface can be a nodule embedded in the ferritic matrix or a void, depending on the crack propagation plane with respect to the nodule position.

Overloads

Overloads applied on DCIs have a different effect depending on the matrix microstructure. All the ferritic-pearlitic DCIs are characterised by the increase of a plastic-damaged zone at the crack tip that becomes more and more evident with the increase of the applied K_I, but:

- Pearlitic DCIs are characterized by a crack branching and a stable crack propagation; crack path after the overload is more tortuous than the path obtained during the fatigue crack propagation [11];
- Ferritic-pearlitic and ferritic DCIs are characterized by a negligible crack stable propagation, with an increase of the crack tip blunting with the increase of the applied K_I [10].

Considering the ferritized DCI investigated in this work, the increase of the crack tip blunting with the increasing of the applied K_I value is still evident, (Fig. 18), but there is also the stable crack propagation, although less important with respect to the same phenomenon in pearlitic DCIs.

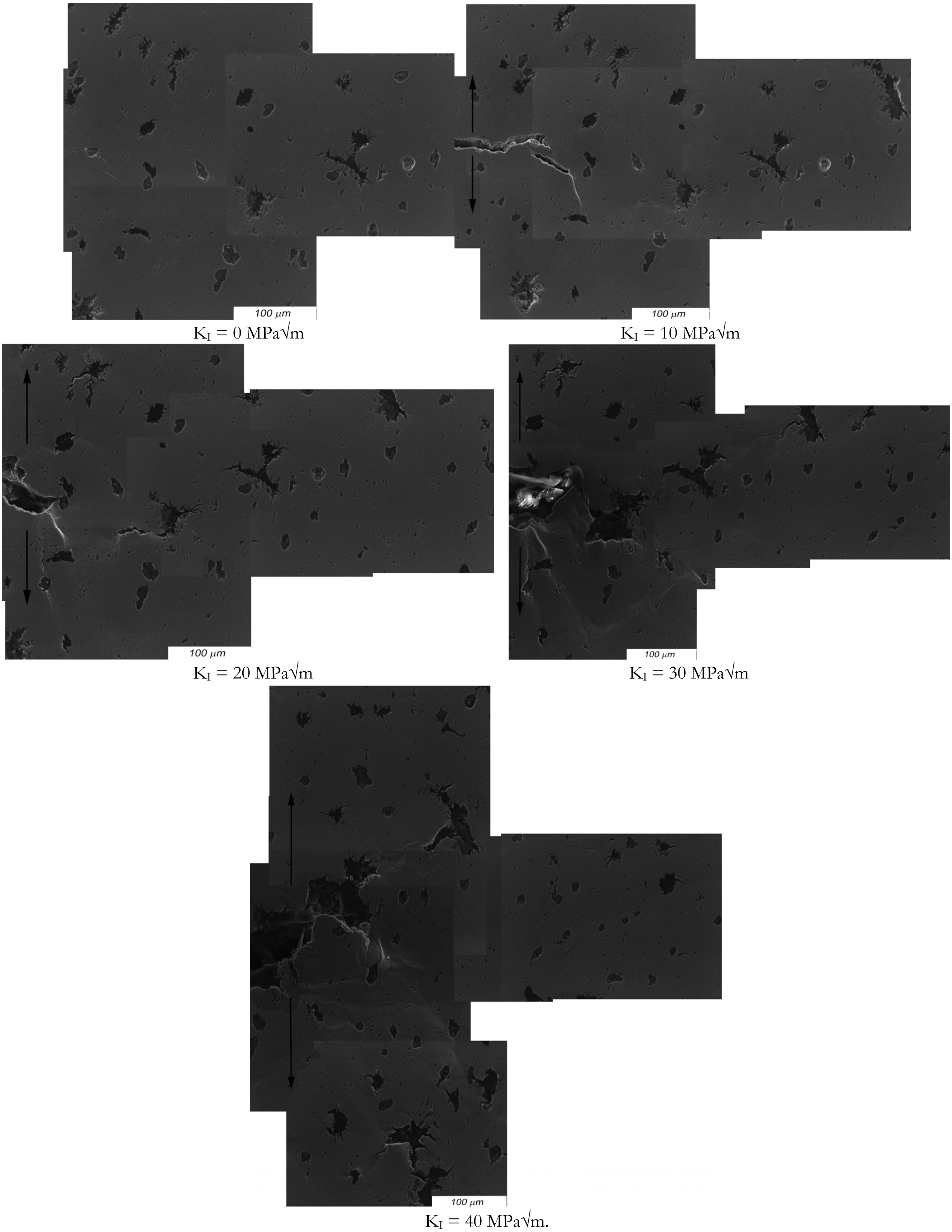

Figure 18: Overloads effect on fatigue crack (arrows shows the loading direction).

Furthermore, the increase of the applied K_I implies the generation of slip bands in the ferritic matric and the nucleation of secondary cracks (especially corresponding to graphite elements), with the coalescence of the secondary cracks that contributes to the stable crack propagation. Analogously to ferritic, ferritic-pearlitic and pearlitic DCIs, around the crack tip the increase of the applied K_I generates a plastic/damaged zone instead of a "mere" plastic zone.
Considering the fracture surface, it seems that graphite residuals are less evident with respect to the fracture surface obtained during the fatigue crack propagation (Fig. 19) and that the voids around the graphite elements are more "developed", with an evident ductile morphology (Fig. 19 and 20)

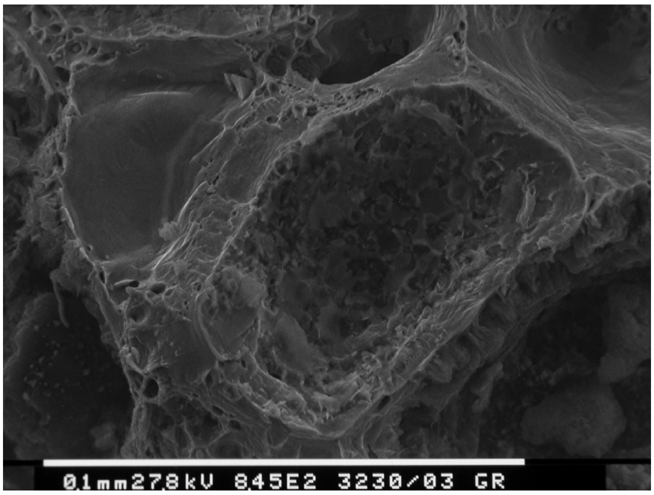

Figure 19: SEM observation of the fracture surface (after overloads).

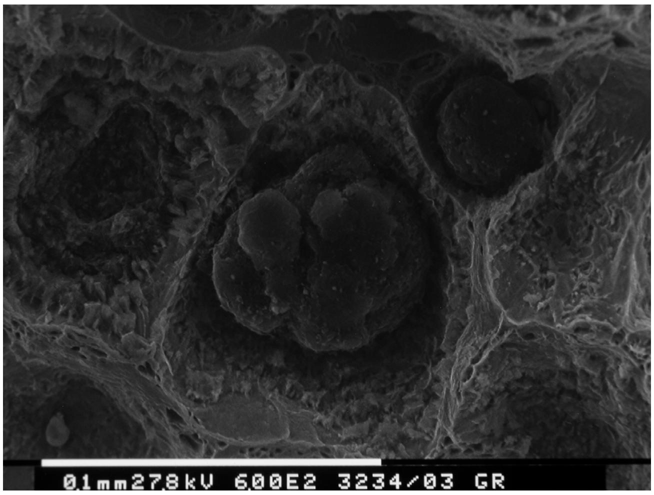

Figure 20: SEM observation of the fracture surface (after overloads).

CONCLUSIONS

In this work, a long annealing heat treatment was performed on a pearlitic DCI in order to activate the carbon atom solid diffusion process and increase the thickness of the outer graphite shield. Fatigue crack propagation tests were performed on a long term annealed DCI and crack paths were investigated by means of scanning electron microscope observations. In addition, overloads effects on crack tip were also investigated.
According to the experimental results, the following conclusions can be summarised:

- The long duration high temperature annealing (170 h at 850°C) allows the activation of the carbon atoms solid diffusion mechanism: the final microstructure shows a ferritic matrix with graphite elements with an increased radius and a modified morphology ("degenerated" nodules).
- Comparing the behaviour of the obtained ferritized DCI with degenerated graphite nodules with a "standard" ferritic DCI, fatigue crack propagation seems to be affected by the different nodules morphology.
- Considering the interaction between the "degenerated" graphite nodules and the fatigue crack, the graphite shield obtained during the annealing treatement is characterized by a different mechanical behaviour if compared to the "original" graphite nodules;
- Considering overload effects, Ferritized DCI with degenerated nodules show both the crack tip blunting (analogously to ferritic DCIs) and the stable crack propagation (as in pearlitic DCIs). Around the crack tip a plastic/damaged zone is observed instead of a "simple" plastic zone.

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