THE INFLUENCE OF PLASMA NITRIDING AND POST OXIDISING TREATMENT ON THE RESISTANCE OF AISI H11 TO CYCLING IMMERSION IN MOLTEN ALUMINIUM ALLOY

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Abstract

This paper is based on an experimental tests plan, which aims at strengthening the knowledge on the influence on die life of plasma nitriding and post oxidising surface modification treatment. An AISI H11 steel obtained through modern vacuum melting and remelting processes was used as base material. Un-notched impact test specimens were fabricated from a core vacuum heat treated block. Some specimens were plasma nitrided and oxidised. Impact toughness was evaluated both on some of the heat treated and surface modified specimens. The other specimens were subjected to a program of cyclic immersion in molten aluminium alloy and cooling in water. The heat checking and soldering phenomena were analysed through periodic inspections and, after the test through, the evaluation of cracks and cavities length and density. At the end of thermal fatigue test the impact test was also performed to assess the toughness modification in comparison with the initial condition. Finally, a metallographic investigation was performed through optical and electron microscopy on all the specimens considered.
Both soldering and heat checking were found to be active damaging mechanisms on both simply heat treated and nitrided and oxidised specimens. However, all the revealed damages resulted to be limited to the surface and sub-surface layers. The clearer effect which was detected was the softening, clearly visible through impact toughness increases and microhardness decreases along with the number of immersion cycles. The amount of softening was found to be limited by the surface modification treatment. The plasma nitriding and post oxidising treatment had also prevented the metallic surface from corrosion attack (acting as a barrier to the aluminium aggression) at least for the first 8,500 immersion cycles. The developed test rig for cyclic immersion in molten aluminium alloy provided results similar to other experiences currently present in the open literature

Riassunto

Il presente articolo è basato su un programma di prove di laboratorio finalizzato a studiare l'influenza di un trattamento combinato di nitrurazione e post-ossidazione in plasma sulla durata degli strati superficiali dello stampo. Come materiale base è stato utilizzato un acciaio AISI H11, ottenuto per fusione e rifusione sotto vuoto. A partire da un blocco prelevato a cuore e trattato termicamente in vuoto sono stati fabbricati dei campioni lisci per prova di impatto. Alcuni dei campioni prodotti sono stati nitrurati e ossidati in plasma. La tenacità a impatto è stata valutata sia su campioni solo trattati termicamente sia su campioni modificati superficialmente. I rimanenti campioni sono stati sottoposti a un programma di immersione alternata in lega di alluminio fuso e in un bagno di raffreddamento. I fenomeni di fatica termica e saldatura sono stati analizzati attraverso indagini periodiche e, a fine prova, attraverso la valutazione della profondità e della densità delle cricche e delle cavità. Dopo l'esecuzione della prova di fatica termica è stata eseguita anche la prova di impatto al fine di valutare le variazioni della resilienza rispetto alle condizioni iniziali. Infine, su tutti i campioni considerati è stato eseguito uno studio metallografico tramite microscopia ottica ed elettronica.
La saldatura e la fatica termica sono risultati essere meccanismi attivi sia sui campioni semplicemente trattati termicamente sia su quelli nitro-ossidati. In ogni caso, tutti i danneggiamenti rilevati sono apparsi essere limitati agli strati superficiali e sub-superficiali. La principale modificazione delle proprietà dei campioni che è stata riscontrata dopo il ciclaggio in alluminio è quella dell'addolcimento: chiaramente identificabile dall'incremento della tenacità a impatto e dalla diminuzione della microdurezza con l'aumentare del numero di cicli di immersione. L'addolcimento è risultato molto intenso nei campioni non trattati, mentre è stato efficacemente limitato dal trattamento di modificazione superficiale. Il trattamento di nitro-ossidazione in plasma ha inoltre prevenuto, almeno per i primi 8500 cicli di immersione, l'attacco corrosivo della superficie metallica, poiché lo strato superficiale ha agito da barriera all'aggressione dell'alluminio. L'apparato sperimentale per immersione ciclica in lega di alluminio fuso ha fornito risultati confrontabili con quelli di altre esperienze correntemente presenti in letteratura.

INTRODUCTION

Die casting is a high volume production process by which a molten alloy is injected under pressure into a die in order to produce high quality workpieces. Parts in zinc, magnesium and aluminium alloys are currently produced through this technique. Die casting of aluminium alloys is of particular interest for the automotive industry. Pieces as light as 0.5 kilograms, with many thin ribs as well pieces as heavy as tens of kilograms such as truck wheels, can be successfully die cast [1]. The service phases impose severe stresses on the moulds and it is generally estimated that two thirds of the downtime, during normal production is caused by die-related problems [2]. Furthermore, the initial tool costs constitute a remarkable part of the production costs and thus the economic success of die casting process strictly depends on die life [3]. Wear and failure of die casting dies involve a complex interaction between various mechanisms. The most important wear and failure modes are summarized as follows [4-6]:

(i) The so-called washout damages on working die surfaces are attributed to erosion, corrosion and soldering. They are the results of the motion of the liquid aluminium alloy, the exposure of the die surface to the liquid aluminium and the solidification and ejection of the casting. Erosion involves the washing away of die material by the impinging jet of a molten metal stream. Corrosion is caused by the fact that iron and most of the alloying elements in the die steel are more or less dissolvable in liquid aluminium. Furthermore, corrosion wear originates from dissolution of the tool material into the liquid metal and formation of intermetallic phases. For aluminium alloys, it may be seen as small protuberances of Al-Fe intermetallic compounds on the die wall [7, 8]. In addition, the high pouring temperatures may cause oxidation of the die surface. Soldering of the casting to the steel surface inside the cavity takes place during solidification. The soldering phenomena can be classified as physico-chemical, mechanical and mixed soldering. It is strictly related to the temperature, the filling pressure, the surface roughness and the number of shots (as the operating stresses influence the surface roughness) [9]. This causes on the one hand sticking problems when the casting is ejected. On the other hand, it can also give rise to adhesive wear when the casting is separated from the die [8].

(ii) Thermal fatigue is the most important failure mode in die casting. Thermal fatigue cracks may be classified by their appearance in heat checks and stress cracks. The characteristic feature of heat checking is a net-shaped crack pattern which occurs predominantly on level surfaces. Stress cracks mainly appear as individual and clearly pronounced cracks as a result of stress concentrations due to the configuration of the die cavity. Generally, thermal fatigue cracks will already appear after a few thousand cycles or even earlier, i.e. in the low cycle fatigue regime. The cracks may be sometimes filled with liquid alloy.

Strongly damaged surfaces led to the end of tools lifetime and soldering of aluminium on the tools can cause high service costs. The adhered aluminium has to be removed mechanically, and after some time, the die cannot be used any further [10]. The attention and the continuous monitoring of dies conditions and performances constitute an important task of the recent industrial policies for internal cost savings, developed by the major automotive producers [11, 12].

To lessen the detrimental effects of gross cracking, heat checking and washout material, toughness and tempering resistance are maximised [13]. Furthermore, the open literature had widely demonstrated the potential benefits of surface modifications. In particular, it was clearly demonstrated that ion nitriding improves significantly the resistance to soldering and dissolution through the reduction of wettability and adhesion [8, 13, 14]. Another benefit of applying nitriding could be the usual compressive stress state which might increase the thermal fatigue limit of near-surface zones of the die [15, 16]. As a further improvement, evidence from production shows that the presence of an oxide layer or lubricant layer reduces the occurrence of adhesion. Actually an oxide layer is naturally present on the die surface due to the oxidation of the die steel during heating and water quenching (steam oxidation) [17]. However, a very efficient protection method is to artificially perform this oxidation onto a nitrided steel surface. This can further improve the corrosion resistance of nitrided steel components. A thin oxide film of Fe_3O_4, having usually a thickness between ~0.2 and ~2 mm, can be produced on the top of the nitride layer using various thermochemical techniques, from salt bath, to steam, gaseous or plasma oxidation. Plasma systems are particularly attractive, since there is the opportunity to perform the nitriding and post-oxidising treatment in a single technological operation by changing the treatment gas mixture, temperature and time [18]. Moreover, the plasma process can improve the quality of interface properties between the oxide film and substrate and also enhance the conformal coverage [19]. The formation of the oxide layer is greatly influenced by the outer porosity of the compound layer, typical of nitrided surface. It has been shown that with an open pore structure the oxygen penetrates deep into to the compound layer and the pores can be totally covered and filled by the oxide.

The paper reports the status of an ongoing research of nitriding and post-oxidising influence on the thermal fatigue and washout resistance of a vacuum heat treated high quality AISI H11, which is one of the typical tool steel for die casting. Impact specimens made from this steel were fabricated and tested in a special equipment that performs a cyclic heating and cooling with an immersion in molten aluminium alloy bath. Although several experiences are present in the open literature using similar thermal fatigue equipments [8, 20, 21, 22], none of them had foreseen the use of specimens that can also be used for the impact test. On the contrary, in this research a direct evaluation of impact toughness modification could be developed. Furthermore, few data are available on the behaviour of plasma nitrided and post oxidised treatment in thermal fatigue experiments and, even to a less extent, in cyclic immersion in molten aluminium alloy experiments.

The assessment of damages on the cycled specimens was performed through optical and SEM analysis of both surfaces and transverse sections, so as to analyse the formation of thermal fatigue cracks and the presence, if any, of soldering and corrosion pits. Furthermore, the impact toughness and microhardness profiles of cycled and not cycled specimens were assessed to detect any modification of material basic properties after the cyclic immersion programme.

2. METHOD

2.1 MATERIALS

The focus of the research is a critical analysis of the efficiency of nitriding and post-oxidising treatment applied on a tool steel for die casting tools, in terms of protection from soldering and corrosion and of heat checking prevention.

A parent block was cut from an annealed AISI H11 hot rolled bar, produced through vacuum melting and remelting processes (see table 1 for the chemical composition). The cut was performed normally to the longitudinal orientation, so that the block was 65 mm long (figure 1a). A test coupon was then machined in a central position of the block (figure 1b). This test coupon was then vacuum heat treated (figure 1c) according to the following parameters: austenitizing at 1,000 °C – quenching with 5 bar nitrogen flow - first tempering at 550 °C – second and third tempering

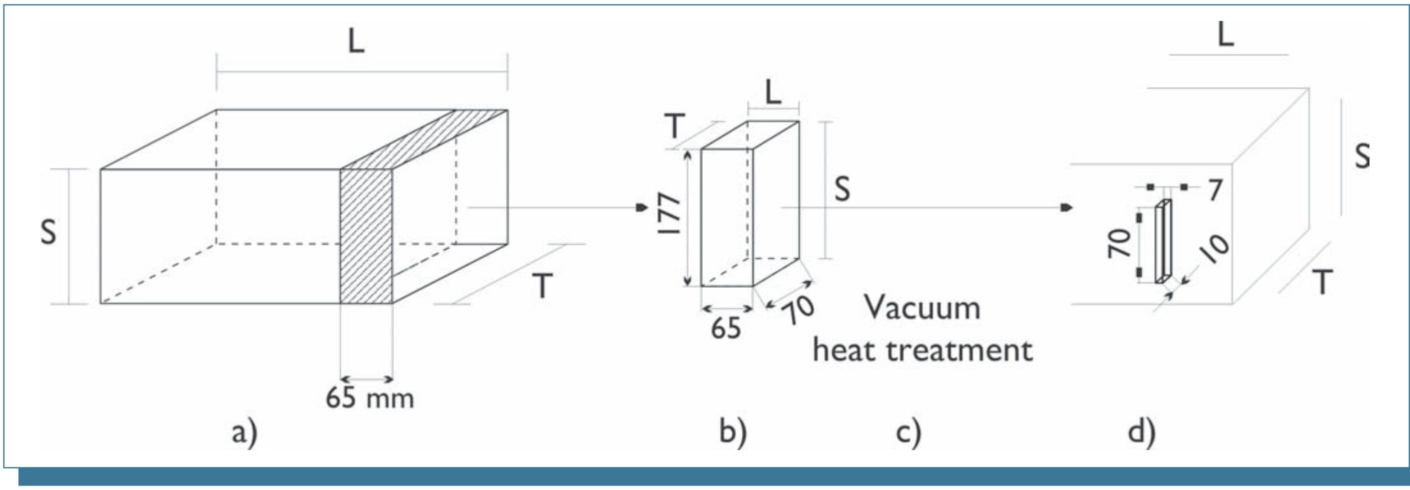


Fig. 1: Schematic of the test coupon removal and specimens preparation from the H11 parent block

at 595 °C to about 45.5 HRC, which is rather typical for hot work tool steels. Finally, unnotched impact specimens (section: 7 x 10 mm) were fabricated from the heat treated test coupon (figure 1d). The specimens were produced through a first gross electro-discharge machining followed by mechanical finishing. The impact specimens were removed from the test coupon according to the short transverse orientation and with the 7 mm dimension in the long transverse section (figure 1d). Actually, the specimens used for this research were initially 70 mm long instead of the classical 55 mm of impact specimens. This was necessary to hold the specimens in the testing rig for cyclic immersion in molten aluminium, as described below.

The first half of the specimens set was maintained in the heat treated state, whereas the other half was subjected to the so-called T-OXI surface treatment (combined process of plasma nitriding and oxidising with controlled potential). In particular, the specimens were plasma nitrided at a temperature of 500 - 580 °C and then oxidized in the same vacuum/plasma chamber. The important factor of the process used lies on the fact that the oxidation is controlled to avoid Fe_2O_3 and produce pure magnetite (Fe_3O_4) which is highly corrosion resistant. Generally, the oxidation period is between 30 and 60 minutes depending on the required thickness of the magnetite layer. The nitrided layer was characterised through optical and electron microscopy, to study the morphology of the surface modified layers, and by X-ray diffraction, to analyse the phase constitution of the layers. The thin layer mode with a 10 ° incident angle was used in the XRD analysis, so as to isolate the diffraction pattern of the very top layers.

After the specimen production, two series of specimens, five heat treated and five nitrided and oxidised, were directly tested with a 450 J Charpy pendulum. The remaining specimens were firstly subjected to a programme of cyclic immersion in molten aluminium (as described below) and then subjected to the impact test.

TABLE 1. CHEMICAL COMPOSITION OF H11; WT%

C	Si	Mn	Cr	Mo	V	P	S
0.377	0.191	0.193	4.930	1.20	0.415	0.006	0.001

2.2 APPARATUS FOR CYCLIC IMMERSION IN MOLTEN ALUMINIUM

Figure 2 shows the testing apparatus developed to simulate thermal cycling conditions that occur at the surface of a high pressure die casting. Four specimens could be mounted simultaneously on the test rig. At the same time, two test specimens were immersed into molten aluminium at 680 °C and the other two were immersed into the cooling bath at 36 °C. Then, the two arms were lifted and the situation was reversed. During the rotation of the arms the specimens passed through a gate made of metal brushes, that helped to remove the solidified aluminium film formed during the extraction from the melt.

The aluminium alloy was AlSi8Cu3Fe, commonly used for die casting, and it was maintained in the

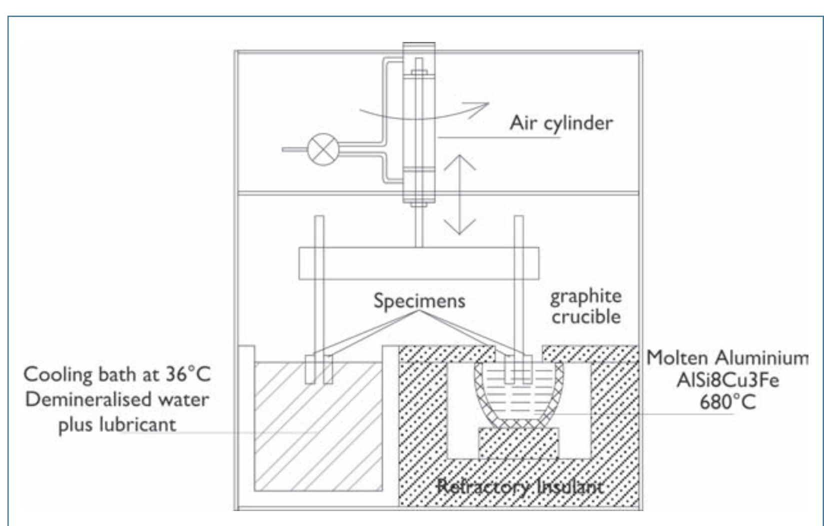


Fig. 2: Cyclic immersion in molten aluminium alloy test rig

molten state into a graphite crucible. The cooling bath was constituted of a diluted solution (1:80) of die lubricant, Metalcote 1973, in demineralised water. The duration of a typical cycle was 30s, with an actual immersion time of 4s for both the aluminium alloy and cooling baths. The immersion time was imposed on the base of a set up experiment, in which a hole was drilled for half of a testimony specimen, 2 mm inside the external skin (close to an edge). In this hole, a thermocouple was placed and a controlled cycling program was performed, modifying the immersion time. The final 4s immersion time was selected because by this way the heating and cooling cycle experienced by the test specimen was measured to range between 100 and ca. 520 °C. A typical die casting cycle measured in actual Teksid production plants ranges between 100 and 480 °C, on the external surface of the die. Therefore, the experimental conditions developed were more critical as the aforementioned temperature limits were suffered by the steel layers placed 2 mm from the skin. This means that specimen surface was subjected to a more severe deterioration cycle, so as to accelerate the degradation mechanisms.

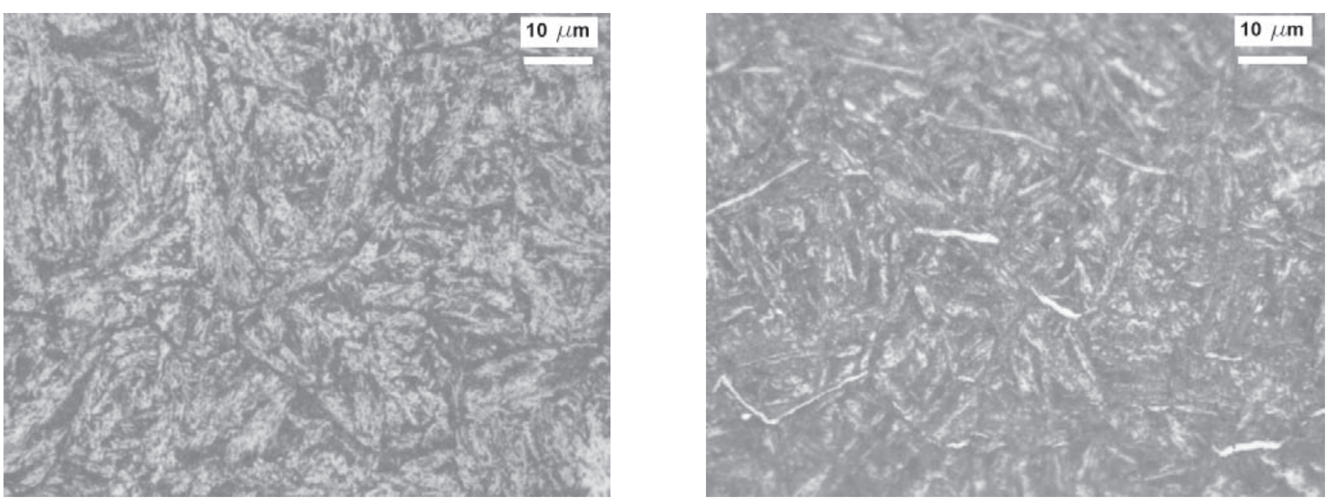
The tests were carried out up to 5,000, 8,500 and 15,000 cycles, both on simply heat treated and nitrided and post oxidised specimens.

During the test, an oxide layer formed on the specimens surface and partially covered the damaging features. It was therefore necessary to develop a specific cleaning procedure to chemically remove this external layer, without producing surface aggressions. Firstly, an immersion in boiling NaOH saturated solution for ca. 10 min was performed. The actual duration of this step is strictly dependant on the cleaning level. Then, the specimens were immersed for 1 min in a 10%vol HCl solution and, finally, for 1 minutes in boiling distilled water. These last two steps of immersion were necessary to stop the NaOH reaction and to safely eliminate the chlorides from specimens surface, respectively.

A periodical inspection of the specimens surface through optical and electron microscopy was performed.

After the cyclic immersion testing programme, the final part of the specimen (where the specimen is clamped onto the apparatus) was cut off, so as to obtain the classical 55 mm long impact testing specimen. Then all the cycled specimens, both heat treated and nitrided-oxidised, were impact tested.

Finally, the transverse sections of all of the specimens cycled were investigated to asses the penetration depth of damaging features, the microstructural and microhardness modifications.



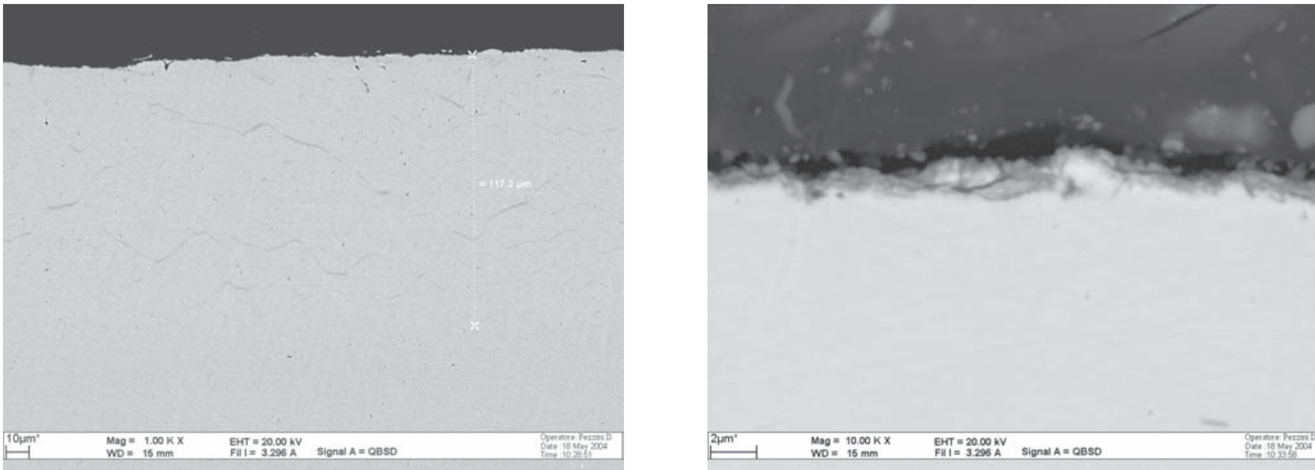
a) b)

Fig. 3: Metallographic observation of (a) an heat treated specimen (core structure on a transverse section) and of (b) a nitrided one (diffusion layer). Nital 3% Etching. LOM

3. RESULTS AND DISCUSSION

Figure 3 reports the microstructural observations of simply heat treated and nitrided-oxidised specimens. The heat treated specimen (figure 3a) exhibited a fine tempered martensite. The nitrided one (figure 3b) exhibited a similar microstructure mixed with small and uniformly distributed nitride precipitates.

Figure 4 reports two SEM observations of a nitrided specimen. The surface layers are constituted by a top layer, ca. 10 μm thick, and a diffusion layer, ca. 100 μm thick. Actually the top layer has a double structure. The nitriding stage of the surface treatment resulted in the development of a porous compound layer with a thickness of around 8 μm. The following oxidising stage provided the formation of an iron oxide layer, ca. 2 μm thick (figure 4b), which grew within the porous structure so as to close the surface



a) b)

Fig. 4: SEM/BSE images of a nitrided specimen. The left image (a) shows the formation of few nitride precipitates in the diffusion layer; the right image (b) clearly demonstrates the mechanism of the oxide formation that interpenetrates the porous compound layer

porosities and achieve an high mechanical keying with the underlying compound layer. The X-ray diffraction pattern (figure 5) helped to clarify that the oxide layer was constituted by magnetite (Fe_3O_4) and the compound layer was mainly constituted by γ' phase (Fe_4N) with a few content of ε phase ($Fe_{2\text{-}3}N$).

The macro-hardness of heat treated specimens was measured. The recorded average values was 44 HRC. The Vickers microhardness profiles of both simply heat treated and nitrided + oxidised specimens are presented and discussed below, with a comparison between the specimens in the delivery state and after the cyclic immersion in molten aluminium alloy.

The specimens subjected to cyclic immersion in molten aluminium alloy were periodically observed. The first signs of damaging mechanisms could be detected just after 5,000 cycles both on the simply heat treated and on the nitrided+oxidised specimens.

After 5,000 cycles, on heat-treated specimens (figure 6) two different damaging modes could be detected: (i) localised soldering points had occurred and eventually the cast metal sticked to these points and removed also part of the steel causing the formation of a surface cavity (figure 6a); (ii) the cyclic application of thermal strains led to the formation of a surface crack network, referred as heat checks (figure 6b). The average dimensions of the surface cavities due to soldering effect were observed to be considerably higher than those of the heat checks, leading to the conclusion that soldering in the case of the heat treated specimens was the predominant damaging mechanism.

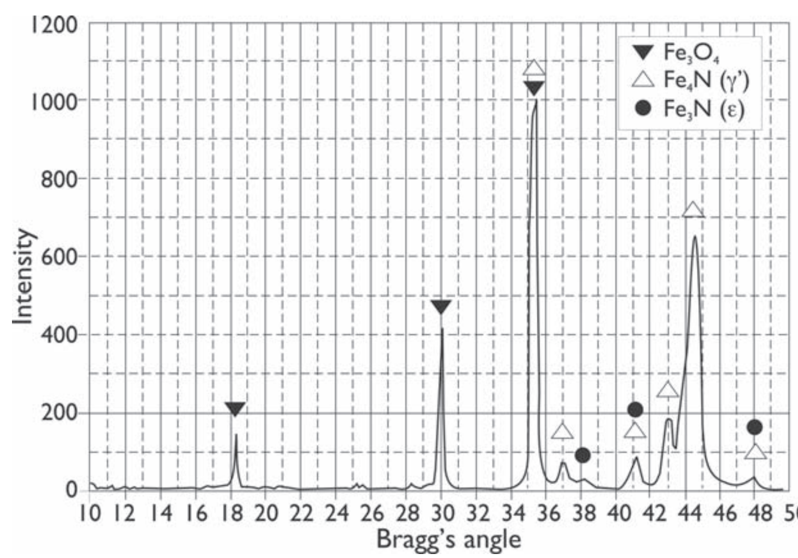
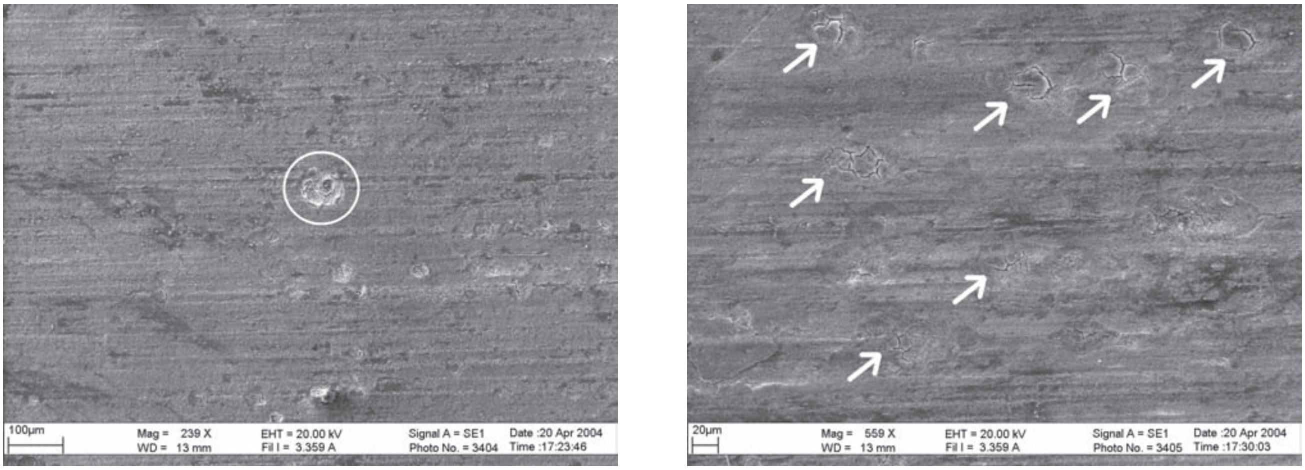
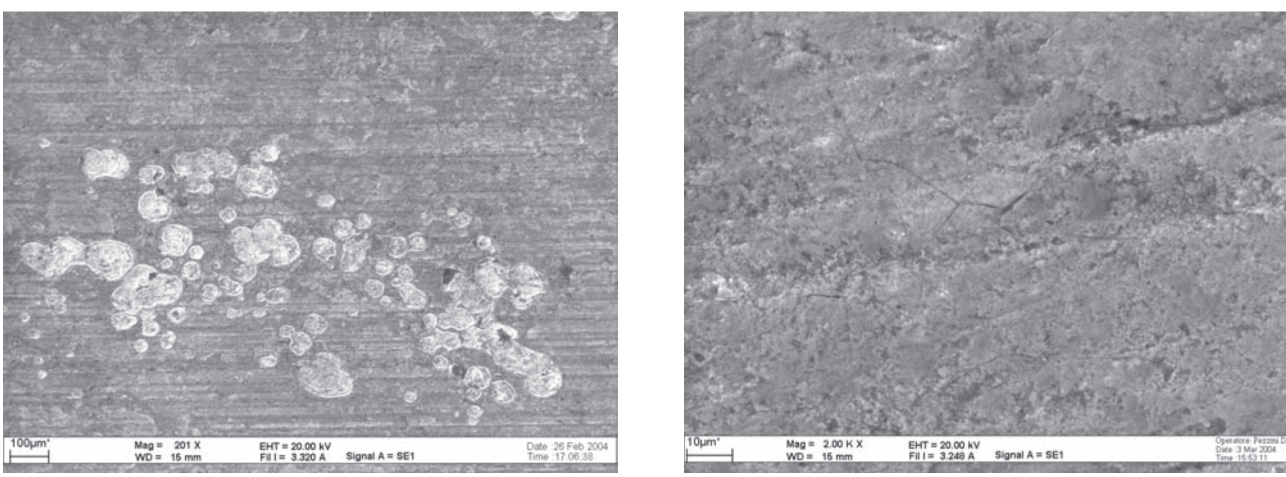


Fig. 5: XRD analysis of the top layer (iron oxide+compound layer) of a plasma nitrided+oxidised specimen: radiation Cu-Ka, incident angle 10¡ (thin layer mode)



a) b)

Fig. 6: Damaging mechanisms signs on simply heat treated specimens after 5,000 cycles in the cyclic immersion apparatus: evidence of (a) surface cavity due to soldering (white circle) and of (b) heat checks due thermal fatigue (white arrows)



a) b)

Fig. 7: Surface damages detected on simply heat treated surfaces after (a) 8,500 cycles and (b) 15,000 cycles. The average lateral dimensions of surface cavities were found to be really higher than the heat checks

With the increase of immersion cycles both soldering and heat checking became more evident on the simply heat treated specimens surface. In particular, as for the surface cavities formation, it was found to be developed to a great extent just after 8,500 cycles (figure 7a). After 15,000 cycles the cavities were found to be even larger and highly present on the surface. On the other hand, the average lateral dimensions of the heat checks were found to be limited to few microns also after 15,000 cycles (figure 7b).
The observations on the nitrided+oxidised specimens revealed again two different damaging modes: (i) the first mode was still the heat checks, typical in the flat surface which do not have any stress concentration; (ii) the second mode was corner cracking, that wasn't detected in the simply heat treated specimens. Corners or edges are more susceptible to cracking. At corners, cracking can take place in two directions (figure 8): (i) perpendicular to the edge because of cyclic strain in direction 1 and (ii) along the edge (dotted line) because of cyclic strain in direction 2 [23].

The heat checks were highly observed whereas only few but long corner cracks were detected on the nitrided+oxidised specimens. Figure 9 reports two examples of heat checks and corner cracks detected on the nitrided+oxidised. The onset of both damaging mechanisms was recorded already after 5,000 cycles. Both of these damages just increased in lateral dimensions and density with the number of immersion cycles. The heat checks revealed on the nitrided+oxidised specimens were very similar to those observed on the simply heat treated specimens. The presence of some long corner cracks was observed only on the nitrided+oxidised specimens and this is probably due to the high residual stresses induced by the surface modification treatment, that may favour the stress concentration along the corner. On the other hand, the composite top layer, provided by the surface modification treatment, acted as a barrier to the soldering of aluminium alloy on the steel. This avoided the formation of the typical surface cavities that were detected on the simply heat treated specimens.

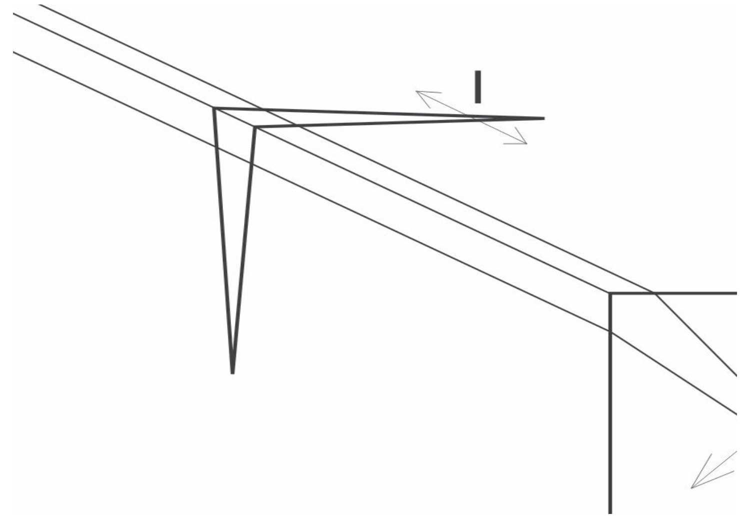
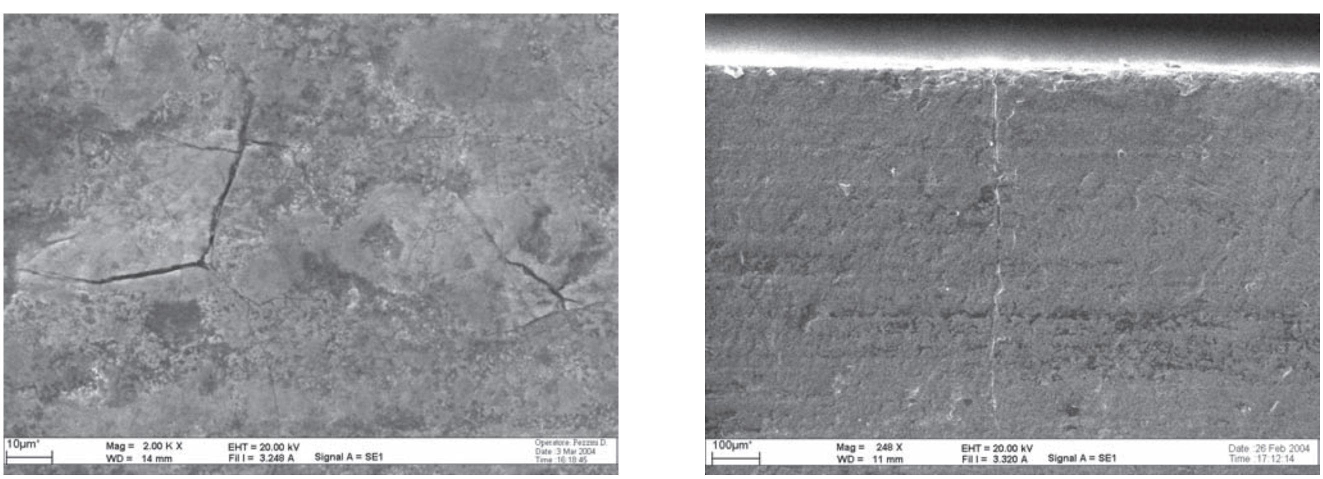


Fig. 8: Schematic of crack direction and stress direction [23]



a) b)

Fig. 9: Surface damages detected on the surface of nitrided+oxidised specimens after 8,500 immersion cycles: (a) heat checks; (b) corner crack

Starting from the observations performed after 8,500 immersion cycles, a localised oxide exfoliation was detected (figure 10). This resulted in the progressive breaking of the barrier to the melt sticking. Between 8,500 and 15,000 cycles this effect was observed to dramatically enhance leading to the formation of surface cavities very similar to those detected on the simply heat treated specimens. A simple remark could further show that from 8,500 to 15,000 cycles the damaging mechanism had changed: the cleaning procedure, which is necessary for the specimen surface observation, was very simple for the specimens subjected to 5,000 and 8,500 cycles, whereas it occurred to be very hard for the specimens subjected to 15,000 cycles. This was due to the large amount of soldering points formed in this long test step. An intermediate analysis will be indeed necessary to better evaluate the actual threshold in the number of immersion cycles prior to the onset of soldering effect also on nitrided+oxidised specimens.

After the completion of the cyclic immersion test, the all of the specimens was subjected to the impact test, to compare their behaviour with that of

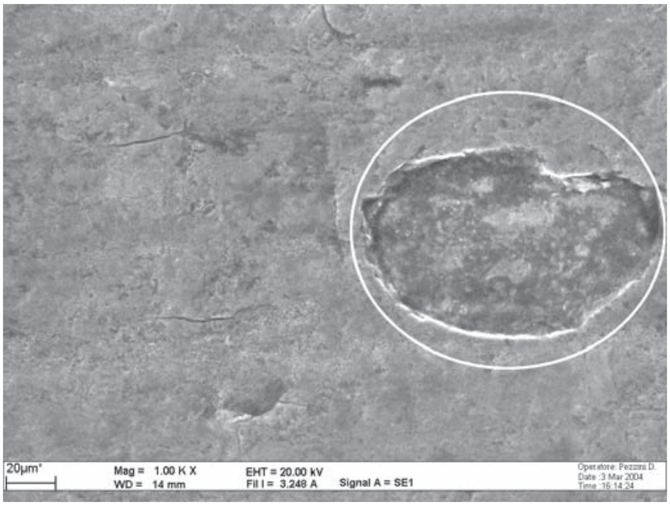


Fig. 10: Oxide exfoliation (white circle) observed on the surface of nitrided+oxidised specimens after 8,500 immersion cycles

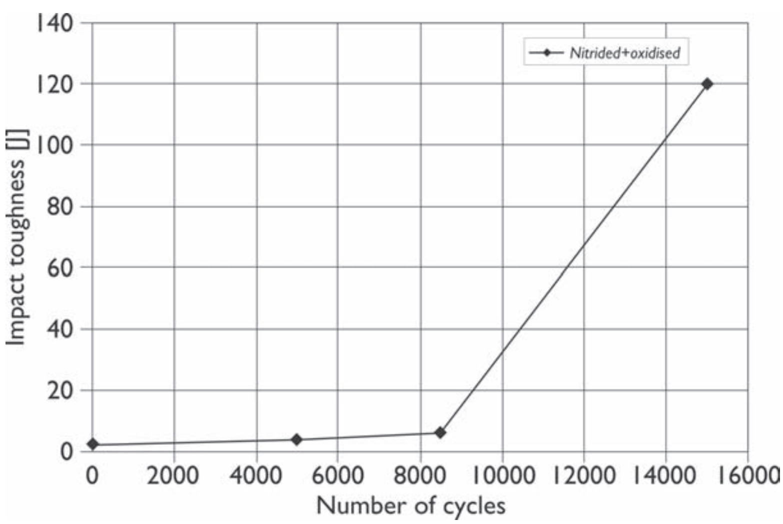


Fig. 11: Impact toughness modification along with the increasing number of immersion cycles (recorded impact toughness vs number of immersion cycles)

the specimens that were maintained in the delivery state (fresh simply heat treated or nitrided+oxidised).

The average impact toughness recorded on the heat treated specimens in the delivery state was 421 J. The heat treated specimens subjected to 5,000, 8,500 and 15,000 immersion cycles never broke in the impact test and thus the recorded impact toughness has to be considered as exceeding 450 J (maximum capacity of the pendulum).

As for the nitrided+oxidised specimens, it could be possible to draw an actual trend with the increasing number of immersion cycles (figure 11). The impact toughness levels recorded on the nitrided+oxidised specimens were very lower if compared with the heat treated ones, due to the embrittlement induced by surface modified layers.

A clear softening effect was recorded by both heat treated and nitrided-oxidised specimens. The metallographic observation didn't revealed any significant microstructural modification due to the cyclic immersion.

On the contrary, a clear loss in hardness (figure 12) was recorded as a direct consequence of material softening. This is clearly detrimental as it provides a lowering of the plastic limit, andplastic deformation in materials of lower hardness occurs sooner. Higher hardness prevents plastic deformation and guarantees that the materials operating performances can be maintained within the elastic region. This results in a considerably higher number of cycles to failure of the die [21]. Although a reduction in surface hardness was recorded also in the nitrided+oxidised specimens, the here reported measurements demonstrate that the surface modified steels can maintain a considerably high hardness level within the first

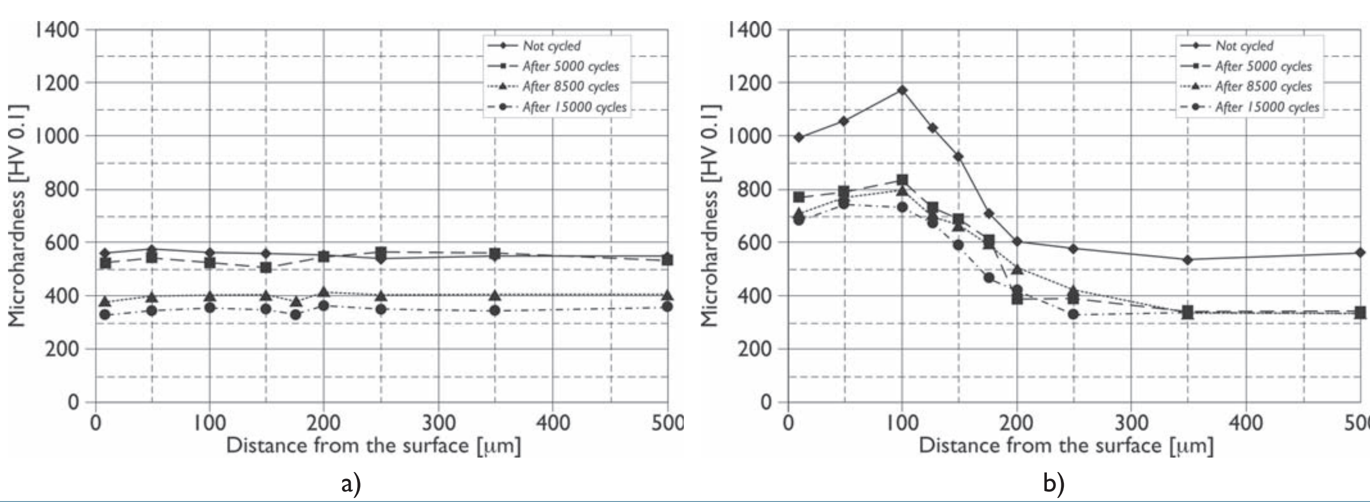
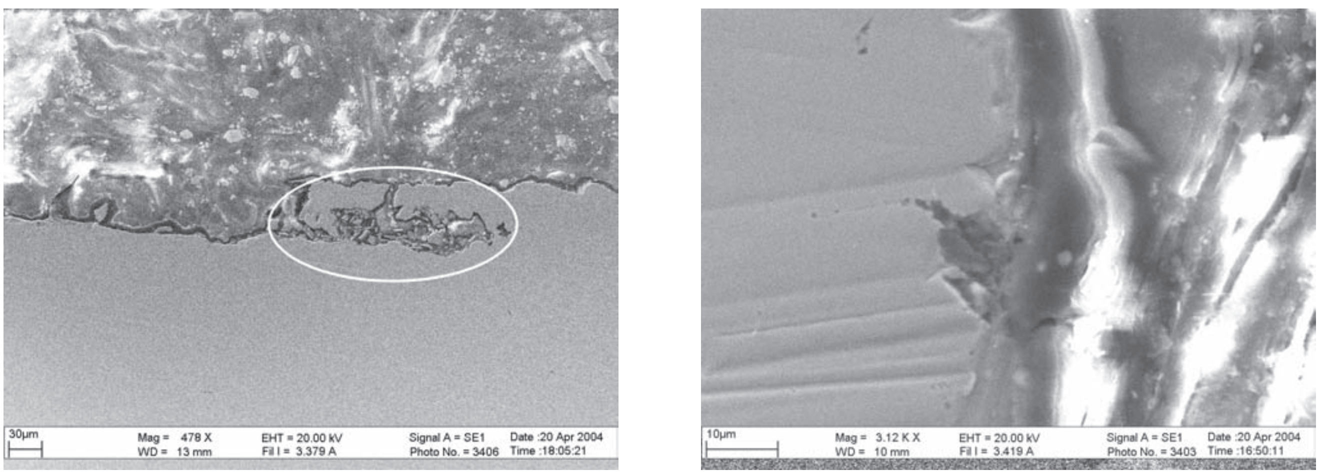


Fig. 12: Microhardness profiles of (a) heat treated and (b) nitrided+oxidised specimens, before and after the cyclic immersion in molten aluminium alloy

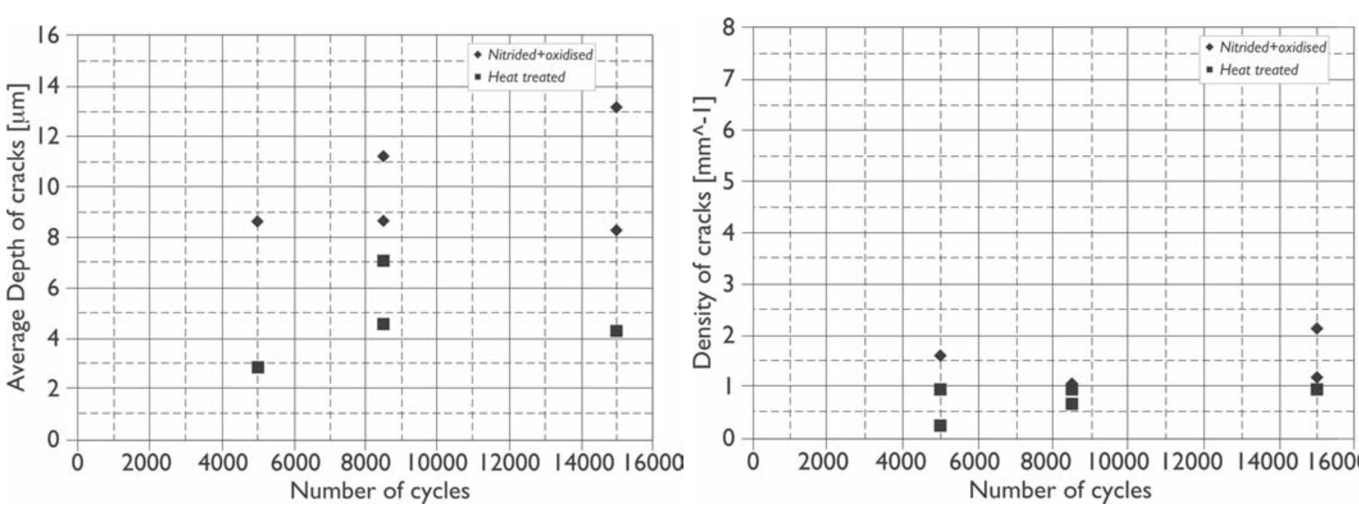
100 μm from the external surface (between 700 and 800 HV also after 15,000 immersion cycles). On the contrary, on the simply heat treated H11 steel the hardness loss due to the cyclic immersion resulted in very poor hardness levels, from the surface to the core of the steel (less than 400 HV just after 8,500 cycles). Therefore, during exposure to thermal fatigue conditions the surface of a plasma nitrided+oxidised steel can continue to withstand severe mechanical stress without going into the plastic deformation regime. The experimental conditions are indeed accelerated if compared with the normal service conditions of die casting, but the amount of softening can be clearly limited by the proposed surface modification treatment.

After the impact test, a deeper analysis of the cracks and cavities generated by the cyclic immersion in molten aluminium alloy was performed on the transverse section of the specimens. Figure 13 reports the main damaging signs (soldering and heat checks) detected on the two types of specimens. Both the phenomena were found to be limited to the very superficial layers (between 10 and 30 μm depth). The soldering phenomenon is due to the high affinity that aluminium has for iron. This provides a vigorous physio-chemical reaction at the steel/melt interface that results in the formation of a series of iron-aluminium-silicon intermetallic compounds over the steel surface (white circle in figure 13a) [22]. On the same transverse sections, an evaluation with the optical microscope and image analysis of both cracks and cavities depth and distribution was performed. A statistical treatment was performed analysing three different portions of the transverse section for each specimen. Although a higher number of measurements would have



a) b)

Fig. 13: SEM/SE images (transverse section) of the damages due to the cyclic immersion in molten aluminium alloy: (a) soldering (white circle) on a heat treated specimens after 8,500 cycles; (b) cracks on a nitrided+oxidised specimen after 15,000 cycles



a) b)

Fig. 14: Heat checks cracks analysis: (a) average depth of cracks vs number of cycles; (b) density of cracks vs number of cycles

to be collated to enhance the statistical analysis, a first impression of the cracks and cavities parameters could be derived (figure 14 ad 15). The density of cracks or cavities is calculated as the ratio between the number of cracks (cavities) and the length of the analysed surface portion.
The recorded average depth of both cracks (figure 14a) and cavities (figure 15a) was very limited (less than 14 μm), however those for the nitrided+oxidised specimens were slightly higher. The measured density of heat checks (figure 14b) was very low for both heat treated and nitrided+oxidised specimens. On the contrary, the density of cavities (figure 15b) was found to be slightly higher for the heat treated specimens and very higher if compared with the cracks densities.
These observations cannot be assumed as conclusive, but they are in good agreement with the qualitative analysis developed during the periodic inspections performed along with the cyclic immersion test and previously exposed.
However, the low penetration depth of cracks and cavities guarantees that the bulk steel was never detrimentally affected by the cyclic immersion in molten aluminium alloy. Furthermore, the results of impact test and microhardness evaluation actually showed that the softening effect was stronger than the onset of any superficial deterioration.

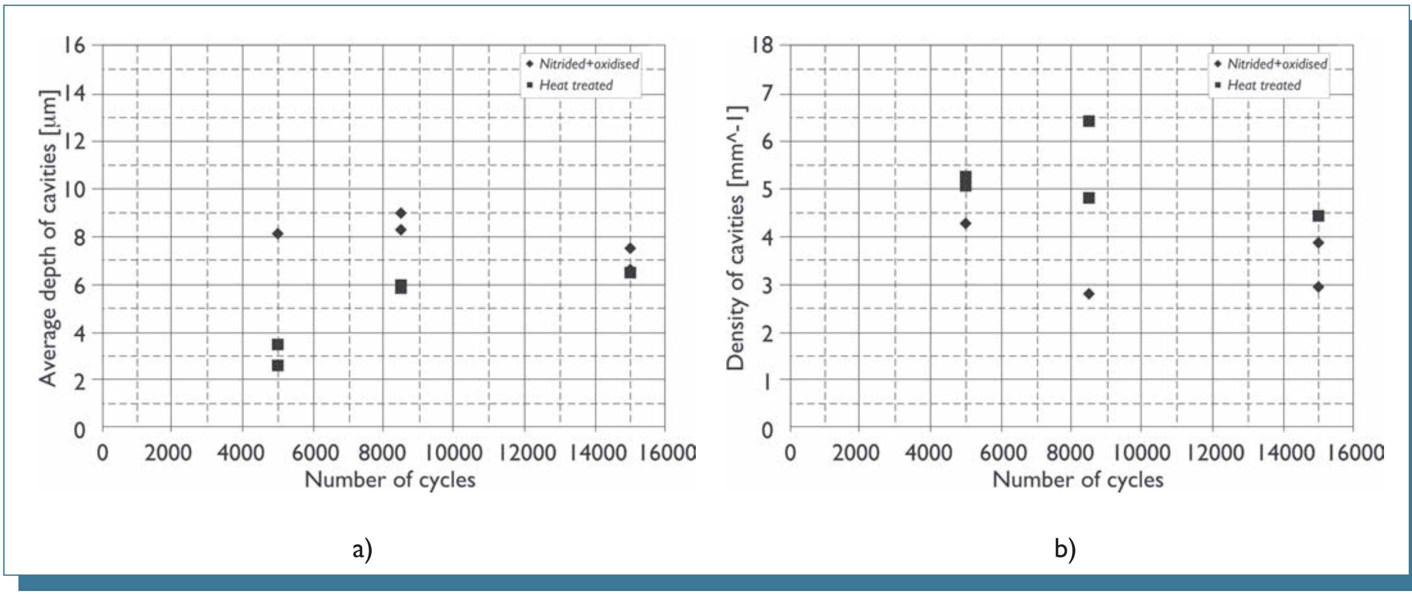


Fig. 15: Cavities analysis: (a) average depth of cavities vs number of cycles; (b) density of cavities vs number of cycles

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5. CONCLUSIONS

This paper reports the first part of a large research programme on thermal fatigue and adhesion wear effects in a cyclic immersion test in molten aluminium alloy. This laboratory test aims at simulating the die degradation mechanisms in light alloys die casting cycles. Simply heat treated and heat treated and nitrided oxidised specimens were tested with a specially developed procedure.
The simply heat treated surface, that has an high affinity with aluminium and silicon, preferentially exhibited adhesion wear. On the contrary, the hard top oxide in the nitrided oxidised specimens acted as a barrier against the soldering effect, but resulted to be prone to heat checking and corner cracks. However, all the damages resulted to be limited

to the surface and sub-surface layers. In the nitrided+oxidised specimens, the barrier provided by the top oxide-compound layer was found to be effective as far as the number of cycles reached 8,500 immersion cycles. Between 8,500 and 15,000 immersion cycles, the onset of an exfoliation effect occurred. This progressively broke the oxide barrier and left the underlying metallic surface free to be attacked by the melt.

The clearer effect which was detected was the softening, clearly visible through impact toughness increases and microhardness decreases with the number of immersion cycles. The softening effect lowers the surface and core hardness and detrimentally impacts on the service performance (i.e. producing plastic deformation on the surface that means end of the service). The plasma nitriding and post oxidising treatment had limited this lowering in hardness and had prevented also the metallic surface from corrosion attack (acting as a barrier to the aluminium aggression) at least for the first 8,500 cycles. The developed test rig provided results similar to other experiences currently present in the open literature. The temperatures recorded on the steel surface were similar to those measured on the real scale die casting dies.

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