

Microstructure, microhardness and impact toughness of low pressure carburized PM steels

E. Santuliana, C. Menapace, A. Molinari, G. Meli

The effect of low pressure carburizing (LPC) on two low alloyed sintered steels processed according to different routes to obtain different amounts of open porosity was studied. In presence of an open porosity, overcarburizing promotes the precipitation of grain boundary carbides in the Cr steel, and the formation of retained austenite in the Cr free one. In presence of a fully closed porosity LPC results in the formation of a homogeneous case. Even a slight amount of open porosity causes overcarburizing. In absence of overcarburizing, impact toughness is typical of a brittle behavior, as expected. However, an unexpected high toughness is measured in the supercarburized steel containing retained austenite.

Keywords:

steel, sintering, secondary treatments

INTRODUCTION

Low pressure carburizing (LPC) is increasingly used for case hardening of steel parts.^{1,2,3} The main advantage of this process, in comparison to conventional gas carburizing, is the absence of oxidizing agents in the atmosphere, which prevents oxidation of steel on heating⁴ and even during isothermal soaking at the carburizing temperature in the case of chromium steels.⁵ In LPC the carbon carrier gas is either acetylene or propane. These gases dissociate at high temperature (900-1000°C) and in vacuum (15mbar) and produce carbon and hydrogen (acetylene) or carbon, hydrogen and methane (propane).¹ Another advantage is the use of gas quenching instead of oil quenching, which results in an increase in the effective case depth and in the reduction of distortion of pieces⁶.

The Powder Metallurgy (PM) industry is looking at LPC with a growing interest for two main reasons. First, quenching oil remains entrapped in the open porosity, and has to be eliminated before tempering for environmental reasons. Second, chromium steels have been recently introduced in the market for the production of structural parts, and are increasingly used by part manufacturers. A carburizing process which does not use quenching oil and does not contain oxidizing agents in the atmosphere might represent a great opportunity to improve wear and fatigue properties of PM chromium steels. A criticism has however emerged, due to the unfavorable combination of the large surface area of porous PM parts on one side and to the very high carburizing potential of LPC on the other. The result of this combination is overcarburizing, which results in the precipitation of grain boundary carbides and the formation of retained austenite.^{7,8,9} To overcome such a limitation, surface densification by rolling is used to eliminate porosity in the surface layers.^{8,9} In this case, the microstructure of the carburized layers becomes homogeneously martensitic.

An alternative to rolling may be represented by shot peening,

which promotes the densification of the surface layers.¹⁰ Moreover, high density parts (over 7.3-7.4g/cm³) contain a prevailing closed residual porosity, then their surface area is comparable to that of fully dense ones. These two alternatives solutions were investigated in the present work to prevent overcarburizing. Specimens were produced by three processing routes:

- cold compaction and sintering, that results in a medium density with prevailing open porosity and shall represent the supercarburized reference;
- warm compaction and sintering, which results in a higher density with a mainly closed porosity;
- cold compaction, sintering and surface densification by shot peening.

Since the microstructure of the overcarburized case depends on the chemical composition of the steel, two different materials, widely used in the industrial production, were investigated: a Cr-Mo steel and a Mo-Ni-Cu steel. The former is expected to form carbides, the latter should form mainly retained austenite. The present work aims at evaluating the effect of different porosity in the surface layers on the microstructure and microhardness, and on the impact toughness of the carburized steels.

EXPERIMENTAL PROCEDURE

Table 1 reports the nominal composition of the powders (Höganäs AB, Sweden) used for the preparation of the specimens, along with the percent of added graphite. Charpy bars (55x10x10 mm) were produced by cold compaction and warm compaction, to obtain green densities around 6.8g/cm³ and 7.3g/cm³, respectively.

Sintering was carried out at 1120°C in a walking beam furnace with 30 minutes isothermal holding in a N₂/H₂ atmosphere. Density and porosity were determined according to ASTM B328 and are reported in Table 2.

	%Cr	%Mo	%Ni	%Cu	%C
ACrL	1.5	0.2			0.2
DLH		0.9	0.9	2	0.2

TAB. 1 Nominal composition (wt.%) of the powder investigated.

Composizione nominale (wt.%) delle polveri.

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Material	ρ [g/cm ³]	ϵ [%]	ϵ_{open} [%]
ACrL cold compacted	6.9±0.04	11.3	8.9
ACrL warm compacted	7.3±0.01	7.5	0.3
DLH cold compacted	6.9±0.06	14.3	13.2
DLH warm compacted	7.3±0.03	7.8	4.3

TAB. 2 Density (ρ), total porosity (ϵ) and open porosity (ϵ_{open}).

Proprietà degli acciai sinterizzati: densità (ρ), porosità totale (ϵ) e porosità aperta (ϵ_{open}).

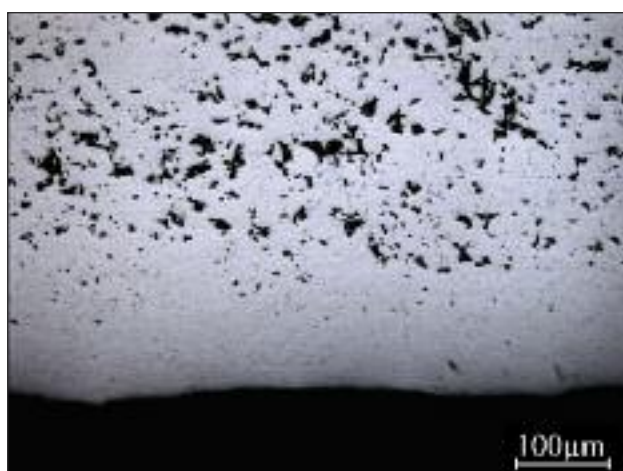


FIG. 1 Microstructure of the shot peened ACrL with the surface densified layer.

Microstruttura dell'acciaio ACrL dopo pallinatura.

As expected, porosity is mainly interconnected in cold compacted materials, whilst the warm compacted ones are significantly different. Despite of the same sintered density, porosity is practically closed in the Cr steel, whilst it is partially interconnected in the other one.

The cold compacted ad sintered specimens were shot peened, with the following parameters: intensity 12 Almen, shot ASH230, coverage 150%. Figure 1 shows, as an example, the microstructure of ACrL after shot peening. The treatment produces a full dense surface layer around 100 μ m thick, with only a few isolated micrometric pores.

Low pressure carburizing (LPC) was carried out in an industrial furnace. Two different treatments were investigated, characterized by three and five boost-and-diffusion cycles, respectively. The carburizing temperature was 945°C and gas was acetylene.

Treatment	Boost + diffusion time (minutes)				
	I	II	III	IV	V
LPC 1	2+15	1+1	1+4		
LPC 2	2+10	1+15	1+19	1+27	1+4

TAB. 3 LPC treatments.

Riassunto dei trattamenti di cementazione di bassa pressione.

Quenching was carried out with a nitrogen flux at 6 bar. Tempering at 180°C for 2 hours was carried out after LPC. The two treatments are described in Table 3.

The microstructural investigation of the samples was carried out by the Light Optical Microscope and the Scanning Electron Microscope, after the metallographic preparation and etching (Nital-Picral). X-Ray Diffractometry was used to investigate the microstructural constituents of the carburized layers, using CuK radiation.

The carbon content of the carburized layers was analyzed by a LECO analyzer. The average content of a 500 μ m thick surface slice was analyzed, to get an evaluation of the carbon pick-up after the treatment.

The HV0.1 microhardness profile was measured on all the specimens. Instrumented impact tests were carried out on three specimens of each material.

RESULTS AND DISCUSSION

Cold compacted materials

Figures 2 and 3 show the microstructure and the microhardness profile of the two materials after the LPC1 treatment. The microstructure of the carburized layer is inhomogeneous in both steels.

Figure 4 shows the extensive precipitation of proeutectoidic carbides at the prior austenitic grain boundary in ACrL. Carbides are recognized as chromium cementite by Kremel et al.⁷ Precipitation involves a surface thickness of around 100 μ m, where microhardness is lower than beneath.

Figure 5 shows the XRD spectrum, elaborated to obtain the quantitative analysis of the microstructural constituents of the outer layer of the carburized surface (approximately 5 μ m thin): martensite (46 %), cementite ((Fe, Cr)₃C, 49%), retained austenite (3%) and chromium carbide M₂₃C₆ (2%).

The microhardness profile of ACrL in Figure 2 is quite sharp, but microhardness is low than it could be expected, since grain boundary carbides do not provide a significant contribution to hardening, being precipitated at the grain boundary.

The Cr-free material does not reveal carbides in the metallographic analysis. It contains quite a large amount of retained austenite (Figure 6a), whose amount was quantified to 25% by XRD analysis (Figure 6b).

The microhardness profile (Fig. 3) is influenced by retained austenite, which is mainly localized in the outer part of the case where microhardness is lower than beneath. Microhardness is still not very high.

Both the grain boundary carbides in ACrL and the retained austenite in DLH are due to overcarburizing. In presence of chromium, carbon reacts forming chromium cementite.⁷ In absence of this element, carbon dissolves into austenite, decreasing Mf-Ms temperatures promoting the stabilization of austenite at room temperature after quenching. Both the results are undesirable from a practical viewpoint, since carbides cause brittleness and retained austenite can impair wear resistance and, if transformed to martensite by stress, dimensional precision. The positive effect of overcarburization on wear resistance has been reported in,¹¹ but in this case carbides are homogeneously distributed and not segregated at grain boundaries. The LPC2 treatment was not carried out on these specimens.

Warm compacted materials

Figures 7 and 8 show the microstructure and the microhardness profile of the two warm compacted materials after LPC1.

Figures 9 and 10 show the microstructure and the microhardness profile of the two warm compacted materials after LPC2. The microstructure of ACrL is homogeneous without grain bound-

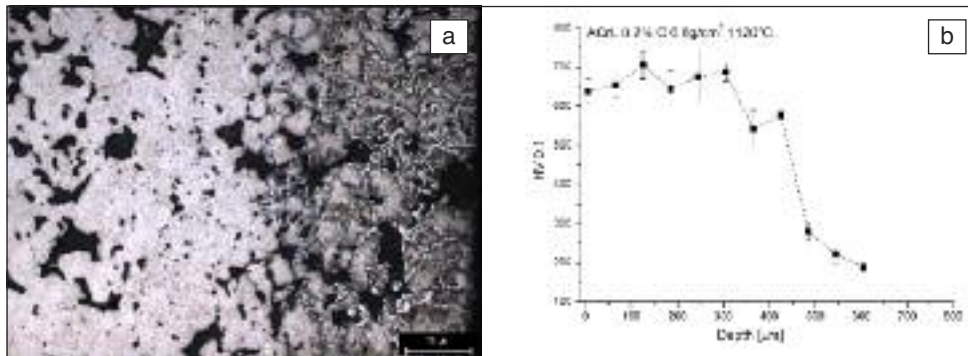


FIG. 2
Microstructure (a) and microhardness profile (b) of LPC1 carburized cold compacted ACrL

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio ACrL compattato a freddo e cementato con LPC1.

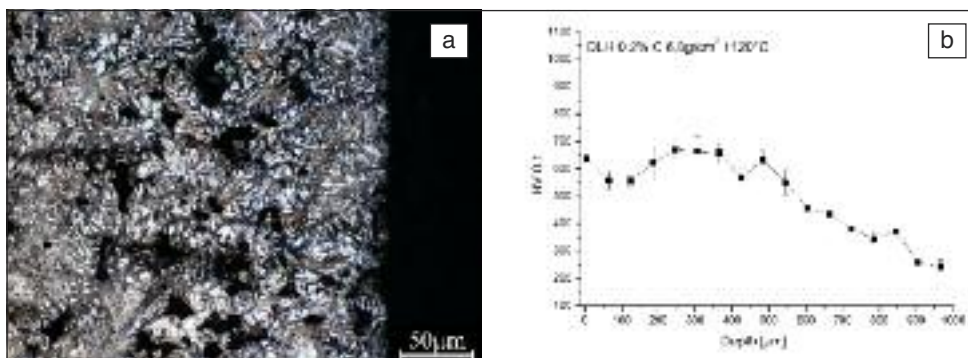


FIG. 3
Microstructure (a) and microhardness profile (b) of LPC1 carburized cold compacted DLH.

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio DLH compattato a freddo e cementato con LPC1.

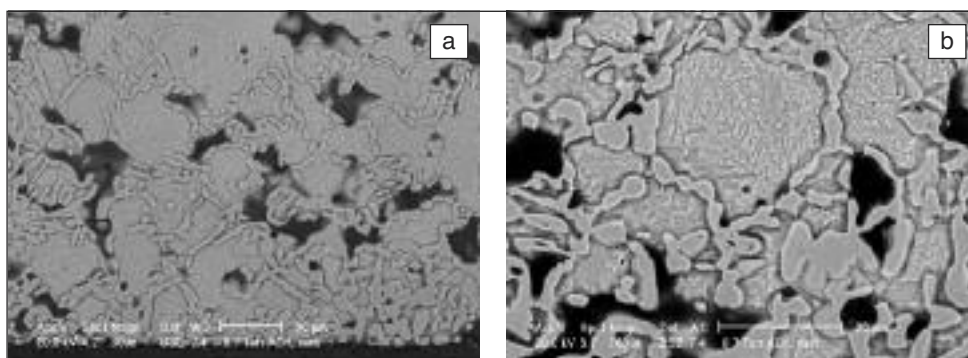


FIG. 4
Grain boundary precipitation in LPC1 carburized cold compacted ACrL.

Precipitazione a bordo grano nell'acciaio ACrL compattato a freddo e cementato con LPC1.

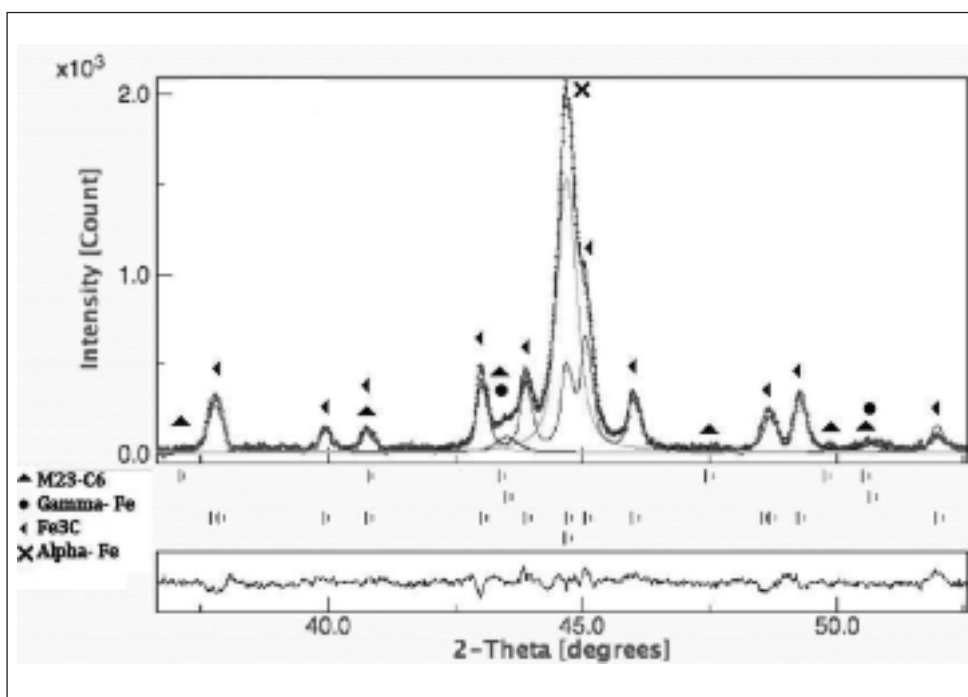


FIG. 5
XRD pattern of LPC1 cold compacted ACrL.

Spettro XRD dell'acciaio ACrL compattato a freddo e cementato con LPC1.

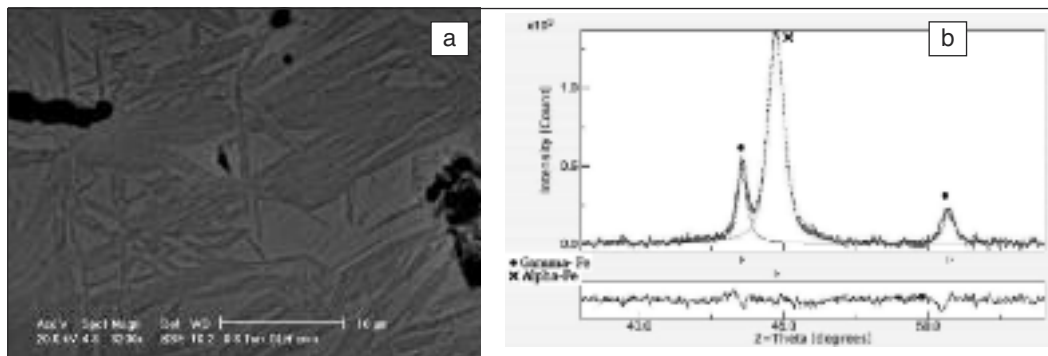


FIG. 6
a) SEM micrograph and b) XRD pattern of the case of LPC1 cold compacted DLH.

a) micrografia SEM e b) spettro XRD dello strato superficiale dell'acciaio DLH compattato a freddo e cementato con LPC1.

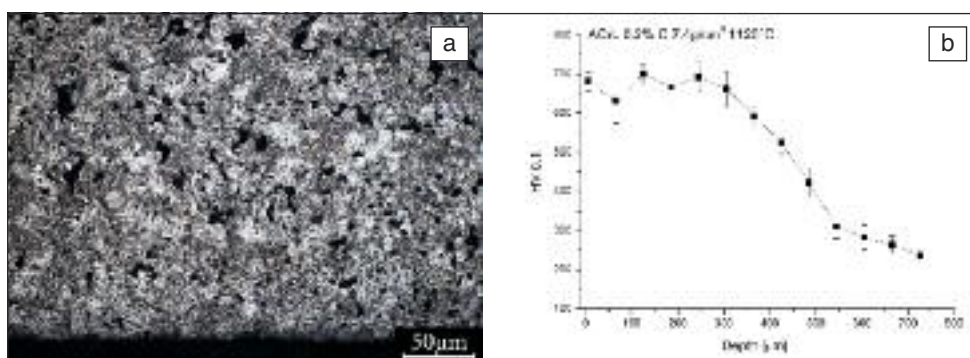


FIG. 7
Microstructure (a) and microhardness profile (b) of LPC1 warm compacted ACrL.

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio ACrL compattato a caldo e cementato con LPC1.

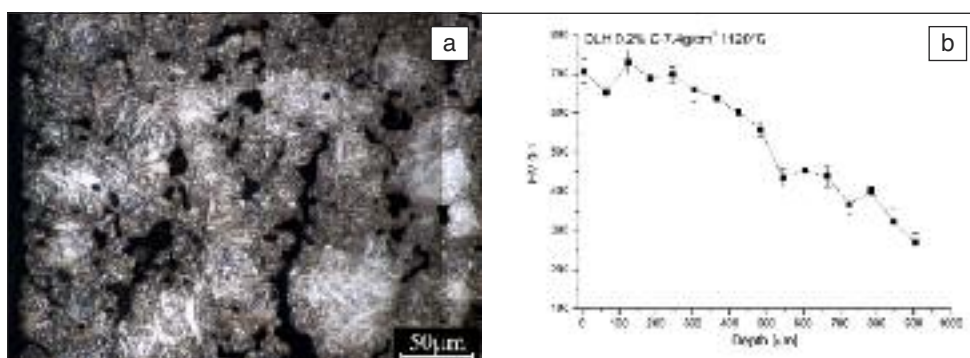


FIG. 8
Microstructure (a) and microhardness profile (b) of LPC1 warm compacted DLH.

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio DLH compattato a caldo e cementato con LPC1.

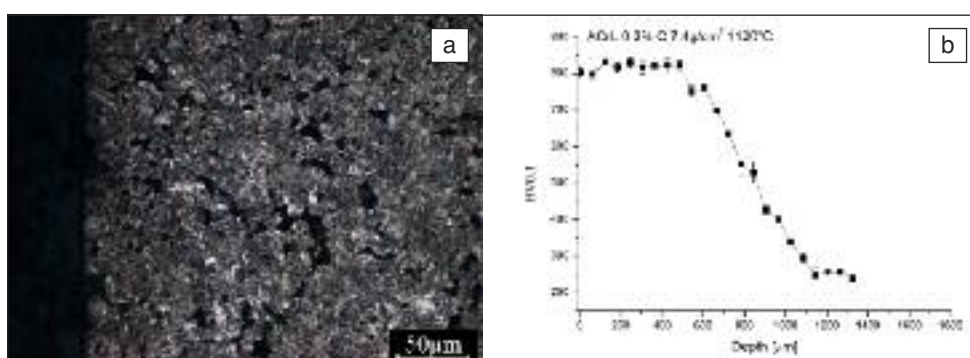


FIG. 9
Microstructure (a) and microhardness profile (b) of LPC2 warm compacted ACrL.

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio ACrL compattato a caldo e cementato con LPC2.

dary carbides. The microhardness profile is better after LPC2, microhardness being higher and depth greater than after LPC1. Contrarily, DLH contains retained austenite after both the treatments, and the microhardness profile is still irregular, with a slight decrease towards the surface.

The average carbon content is 0.44% in ACrL and 1% in DLH. The latter material, despite of warm compaction, is still overcarburized, whilst ACrL seems properly carburized and the absence of carbides is due to the low carbon pick-up. The different response of the two materials to the same carburizing processes is due to the different characteristics of porosity. Despite of

the same density, Table 2 shows that porosity is almost completely closed in ACrL, whilst there is still a significant amount of open porosity in DLH. In presence of 4.3% open porosity, the exchange surface area is significantly larger than the geometrical surface of the parts, and this causes supercarburizing.

The results are quite interesting from a practical viewpoint, since they reveal the role of porosity, given the density range obtained by warm compaction. In particular, the reference to density is not reliable, since two different materials having almost the same density may differ significantly in open porosity, resulting in quite different response to LPC.

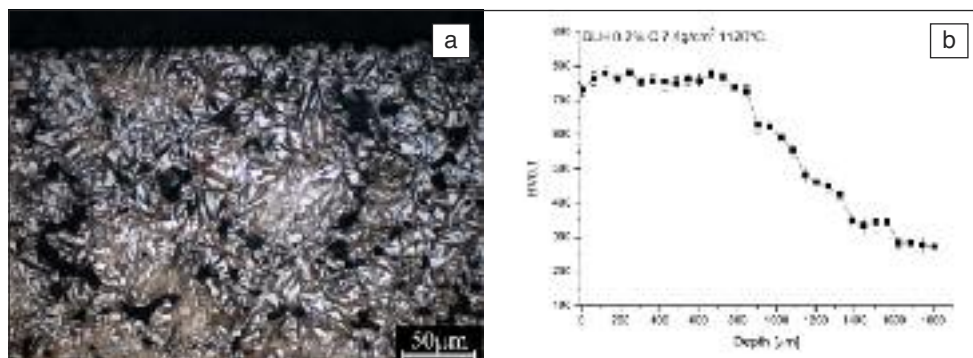


FIG. 10
Microstructure (a) and microhardness profile (b) of LPC2 warm compacted DLH.
Microstruttura (a) e profilo di microdurezza (b) dell'acciaio DLH compattato a caldo e cementato con LPC2.

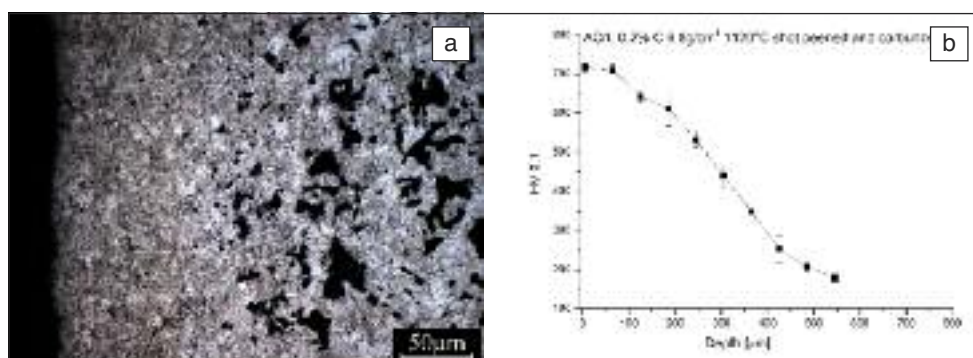


FIG. 11
Microstructure (a) and microhardness profile (b) of LPC1 shot peened ACrL.
Microstruttura (a) e profilo di microdurezza (b) dell'acciaio ACrL pallinato e cementato con LPC1.

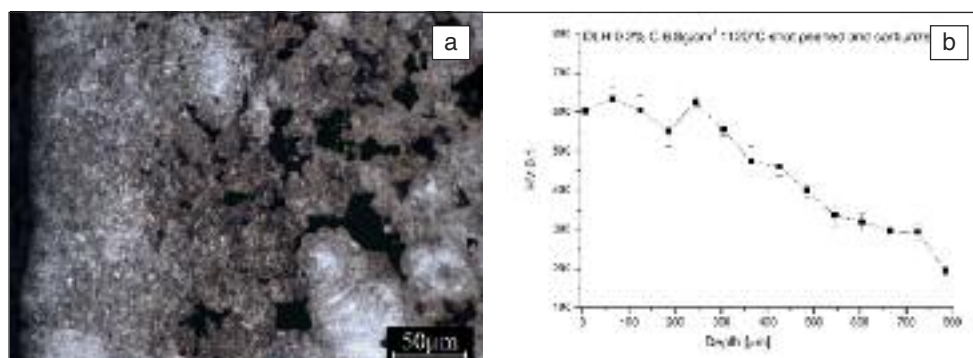


FIG. 12
Microstructure (a) and microhardness profile (b) of LPC1 shot peened DLH.
Microstruttura (a) e profilo di microdurezza (b) dell'acciaio DLH pallinato e cementato con LPC1.

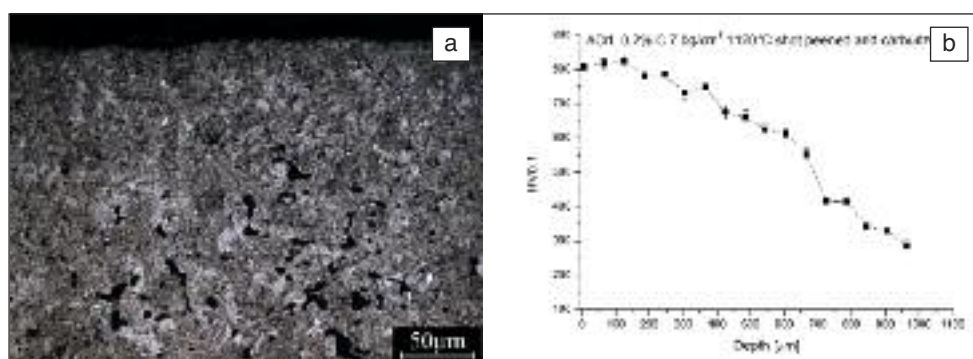


FIG. 13
Microstructure (a) and microhardness profile (b) of LPC2 shot peened ACrL.
Microstruttura (a) e profilo di microdurezza (b) dell'acciaio ACrL pallinato e cementato con LPC2.

Surface densified materials

Figures 11 and 12 show the microstructure and the microhardness profile of the two materials with densified surfaces, after LPC1.

Figures 13 and 14 show the microstructure and the microhardness profile of the two materials with densified surface, after LPC2.

The microstructure seems homogeneous in the two materials, neither carbides nor retained austenite is observed. The microhardness profiles are regular after both the treatments and surface hardening is more intense after LPC2.

The absence of porosity on the densified surface prevents overcarburizing. The average carbon content after LPC2 in the two materials is 0.41% and 0.49%, respectively.

Warm compaction vs. surface densification and the effect of base material

The comparison between materials with the densified surface and those produced by warm compaction demonstrates that only surface densification prevents overcarburizing in DLH, whilst in ACrL the two alternative processing routes lead to similar results.

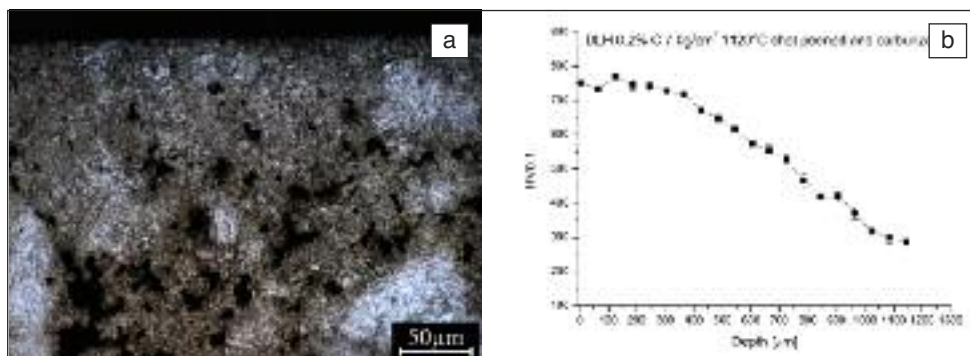


FIG. 14
Microstructure (a) and microhardness profile (b) of LPC2 shot peened DLH.

Microstruttura (a) e profilo di microdurezza (b) dell'acciaio DLH pallinato e cementato con LPC2.

Material	LPC 1			LPC 2		
	HV0.1 _{surf}	HV0.1 _{max}	d ₅₅₀ (μm)	HV0.1 _{surf}	HV0.1 _{max}	d ₅₅₀ (μm)
ACrL warm compacted	700	710	320	800	850	750
ACrL shot peened	690	690	200	800	830	650
DLH shot peened	600	660	200	760	770	700

TAB. 4
Representative parameters of the microhardness profile.

Parametri rappresentativi dei profili di cementazione.

Material	Processing route	Impact energy (J)			
		as sintered	carburized	as sintered	carburized
ACrL	warm compaction	72	13		
	cold compaction and shot peening			21	13
DLH	warm compaction	47	39		
	cold compaction and shot peening			11	16

TAB. 5
Charpy impact properties of as sintered and carburized materials.

Proprietà ad impatto degli acciai sinterizzati e cementati.

It may be interesting to compare the microhardness profiles obtained with the two alternative routes in ACrL. Table 4 reports the significant parameters of the microhardness profile: the surface microhardness (HV0.1_{surf}), the maximum microhardness (HV0.1_{max}) and the case depth (d₅₅₀), expressed as the distance, in micron, from the surface at which 550HV0.1 is measured. The results for the surface densified DLH are reported, as well.

There are slight differences in microhardness, in particular after LPC1, between the two ACrL materials, whilst case depth is significantly greater in the warm compacted steel. Here the effect of the residual surface porosity can be claimed to justify the different depth. Even if not communicating with the internal pores, it might effectively increase the total exchange surface area increasing the carbon pick-up.

Among the two steels, ACrL results in a greater hardness and a smaller case depth. The relevance of this difference has to be evaluated by means of fatigue and wear tests. In the present work, Charpy impact tests were carried out, to get an evaluation of the embrittlement provided by LPC2. These results are reported in Table 5.

In the as sintered condition, ACrL has a higher impact toughness than DLH because of its ferritic/pearlitic microstructure, which is more ductile than the mainly bainitic microstructure of DLH.¹² This difference is clearly highlighted by the load deflection curves reported in Figure 15.

LPC causes a decrease of impact toughness in ACrL in both cases, as it was expected, since surface hardening decreases ductility. The effect of carburizing on DLH is different, since impact toughness decreases much less in the warm compacted spe-

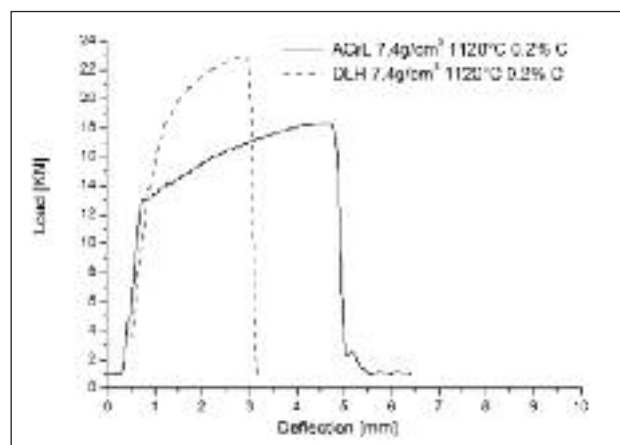


FIG. 15 **Load deflection curves of the warm compacted and sintered ACrL and DLH steels.**

Curve d' impatto degli acciai ACrL e DLH compattati a caldo.

cimens and increases in the cold compacted and shot peened ones. In particular, the high impact toughness of the warm compacted material is noticeable. Figures 16 and 17 compare the fracture surfaces of warm compacted ACrL and DLH in the as sintered (a and b) and in the as carburized (c and d) conditions. The fracture morphology of the case (a and c) and of the bulk (b and d) is shown.

In as sintered ACrL fracture is highly localized and ductile both

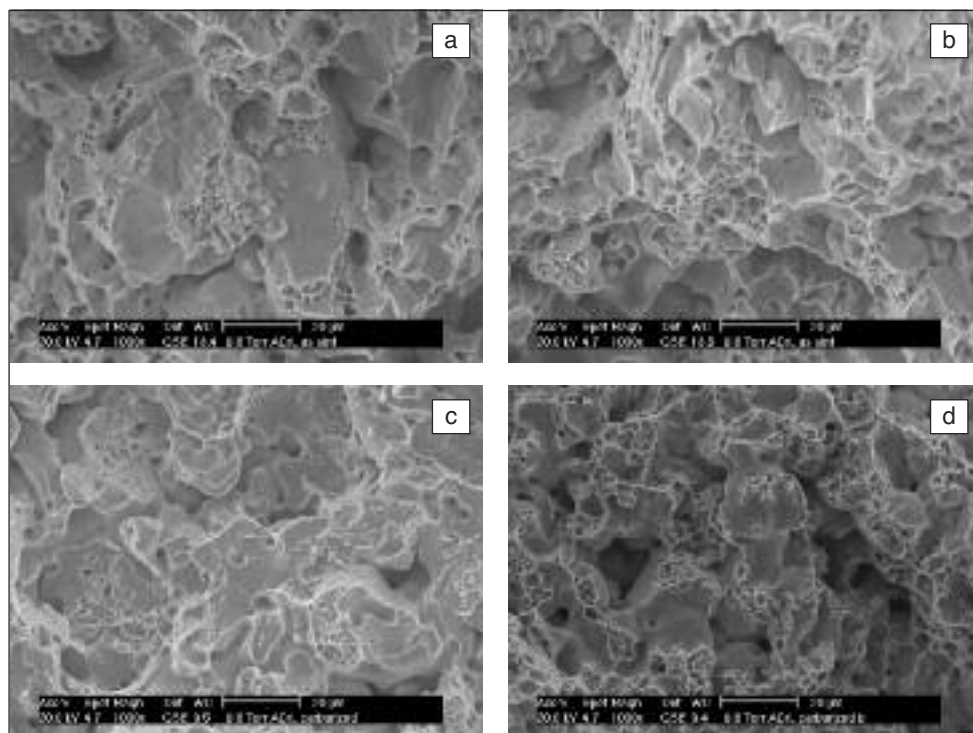


FIG. 16
Fracture surface of as sintered and carburized warm compacted ACrL: a) and c) fracture morphology of the case and b) and d) of the bulk.

Superficie di frattura dell'acciaio ACrL pressato a caldo dopo sinterizzazione e cementazione: a) morfologia dello strato superficiale e b) del cuore.

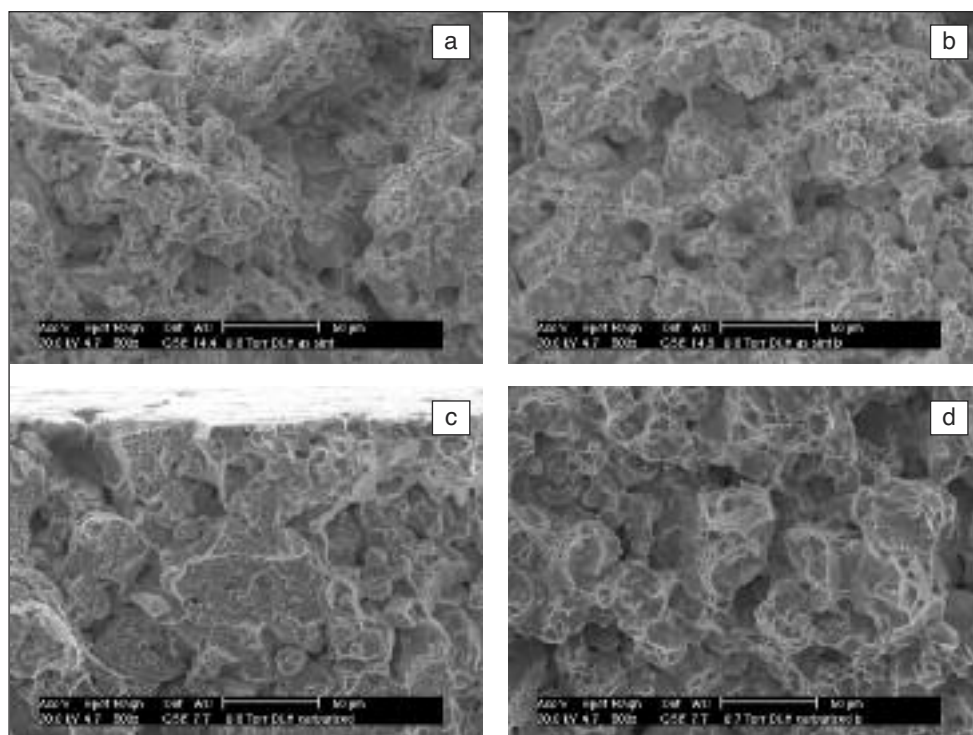


FIG. 17
Fracture surface of as sintered and carburized warm compacted DLH: a) and c) fracture morphology of the case and b) and d) of the bulk.

Superficie di frattura dell'acciaio DLH pressato a caldo dopo sinterizzazione e cementazione: a) morfologia dello strato superficiale e b) del cuore.

in the case and in the bulk, and remains very similar after carburizing, with the presence of some small cleavages areas in the case. In as sintered DLH, fracture is still ductile and localized at the neck regions, but after carburizing it changes significantly in the case. Indeed, the bulk fracture morphology is still localized and ductile, whilst several large fracture areas with quasi-cleavage are observed in the case. They contribute to the spreading of deformation in the bulk of the original powder particles, which is unusual in a carburized layer. Spreading of deformation from the neck regions into the core of the particles is promoted by a strain hardenable matrix, containing retained au-

stenite. Retained austenite may contribute to toughness by either adsorbing plastic energy or transforming into martensite, depending on its stability against the strain induced transformation. The metallographic analysis of the fractured specimen, just close to the fracture surface, confirms the presence of retained austenite, as shown in Figure 18. The toughening mechanism does not involve any transformation, since the high carbon austenite is quite stable.

CONCLUSIONS

The effect LPC on two low alloyed sintered steels processed ac-

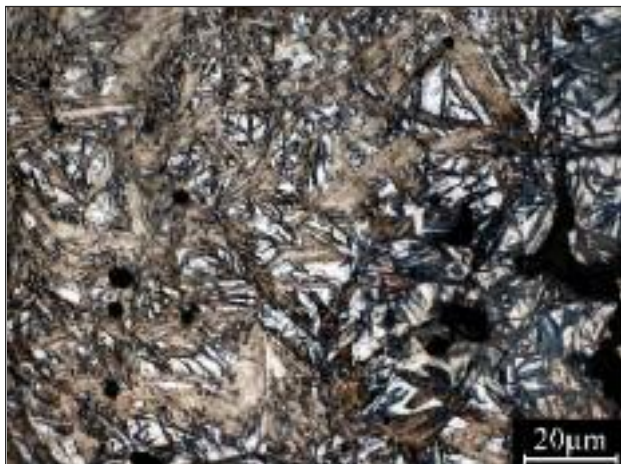


FIG. 18 *Microstructure of LPC2 DLH close to the impact fracture surface.*

Microstruttura in prossimità della superficie ad impatto dell'acciaio DLH cementato con LPC2.

cording to three different routes was studied. The following alternatives were investigated:

- cold compaction and sintering, that results in a medium density with prevailing open porosity;
- warm compaction and sintering, which results in a higher density and mainly closed porosity;
- cold compaction, sintering and surface densification by shot peening.

In presence of an open porosity (variant a) all the materials are overcarburized. The microstructure contains grain boundary carbides in the Cr-containing material, and retained austenite in the Cr free. In presence of a densified surface (variant c), LPC forms a homogeneous case, which is slightly harder and thinner in the Cr containing steel.

The materials processed by warm compaction give different results, since they develop after sintering a significantly different porosity. The Cr containing material has a fully closed porosity,

whilst the other one still has a significant amount of open porosity. Consequently, the case in the Cr steel is homogeneous, whilst it contains retained austenite in the other one because of some overcarburizing.

Impact tests were carried out to investigate the embrittlement provided by LPC. Impact toughness of the surface densified steels is quite similar (13-16J), since both the steels have a brittle behavior. The warm compacted steels are quite different. Whilst the Cr steel is also brittle, the Cr free one possesses an unexpected impact toughness, due to the presence of retained austenite, which should however have a negative effect on wear resistance. Work is in progress to confirm these hypotheses.

This work confirms that LPC can be used to improve the surface mechanical properties of sintered steels, provided that they do not contain a large amount of open porosity. In principle, both the materials are suitable to LPC after surface densification.

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Abstract

Effetto della cementazione in bassa pressione sulla microstruttura, microdurezza e resistenza ad impatto di acciai sinterizzati

Parole chiave: acciaio, sinterizzazione, trattamenti superficiali

L'utilizzo sempre più diffuso di acciai ottenuti per metallurgia delle polveri (PM) in campo automobilistico, ha portato allo sviluppo di nuove polveri e all'utilizzo di trattamenti termochimici e meccanici al fine di aumentarne le prestazioni. Fra le polveri di più recente formulazione possiamo annoverare le prelegate a basso tenore di Cr e Mo, in grado di fornire una microstruttura bainitico- martensitica in seguito a sinter-hardening. Il trattamento di cementazione in bassa pressione (LPC) riveste sicuramente un ruolo importante tra i trattamenti termochimici che si effettuano dopo sinterizzazione, al fine di migliorare alcune proprietà, come resistenza all'usura e alla fatica.

La possibilità di utilizzare la cementazione in bassa pressione, elimina la presenza di agenti ossidanti in atmosfera avviando così all'ossidazione dell'acciaio.

L'assenza di agenti ossidanti rappresenta un'ottima opportunità per cementare anche acciai contenenti elementi in lega con elevata affinità con l'ossigeno, come per l'appunto il cromo.

I gas più utilizzati nella LPC sono acetile e propano. Questi ultimi si dissociano ad alta temperatura (900-1000°C) e in bassa pressione (15mbar), producendo carbonio e idrogeno nel caso dell'acetilene, e carbonio, idrogeno e metano, per il propano.

Il flusso di carbonio risulta maggiore utilizzando acetilene piuttosto che propano, in quanto quest'ultimo dissociandosi forma metano, ovvero un gas inerte alle condizioni di temperatura e pressione previste dal trattamento di cementazione. Per la sua decomposizione sono richieste temperature maggiori e vuoto spinto.

Un ulteriore vantaggio deriva dalla possibilità di raffreddare con un flusso di gas piuttosto che temprare in olio: questo permette di ridurre le distorsioni del pezzo e l'impregnazione della porosità con l'olio di tempra.

Tuttavia, la combinazione sfavorevole tra l'elevata area superficiale degli acciai sinterizzati e l'elevato potenziale di carbonio relativo al processo di cementazione in bassa pressione induce una sovracementazione. La conseguenza è la precipitazione di carburi a bordo grano negli acciai contenenti cromo e la formazione di austenite residua, in quelli in cui il cromo è assente.

Per ovviare a questa limitazione è necessario ridurre la porosità superficiale degli acciai, promuovendo la densificazione superficiale o l'aumento della densità stessa. In letteratura si trovano molteplici lavori che sfruttano la rullatura per promuovere la densificazione superficiale, tuttavia una buona alternativa può essere la pallinatura.

Proprio la validità di quest'ultimo trattamento meccanico è stata studiata in questo lavoro contrapposta alla possibilità di aumentare la densità mediante "warm compaction".

Sono stati investigati due differenti materiali, un acciaio contenente Cr-Mo e uno contenente Mo-Ni-Cu, prodotti attraverso: pressatura convenzionale con densità dopo sinterizzazione medio bassa (a), warm compaction e sinterizzazione con incremento della densità finale (b) e pallinatura sugli acciai con bassa densità (c) al fine di promuovere densificazione superficiale.

E' stato quindi studiato l'effetto del trattamento di cementazione sulla microstruttura, sulla microdurezza e sulla resistenza ad impatto di questi acciai.

In presenza di porosità aperta (a) il processo di cementazione in bassa pressione induce supercementazione in tutti i materiali: la microstruttura è caratterizzata dalla presenza di carburi a bordo grano nei campioni contenenti cromo e dalla formazione di austenite residua negli acciai senza cromo. In presenza della densificazione superficiale (c) LPC forma uno strato superficiale martensitico omogeneo e sufficientemente indurito.

Tuttavia quest'ultimo ha spessore inferiore a quello misurato nei campioni compattati a caldo con densità medio alta. Questo è dovuto probabilmente al ruolo della porosità superficiale, la quale anche se non è comunicante con quella interna, incrementa la superficie di scambio tra acciaio e atmosfera, aumentando l'arricchimento di carbonio.

Vi è una sostanziale differenza, a livello microstrutturale, tra i due acciai con medio alta densità; quelli contenenti cromo presentano una microstruttura martensitica omogenea, mentre dell'austenite residua è ancora presente nello strato di diffusione dei materiali senza cromo. La giustificazione ancora una volta risiede nel ruolo della porosità superficiale, sostanzialmente chiusa nei campioni contenenti cromo e prevalentemente aperta negli altri.

Per quanto riguarda le proprietà ad impatto dei due acciai cementati, si è riscontrato l'infragilimento indotto dal trattamento stesso e quindi dei valori relativamente bassi di energia ad impatto.