Vacuum heat treatment of components for automotive application

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The possibility to carry out vacuum heat treatment with gas quenching, as well as to combine sintering and heat treatment in one single step process, has been investigated on some medium-low alloy steels. In addition to through hardening, which greatly improves mechanical properties of a chromium steel, and to austempering, which can be carried out with a accurate control of the temperature profile during the isothermal step at the bainitic transformation temperature, some examples of vacuum sintering processes, characterized by a specific quenching strategy (sinterhardening and sinteraustempering) have been studied successfully. Vacuum heat treatment with gas quenching opens new possibilities to expand the applications of sintered steels for automotive applications.

Key words: vacuum heat treatment, vacuum sintering, sinterhardening, chromium steels

INTRODUCTION

The largest share of the market of Powder Metallurgy (PM) is represented by the automotive industry. For instance, 56% of the Italian production of PM components in 2004 has been addressed to that sector. A similar situation characterizes the European, American and Japanese industry.

The PM industry has grown almost continuously in the least years, and a significant expansion of the applications of PM products could arise from the common efforts of mechanical designers (end user side) and manufacturers (producer side) to traduce into new parts the results of technological innovation in raw material (powder) and processing. In the frame of the structural applications, as far as the raw material is concerned medium-low alloy iron powders with high compressibility have been developed /1, 2, 3/, which offer the opportunity to improve the properties of the metallic matrix by alloying, without to depress green density excessively. On the processing side, new compaction technologies to increase green density (warm compaction /4/ and high velocity compaction /5/), high sintering temperature /6/, new heat and surface treatments /7, 8, 9, 10/ and specific secondary operations, as surface rolling to densify the surface of the parts /11/, have been proposed.

The recent development of chromium containing iron powders required the use of vacuum heat treatment with high pressure gas quenching for through hardening. This process, which is extensively used since decades for the heat treatment of tools and dies, has a great potentiality in PM since the furnace may be used for sintering, as well, in particular when high temperature is requested. Of course, vacuum furnaces are well suitable for sinterhardening, too.

The chance to change the cooling rate quite easily by varying the pressure of the cooling gas, and more in general, to adapt the cooling strategy to any specific scope, offers the possibility to obtain different microstructural constituents in a reliable way. Simply speaking, we can say that the vacuum sintering process takes care of the neck consolidation during the isothermal holding at high temperature, and of the transformations of austenite during cooling, tailoring the as sintered material (density, porosity and microstructural constituents) to the technical requirements of the specific applications. Therefore, the sintering process assumes the meaning of a two steps operation, sintering and heat treatment, the latter being very relevant with the aim to optimize the industrial production. Moreover, it has to be considered that vacuum atmosphere is almost inert with respect to iron and its main alloying elements used in PM, and that the use of gas for cooling eliminates the problems linked to oil, first of all the necessity to remove it from the open porosity prior to tempering.

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In this paper, some of the results obtained at the Department of Materials Engineering and Industrial Technologies at Trento University, in the frame of various research programmes, are presented. The common aim of these projects is that of studying the optimum “material-process” combination to improve mechanical properties of sintered steels and to promote the expansion of their structural application in several fields, the automotive one included. The common character of the studies here presented is the use of the vacuum technology.

MATERIALS AND EXPERIMENTAL PROCEDURES

Tab.I reports the chemical composition of the powders used for the production of the specimens. They are low-medium alloyed iron powders produced by Höganäs AB, Sweden. Some of the powders have been recently introduced in the market.

Material	Chemical composition	Remarks
A	0.85% Mo	Mo prealloyed
A2N	0.85% Mo – 2% Ni	Mo prealloyed - Ni “diffusion bonded”
AMo	1.5% Mo	Mo prealloyed
DDC	1.5% Mo – 2% Ni	Mo prealloyed - Ni “binder bonded”
ACrL	1.5% Cr – 0.2% Mo	Cr e Mo prealloyed
ACrM	3% Cr – 0.5% Mo	Cr e Mo prealloyed

Table I – Chemical composition of the powders used.

Tab. I – Composizione chimica delle polveri utilizzate.

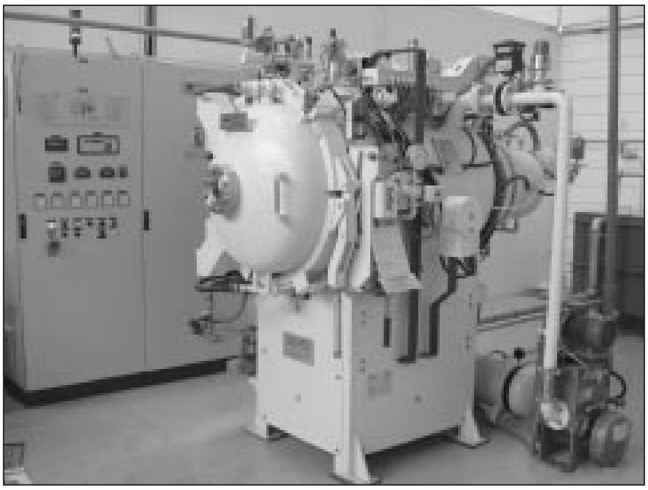

Fig. 1 – Il forno TAV Minijet-HP.

Fig. 1 – The TAV Minijet-HP fornace.

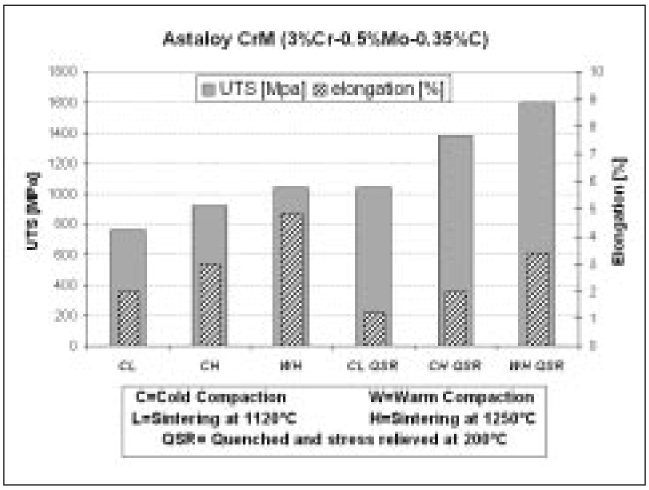

Fig. 2 – Proprietà a trazione dell'acciaio 3% Cr, 0.5% Mo e 0.35% C, sinterizzato e dopo tempra in vuoto e distensione a 200 °C.

Fig. 2 – Tensile properties of 3% Cr, 0.5% Mo e 0.35% C steel, as sintered and vacuum quenched and 200 °C tempered.

Tensile (ISO 2740) and unnotched impact (ISO 5754) specimens were pressed to 7.1 g/cm^3 e 7.4 g/cm^3 green density by cold compaction and warm compaction, respectively. Sintering was carried out in a TAV Minijet-HP vacuum fornace (figure 1) at two different temperatures (1120 °C and 1250 °C), with a vacuum level of 9x10^{-2} mbar, and nitrogen backfilling at 10 mbar. The cooling rate was varied in the range 0.2-8 °C/s. When not differently specified, the cooling rates mentioned in the following are those measured by means of two thermocouple, the first in the central axis and the second close to the surface of a Charpy testpiece (10x10) mm^2 cross section.

The microstructural analysis, the microhardness and hardness measurements and mechanical tests have been carried out according to the standard procedures adopted for sintered steels.

RESULTS AND DISCUSSION

Vacuum through hardening has been applied to the 3% Cr and 0.5% Mo steel alloyed with two different carbon contents: 0.35% and 0.5% /12/. The best results in terms of mechanical properties have been obtained with the steel containing 0.35% C, since the martensite hardness of the steel containing 0.5% C results too high and, as well known, /13/, tensile properties are depressed owing to the negative effect of porosity in an excessively hard matrix.

Figure 2 shows the results of tensile tests on specimens quenched and tempered at 200 °C, in comparison with those of the as sintered specimens. Heat treated materials have a fully martensitic microstructure; density is 7.04 g/cm^3 (CL: cold compaction and sintering at 1120 °C), 7.15 g/cm^3 (CH: cold compaction and sintering at 1250 °C), 7.29 g/cm^3 (WH: warm compaction and sintering at 1250 °C). Through hardening increases UTS and yield strength and decreases elongation at fracture. Strength and ductility of the heat treated materials are very high, in particular if they have been sintered at high temperature (CH e WH). Properties of the warm compacted and high temperature sintered material are outstanding, being very close to the upper limit of the potentiality of PM steels. The same heat treatment has been applied to other low alloyed steels /14/, some of them characterized by a lower hardenability than the chromium steels above presented, obtaining results comparable to those attainable with the conventional heat treatment in belt furnaces and endogas atmosphere.

The diffusion of sinterhardening in the PM industry is rapidly growing. In order to optimize this process, the actual cooling rate in the core of the specimens has to be control-

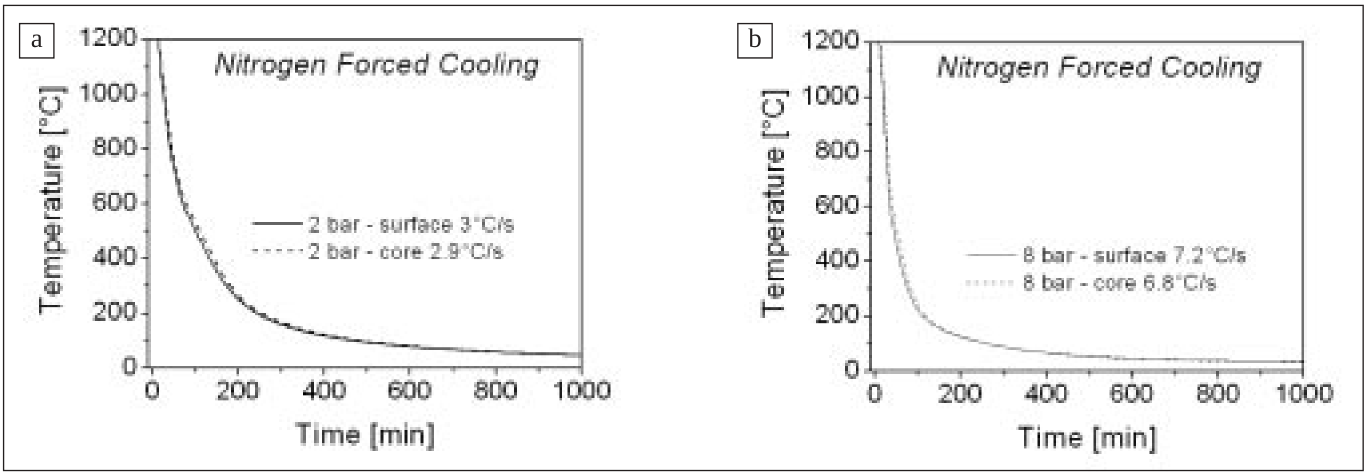

Fig. 3 – Velocità di raffreddamento in superficie ed a cuore di un provino Charpy (sezione 10x10mm^2) con raffreddamento forzato in flusso di azoto a 2 bar (3a) e 8 bar (3b).

Fig. 3 – Surface and core cooling rate in a Charpy testpiece (10x10mm^2 cross section) with nitrogen forced flux at 2 bar (3a) and 8 bar (3b).

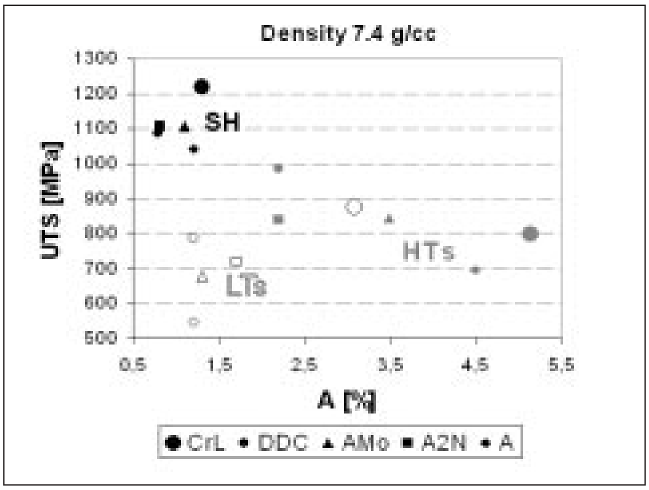

Fig. 4 – UTS e allungamento a rottura di acciai contenenti 0.6% C sinterizzati a 1120°C (LTs), 1250°C (HTs)e sinterotemprati da 1250 °C (SH).

Fig. 4 – UTS elongation at fracture of 0.6% C steels, sintered at 1120°C (LTs), 1250°C (HTs) and sinterhardened from 1250 °C (SH).

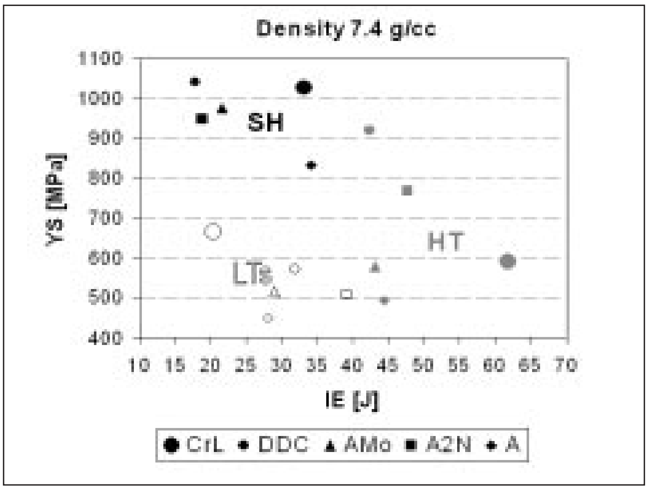

Fig. 5 – Carico di snervamento e energia all'impatto di acciai contenenti 0.6% C sinterizzati a 1120°C (LTs), 1250°C (HTs) e sinterotemprati da 1250 °C (SH).

Fig. 5 – Yield strength and impact energy of 0.6% C steels, sintered at 1120°C (LTs), 1250°C (HTs) and sinterhardened from 1250 °C (SH).

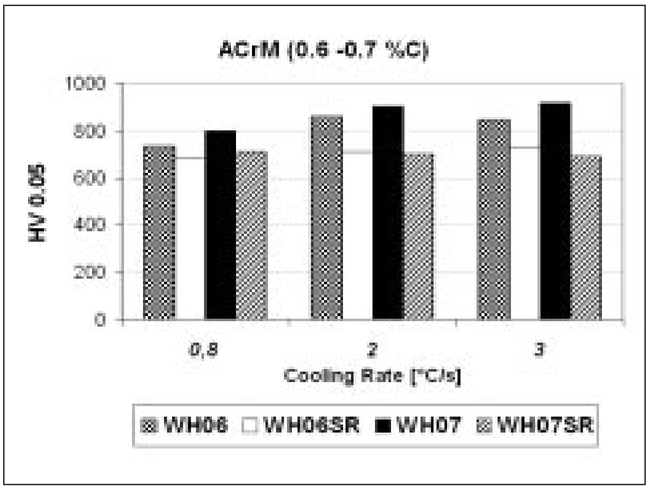

Fig. 6 – Microdurezza dell'acciaio 3% Cr, 0.5% Mo e 0.6-0.7% C in funzione della velocità di raffreddamento.

Fig. 6 – Microhardness of the 3% Cr, 0.5% Mo and 0.6-0.7% C steel as a function of the cooling rate.

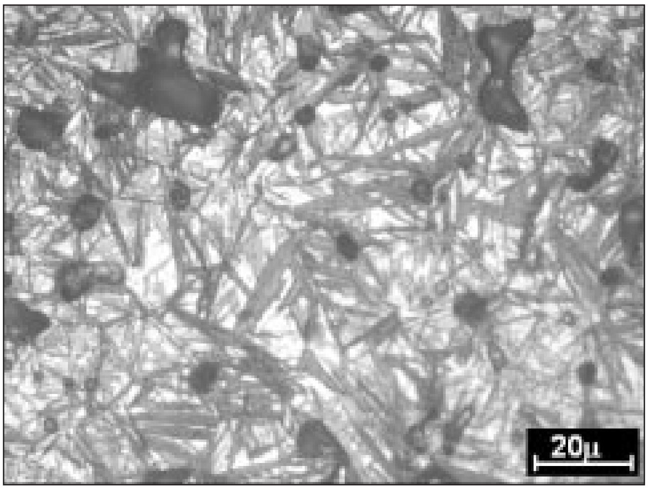

Fig. 7 – Microstruttura dell'acciaio 3% Cr, 0.5% Mo e 0.6-0.7% C sinterizzato con raffreddamento a 0.8 °C/s.

Fig. 7 – Microstructure of the 3% Cr, 0.5% Mo e 0.6-0.7% C steel, sintered with 0.8 °C/s cooling rate.

led. This is quite easy in the batch treatments, as the vacuum quenching is. In the frame of an extensive investigation on the sinterhardening behaviour of several low alloyed steels, a preliminary evaluation of the effect of the nitrogen pressure on the forced cooling rate both at the surface and in the core of a Charpy testpiece (cross section 10x10 mm^2) was carried out.

The curves in figure 3 show the results for two different nitrogen pressures: 2 bar (fig. 3a) e 8 bar (fig. 3b). In both cases, no significant difference between the surface and the core was measured, even at the lowest nitrogen pressure, the actual cooling rate at the core is high enough to quench most of the sinterhardening materials available on the market. For a reliable and safe sinterhardening, the core cooling rate has to be higher than the critical quenching temperature of the material. An excessive "overquenching" does not add benefits but, contrarily, may enhance quenching stresses. By means of the vacuum quenching with gas cooling, the cooling rate may be quite easily tailored to the chemical composition of the steel and reproduced in the industrial production.

Some of the results of this investigation are shown in figures 4 and 5, which report Ultimate Tensile Strength (UTS) and elongation at fracture (A%) (figure 4) and yield strength (YS) and impact energy (IE) (figure 5). Specimens have been produced in three different conditions: standard sintering at 1120 °C (LTs), vacuum sintering at 1250 °C (HTs) and sinterhardening at 1250 °C in vacuum (SH). The five materials considered were alloyed with 0.6 %C and warm compacted to 7.4 g/cm^3 green density /15/. With reference to the different processing conditions, it may be noticed that high temperature sintering increases both strength and ductility of the materials, as expected. A similar improvement was obtained with high temperature sintering in atmosphere furnaces, as well, and therefore the improvement has to be attributed to the effect of temperature rather than to the vacuum atmosphere /14/. Sinterhardening increases the martensite content in the microstructure (some of the materials contain other microstructural constituents, as bainite and pearlite in traces, due to their low hardenability), and this results in a further increase in strength with a decrease of ductility and toughness. Anyway, properties obtained with sinterhardening have a practical interest, in particular if evaluated in combination with the good compressibility of the base materials.

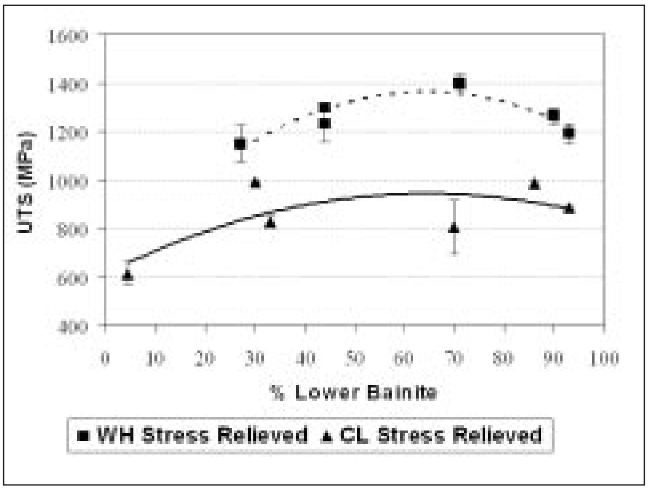

Fig. 8 – UTS dell'acciaio 3% Cr, 0.5% Mo e 0.6-0.7% C in funzione della percentuale di bainite della matrice.

Fig. 8 – UTS of the 3% Cr, 0.5% Mo and 0.6-0.7% C steel as a function of the percentage of bainite in the matrix.

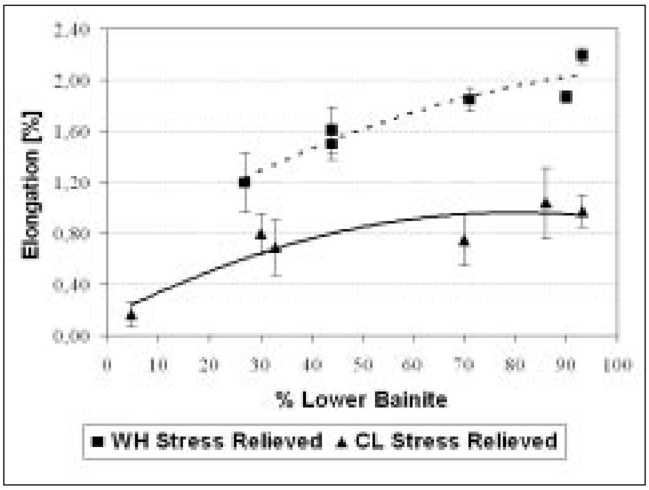

Fig. 9 – Allungamento a rottura dell'acciaio 3% Cr, 0.5% Mo e 0.6-0.7% C in funzione della percentuale di bainite della matrice.

Fig. 9 – Elongation at fracture of the 3% Cr, 0.5% Mo and 0.6-0.7% C steel as a function of the percentage of bainite in the matrix.

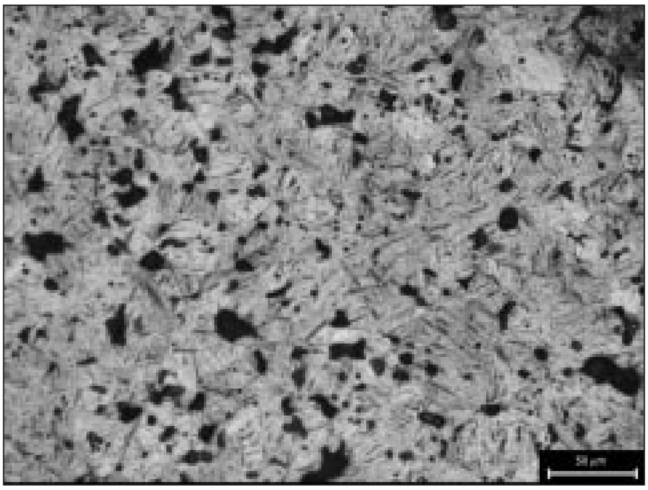

Fig. 10 – Microstruttura dell'acciaio 3% Cr, 0.5% Mo e 0.5% C, sinterotemprato da 1250 °C con velocità di raffreddamento di 3 °C/s.

Fig. 10 – Microstructure of the 3% Cr, 0.5% Mo e 0.5% C steel, sinterhardened from 1250 °C with 3 °C/s cooling rate.

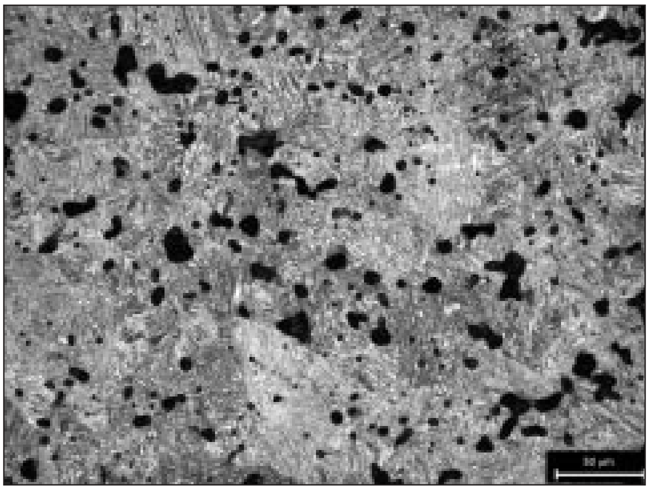

Fig. 11 – Microstruttura dell'acciaio 3% Cr, 0.5% Mo e 0.5% C, sinteroaustemperato a 350 °C.

Fig. 11 – Microstructure of the 3% Cr, 0.5% Mo e 0.5% C steel, sinteraustempered at 350 °C.

Among the five materials investigated, the best results have been displayed by ACrL, the chromium steel containing the lower amount of chromium and molybdenum (Tab.I). When sinterhardened, it shows both yield strength and elongation at fracture comparable to those of the other steels, but UTS and impact energy are significantly better. This material, when properly sinterhardened, has a relevant interest for structural applications.

The 3% Cr and 0.5% Mo steel has the greatest hardenability among all the materials here considered. By adding a high carbon content (ranging between 0.6% and 0.7%), the as sintered microhardness rises even using a medium cooling rate, as shown in figure 6 relevant to specimens warm compacted, sintered at 1250 °C and tempered at 200 °C /16/. The microstructure is fully martensitic with a cooling rate of 2 °C/s, so that microhardness does not increase with a further increase in the cooling rate. The specimens cooled down at the lowest rate displayed a martensitic-bainitic microstructure, an example of that is reported in figure 7. As previously mentioned, when the matrix microhardness is very high, the steel tends to have a brittle behaviour, since pores act as pre-existing cracks in a matrix characterized by a low fracture toughness /13/. In these conditions, the mechanical strength depends on the fracture toughness of the matrix, which decreases on increasing its microhardness. As a consequence, the dual phase materials have the highest strength /16/. The possibility to vary the cooling rate opens the chance to optimize this dual phase microstructure /17/. By increasing the cooling rate, the martensite content of the matrix increases, as expected, but tensile strength does not change in the same way. Figures 8 and 9 show UTS and elongation at fracture, respectively, as a function of the bainite content of the matrix, for specimens cold compacted and sintered at 1120 °C (CL) and specimens warm compacted and sintered at 1250 °C (WH). In both cases, specimens have been tempered at 200 °C. While UTS reaches a maximum in correspondence of 50-60% bainite, elongation increases with the bainite content. Results have a practical interest, in particular the combination of properties displayed by the WH material: 1400 MPa UTS and 1.8% elongation at fracture.

Recently, the possibility to carry out the austempering heat treatment in the vacuum furnace has been investigated. Austempering consists in an austenitization followed by a rapid quenching down to a temperature higher than martensite start, an isothermal holding at this temperature to transform austenite in bainite and finally cooling down to room temperature. Lower bainite has mechanical strength slightly lower

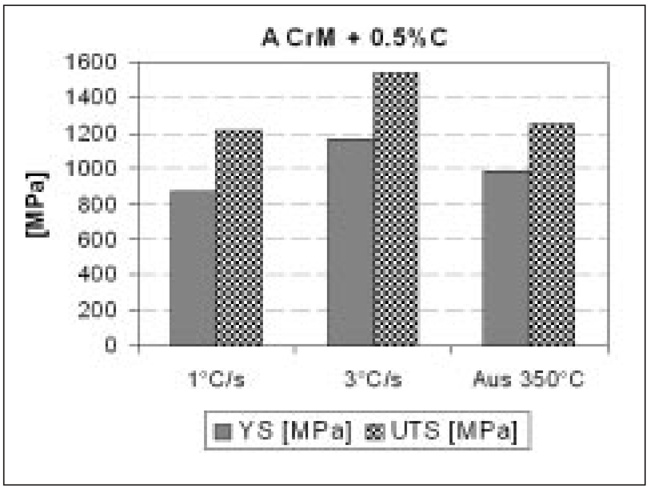

Fig. 12 – UTS e carico di snervamento dell'acciaio 3% Cr, 0.5% Mo e 0.5% C, sinterotemprato e sinteraustemperato.

Fig. 12 – UTS and yield strength of the 3% Cr, 0.5% Mo e 0.5% C steel, sinterhardened and sinteraustempered.

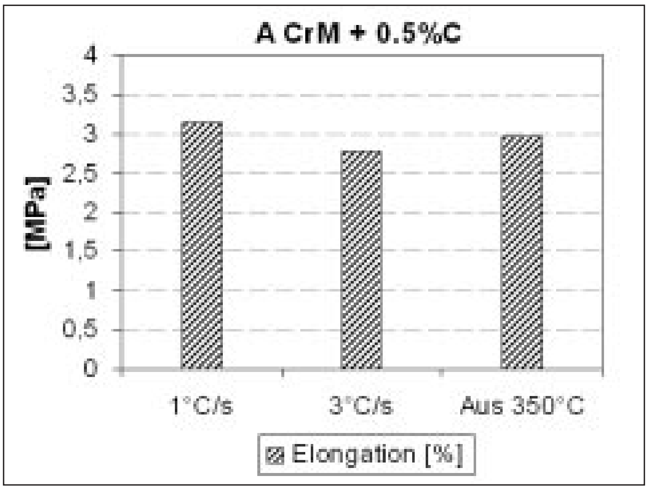

Fig. 13 – Allungamento a rottura dell'acciaio 3% Cr, 0.5% Mo e 0.5% C, sinterotemprato e sinteraustemperato.

Fig. 13 – Elongation at fracture of the 3% Cr, 0.5% Mo e 0.5% C steel, sinterhardened and sinteraustempered.

than that of martensite but it does not requires tempering. Even though harder than upper bainite, it is less brittle because of the finer microstructure and the absence of interlath carbides /18/. Since the type of bainite formed on cooling depends on the temperature at which transformation of austenite occurs, the isothermal transformation allows a much better control of the final microstructure than continuous cooling transformation.

In the industrial practice, austempering is carried out in salt bath. Sintered steels are not well suitable to be treated in salt baths, since some salt residuals remain entrapped in the open porosity and they have to be removed after the treatment. The heat treatment has been set up in the vacuum furnace obtaining an excellent control of the temperature profile of the surface and the core of a Charpy testpiece (maximum difference less than 5 °C at the beginning of the isothermal step). As a logical development, such a cooling has been combined to the sintering cycle, to carry out a "sinteraustempering" process. The aim of this process is that of obtaining a performing microstructure directly on sintering, avoiding tempering of pieces. Experiments have been carried out on the ACrM steel bonded with 0.5% C. Green pieces were obtained by warm compaction, and the sintering temperature was 1250 °C. Two sinterhardning processes were carried out, as well, with 1°C/s and 3°C/s cooling rate. Figures 10 and 11 show the microstructure obtained with sinterhardening at 3 °C/s (fully martensitic) and with the "sinteraustempering" at 350°C (fully bainitic), respectively. Sinterhardening with the lower cooling rate produces some bainite in a prevailing martensitic microstructure. Figures 12 e 13 show the results of tensile tests: UTS and yield strength in figure 12, elongation at fracture in figure 13. The properties of the sintraustempered material are better than those of the material sinterhardened 1°C/s, and only sligthly lower than those of the fully sinterhardened material. These preliminary results are very encouraging and show that this new combined treatment may be carried out in the vacuum furnace with gas cooling. Further experiments are in course to optimize the process for different PM steels.

CONCLUSIONS

The vacuum heat treatment with gas quenching has been experimented on several PM medium-low alloyed steels, both as a secondary operation on previously sintered materials and as a final step of a process in which vacuum sintering is followed by a controlled cooling to produce a specific microstructure.

As a secondary operation for previously sintered steels, vacuum quenching is well suitable to through harden chromium steels, which cannot be treated in the conventional belt furnaces working in endogas, which are used in the standard production of the PM industry. Of course, the use of gas instead of oil for quenching eliminates the necessity to remove the quenching media from the open porosity.

Since vacuum furnaces are also used for sintering, in particular at high temperature, the possibility to combine high temperature sintering with a controlled cooling may be exploited to carry out sinterhardening with optimum cooling rate, i.e. with the minimum cooling rate requested by the material hardenability avoiding any "over-quenching" which may generate excessive quenching stresses. Vacuum sinterhardening has been experimented successfully on some low-medium alloyed steels.

As a particular case of sinterhardening, dual phase bainitic-martensitic microstructures have been obtained on high carbon chromium steels, and tensile properties have been optimized in relation to the bainitic content.

Finally, the austempering treatment was investigated, to obtain a fully lower bainitic microstructure. Once the cooling strategy has been set up, austempering was combined to sintering in a single process called "sinteraustempering". Mechanical properties are slightly lower than those attainable with sinterhardening, but pieces do not require any tempering. A further improvement of the properties may be expected.

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ABSTRACT

TRATTAMENTO TERMICO IN VUOTO DI COMPONENTI PER APPLICAZIONI AUTOMOBILISTICHE

Parole chiave: tempra in vuoto; sinterizzazione in vuoto, sinterotempra, acciai al cromo

In questo lavoro vengono riassunti i risultati ottenuti all'Università di Trento, nell'ambito di alcuni progetti di ricerca industriale, sull'utilizzo dei forni di tempra in vuoto per il trattamento termico e per la sinterizzazione di acciai per applicazioni strutturali. La possibilità di variare la velocità di raffreddamento in modo semplice ed affidabile consente di ottimizzare sia il trattamento di tempra, sia la sinterizzazione, in relazione al materiale utilizzato e alle specifiche tecniche dell'applicazione.

La tabella 1 riporta la composizione chimica delle polveri utilizzate. I campioni per le prove di trazione ed impatto sono stati pressati a 7.1 e 7.4 g/cm3, e sinterizzati a 1120°C o 1250°C in un forno TAV Minijet_HP (figura 1), con un livello di vuoto di 9x10-2 mbar e back-filling di 10 mbar in azoto.

La figura 2 riporta i risultati delle prove di trazione sull'acciaio con 3% Cr e 0.5% Mo contenente 0.6% C, sinterizzato, temprato e disteso a 200°C, in confronto con quelli sull'acciaio solo sinterizzato. In particolare le proprietà dell'acciaio WH (pressato con "warm compaction" e sinterizzato ad alta temperatura sono eccellenti, prossime al limite superiore delle proprietà ottenibili con gli acciai sinterizzati.

Il forno in vuoto si presta molto bene al processo di sinterotempra, anche perché consente un efficace controllo della velocità di raffreddamento al cuore dei pezzi. Le curve di figura 3 mostrano le velocità di raffreddamento in convezione forzata a cuore ed in superficie di provini Charpy (sezione 10x10 mm2) per due valori della pressione dell'azoto di spegnimento: 2 bar (fig. 3a) e 8 bar (fig. 3b). Anche alla pressione minore, la velocità di raffreddamento al cuore è sufficiente per sinterotemprare i materiali esistenti sul mercato.

Le figure 4 e 5 mostrano i risultati delle prove di trazione ed impatto sui materiali sinterotemprati (SH), in confronto con quelli sinterizzati sia a 1120°C (LTs) che a 1250°C (HTs). Tutti i materiali contengono 0.6%C al verde e sono stati pressati a 7.4 g/cm3. Le proprietà ottenibili con la sinterotempra, in particolare nell'acciaio al cromo, sono molto interessanti soprattutto se valutate in combinazione con l'ottima comprimibilità delle polveri.

Fra tutti i materiali studiati, quello con il 3% Cr e 0.5% Mo presenta la maggiore temprabilità, quindi è stato utilizzato per ottenere microstrutture dotate di elevata resistenza meccanica anche con velocità di raffreddamento medio-basse, aggiungendo elevate quantità di carbonio. La figura 6 mostra la microdurezza dei campioni compattati con "warm compaction", sinterizzati a 1250°C e distesi a 200°C. Solo il campione raffreddato a 0.8 °C/s non è completamente martensitico, come mostrato dalla figura 7. La sua microstruttura bainitico-martensitica conferisce tuttavia la maggiore resistenza, in quanto l'elevata durezza della martensite, in un materiale poroso, determina un'eccessiva fragilità e nei materiali a comportamento fragile la resistenza meccanica dipende dalla tenacità alla frattura della matrice. Quindi è stato possibile, con ulteriore sperimentazione, ottimizzare la microstruttura bifasica, potendo variare la velocità di raffreddamento in modo agevole e riproducibile. Le figure 8 e 9 mostrano i valori di UTS e di allungamento a rottura in funzione della percentuale di bainite della matrice.

Recentemente, il forno di trattamento termico in vuoto è stato utilizzato per effettuare il trattamento di austempering, per ottenere in modo isotermo, e quindi affidabile, una microstruttura completamente bainitica inferiore. Da questa microstruttura ci si può aspettare un insieme di proprietà meccaniche non troppo inferiori a quelle della martensite, senza però dover effettuare il trattamento di rinvenimento. Il trattamento in vuoto, nel caso dei materiali sinterizzati, è decisamente preferibile a quello convenzionale in bagno di sale, in quanto i sali possono rimanere intrappolati nella porosità aperta e devono essere rimossi. Gli esperimenti condotti hanno dimostrato che è possibile realizzare un controllo molto accurato della temperatura nel pezzo (è stato impiegato il provino Charpy), e come logico sviluppo sono stati realizzati dei cicli di "sinteroaustempering". Le figure 10 e 11 mostrano la microstruttura dell'acciaio contenente 3% Cr, 0.5% Mo e 0.5% C, pressato con "warm compaction" e sinterotemprato (velocità di raffreddamento pari a 3 °C/s) o sinteroaustemperato a 350 °C. In entrambi i casi la temperatura di sinterizzazione era 1250°C. La resistenza a trazione e il carico di snervamento sono riportati in figura 12, l'allungamento a rottura in figura 13. In entrambe le figure sono riportate le proprietà meccaniche anche del materiale sinterotemprato con velocità di raffreddamento di 1 °C/s, che produce una microstruttura non completamente martensitica.

I risultati ottenuti con il processo di sinteroaustempering sono decisamente incoraggianti e lo studio per l'ottimizzazione del trattamento è in corso.

I risultati di questi studi dimostrano che il trattamento termico in vuoto è applicabile con successo anche ad acciai sinterizzati con sufficiente temprabilità. Inoltre, nel forno in vuoto è possibile combinare la fase di sinterizzazione ad alta temperatura con il trattamento termico e ottenere acciai con microstrutture adatte alle specifiche applicazioni in modo affidabile e riproducibile.