EFFECT OF THE HEAT TREATMENT ON THE MECHANICAL PROPERTIES OF A PRECIPITATION HARDENING STEEL FOR LARGE PLASTIC MOLDS


Continuously growing activity in the area of the engineering plastics led to the necessity of developing new low-cost, high-performance plastic mold steels. In fact, when it is necessary to fabricate large plastic components, such as bumpers and dashboards for motor vehicles, the traditionally adopted ISO 1.2738 plastic mold steel exhibits low fracture toughness and highly inhomogeneous microstructures (continuously varying from surface to core), as obtained from the pre-hardening (quenching and tempering) of large blooms. New alloys and alternative manufacturing routes may allow to obtain plastic injection molds with good mechanical, wear and weldability properties. Precipitation hardening tool steels are being proposed for such an application, yielding improved mechanical properties and lower overall costs and lead-time. A precipitation hardenable steel, developed for injection molding of large engineering polymer components, was investigated.

The microstructures and the mechanical properties of the precipitation hardenable steel bloom were investigated after the steelwork heat treatment. Moreover, the strengthening mechanism by means of aging heat treatments was examined on samples subjected either to the steelwork heat treatment only, or also to a successive laboratory heat treatment. To the purpose, X-rays diffraction and EDS analyses were carried out in order to indentify second phases electrochemically extracted from aged and not aged samples.

KEYWORDS: plastic mold steel, precipitation hardening, metallography, mechanical properties, fracture toughness, fractography

INTRODUCTION

Large steel molds are employed in injection molding processes to fabricate massive plastic automotive components (such as bumpers and dashboards), by using glass-reinforced thermoplastic polymers. During the service, several stresses act on a plastic mold: polymer’s injection pressure, mechanical and thermal fatigue (a few millions of pieces can be fabricated with one mold), and wear from reinforced resin flows; stresses can be further enhanced by notch effects and by abnormal shop operations. The molds are commonly machined from large quenched and tempered blooms, typically with 1x1 m cross-section and more than 1 m length. The ISO 1.2738 (or 40CrMnNiMo8-6-4 [1]) alloy steel grade is the most used steel. Due to the large section, blooms of the above steel exhibit after heat treatment, inhomogeneous microstructures and mechanical properties continuously varying from the surface to the core of the bloom; impact notch strength and fracture toughness are everywhere quite low (at the 10 J and 40 MPa√m level, respectively [2]). Moreover, the ISO 1.2738 steel is difficult to weld (1.16 carbon equivalent index [3]), although weld bed deposition operations are usually necessary to modify the mold face, also to extend the service life during model revamping.

Several precipitation hardening steels have been proposed as an alternative, with the aim of yielding more uniform microstructures and better properties throughout the mold sections, and to improve weldability (a carbon content lower than 0.4% may be adopted). The P21 [4,5] standard grade steel, for example, contains 0.2% C, 4% Ni, 1.2% Co, and lower amounts of V, Al, Mn, Si, Cr [4]; yet, most grades are proprietary and not disclosed in detail [6]. The solubilization temperature can be subcritical, as for the P21 grade [4,7] (albeit after an hypercritical annealing [7]), or hypercritical, for some proprietary grades, whereas the aging temperature is always subcritical (e.g. 530 °C for the P21 grade [4,7]), and therefore yields only very limited dimensional variations. The final serv-
ice) hardness is usually in the 37-42 HRC [6] range. The precipitation hardening heat treatment can be performed after mould machining, owing to the fact that it induces only very limited deformations [8], and a suitable aging treatment may yield homogeneous microstructures and mechanical properties in geometrically complex and large moulds. Furthermore, in some cases (e.g. for the here proposed steel), the preliminary heat-treatment performed on the as-forged bloom can employ air cooling after austenitization, as opposed to oil quenching in the case of traditional hardened steels, thus yielding much lower temperature gradients and minimizing residual stresses.

In the present work a precipitation hardenable steel is investigated; its chemical composition is listed in Tab. 1. The aging of this steel in the 550 to 630 °C temperature range was previously studied by means of hardness tests performed after increasing aging durations [9], evidencing overaging peaks after 1 or 2 h aging at 630 or 590 °C, respectively, as opposed to almost asymptotic aging at 550 °C up to 20 h duration. For this reason, and since the homogeneous heating of large moulds may require several hours and implies different actual durations at temperature from surface to core, only aging temperatures equal or lower than 550 °C are considered here.

**EXPERIMENTAL PROCEDURES**

The bloom production cycle, performed in the steelwork, consisted of several steps: ingot casting, hot forging (in order to reduce the microstructural and chemical inhomogeneities and to obtain a 2400 (L) x 1500 (T) x 500 (S) mm bloom), preliminary heat treatment (including a dehydrogenization process), austenitization/solubilization, air quenching, low-temperature tempering. The heat treatment schedule is displayed in Fig.1a.

By superimposing the austenite cooling stages (of the bloom heat treatment) to the steel’s CCT diagram (Fig. 1b), it can be hypothesized that: i) during the first austenitization, most of the primary Mo and V carbides were dissolved; ii) during the first air cooling and furnace cooling stages, the austenite-to-pearlite transformation was avoided, a fine and homogeneous carbides re-precipitation occurred, and the austenite was finally transformed into bainite at lower temperatures, no transformation taking place in the final uncontrolled air cooling to room temperature; iii) the de-hydrogenization treatment, performed in two steps, caused (tempering and) aging of the bainitic matrix and carbides coarsening. Moreover, as it regards the final heat treatment, the bloom was austenitized at a lower temperature (1020 °C), so that Mo and V carbides were partially dissolved, and it is hypothesized that, after the air quenching, a homogeneous and fully bainitic microstructure was obtained both in the surface and in the core of the bloom. Finally, the steel was subjected to a double tempering at 400 °C.

All the examined samples were cut from the bloom at a distance of less than 170 mm from the bloom surface in the S direction.

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**Table 1**

Nominal chemical composition of the proposed steel (wt. %) (actual analysis covered by industrial confidentiality).

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Si</th>
<th>V</th>
</tr>
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<tr>
<td>0.05 - 0.15</td>
<td>0.1 - 1.1</td>
<td>0.1 - 0.9</td>
<td>2.5 - 4.5</td>
<td>2.5 - 4.5</td>
<td>0.1 - 1.1</td>
<td>0.05 - 0.20</td>
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</table>
Moreover, some samples were subjected to the following laboratory re-heat-treatment: austenitization/solubilization at 1050 °C, water quenching, double tempering at 400 °C. Sets of either as-received or laboratory re-heat-treated samples were then aged at three different temperatures: 470, 510, or 550 °C. Different samples of each set were extracted from the furnace after aging durations increasing up to 8 hours, and water quenched.

The microstructure was examined by optical and electronic microscopy, after Nital or Picral etch, and the austenitic average grain size was measured by using the circular intercept method [11], after Bechet-Beaujard etch. Standard tensile tests, plain-strain fracture toughness tests, Charpy-V impact tests, Vickers hardness tests, and FIMEC (Flat top cylindrical Indentations for Mechanical Characterization) test were performed upon samples cut from the steel bloom, either in the as-received state or after the above described re-heat treatments. The reported hardness values are averages of 3 indentations. Fracture toughness tests were performed on 35 mm thick SENB (Single Edge Notch Bend) specimens [13]. The FIMEC indentation tests [14,15,16,17] were performed with a flat cylindrical indenter (1 mm diameter) and a 1.66 μm/s displacement rate. The fracture surfaces of tensile and fracture toughness samples were examined by Scanning Electron Microscopy (SEM).

X-ray diffraction and EDS analyses were performed on electrochemically extracted second phases (carbides and inclusions), in order to detect the nature of the particles precipitated during the aging heat treatment. The sample was dissolved in ethanol and hydrochloric acid (10% vol.), the undissolved second phases were collected on a filter (0.1 mm mesh size), and the filter was subjected to X-ray diffraction analysis (Co-Kα radiation). For comparison, the same analysis was carried out on an unused filter. EDS analyses was performed on compacted second phase powder.

RESULTS

Microstructures

After the steelwork heat treatment, the as-received microstructure is homogeneous bainite, modified by tempering (Fig. 2). Small randomly distributed carbide particles, not completely resolved by optical microscopy, are present in the bainitic matrix, probably Mo and V carbides. The previous austenite grain boundaries are clearly evident (Fig. 2c,d), probably due to the occurrence of a precipitated carbides layer, not always
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Continuous, and partially removed during the metallographic preparation. The average austenitic grain size is 130 µm (mean of 263 intercepts).

As it regards the laboratory re-heat-treatment, after water quenching, a martensitic microstructure with lath morphology (typical of low carbon steels) was obtained. The microstructure exhibits 445 HV_{100} hardness and 980 MPa yield strength (determined by FIMEC test). The tempered martensite, obtained after the first tempering at 400 °C, exhibited a morphology similar to the previous as-quenched martensite, but with a lower hardness (420 HV_{100}) and yield strength (910 MPa, by FIMEC test). Both the microstructure and these mechanical properties did not change noticeably after the second tempering.

The optical metallographic analysis of as received samples has not detected important microstructural variation after aging (Fig. 3). The previous austenite grain boundary precipitates, observed in the as-received samples, are not present in the re-heat treated samples, neither after the quenching nor after the double tempering at 400 °C (Fig. 4a); however, they are again visible, in increasing amount, after aging the re-heat-treated samples at temperatures increasing from 470 to 550 °C (Fig. 4b,c,d).

Detailed SEM examination of aged samples (initially being either in the as-received or in the re-heat-treated condition) showed that the amount of detectable carbides mostly decreases with the aging duration and temperature (Fig. 5); therefore, it is hypothesized that during the aging treatments the previously existing carbides, formed during the previous heat treatments and probably not thermodynamically stable in the aging temperature range, are progressively solubilized, while finer (undetectable) carbides, possibly with a different composition, are re-precipitated and may be the origin of the detected hardness increase (see below).

The X-ray analysis of the second phases (carbides and inclusions) electrochemically extracted from the re-heat-treated sample aged at 550 °C for 440 min is displayed in Fig. 6. The most important diffraction peaks can be attributed to the η-MoC carbide, with the possible presence of the V_{13}C_{6} carbide. Moreover, the EDS analysis, carried out on the same second phases compacted powder, has confirmed the occurrence of molybdenum, that has the highest peak, and of
others elements, such as V, Fe, Cr, Si.

**Mechanical tests**

The results of the tensile and fracture toughness tests are listed in Tab. 2 and 3, and compared with the previously assessed properties of the ISO 1.2738 steel [2]. In particular, the fracture toughness value of the examined steel in the as-received condition is somewhat higher than that of the ISO 1.2738 steel, whereas the tensile properties are comparable.

The hardness curves relative to the age hardening heat treatment on the as-received and re-heat-treated samples are displayed in Fig. 7. The 550 °C aging temperature yielded the
Frattura di trazione e tenacità a frattura dell'acciaio esaminato, in diverse condizioni metallurgiche, confrontato con l'acciaio 1.2738 (valori medi per ciascun acciaio o posizione). YS: tensione di snervamento; UTS: tensione di rottura; n: esponente di incrudimento; $E_l$: allungamento uniforme; $E_t$: allungamento a rottura. Tempo di invecchiamento 2,5 h.

Tab. 2
Tensile properties of the examined steel, in different metallurgical conditions, compared with the 1.2738 steel (average values for each steel or position). YS: Yield Strength; UTS: Ultimate Tensile Strength; n: hardening exponent; $E_l$: uniform elongation; $E_t$: elongation at fracture. Aging duration 2.5 h.

Tab. 3
Fracture toughness ($K_{IC}$) of the examined steel, in different metallurgical conditions, compared with the 1.2738 steel (average values).

higher hardness values: 490 HV$_{100}$ for the re-heat treated sample and 485 HV$_{100}$ for the as-received one, starting from 420 and 380 HV$_{100}$ respectively. As a consequence, the as-received specimens are more sensitive to the aging heat treatment than the re-heat-treated ones, achieving a similar hard- ness notwithstanding their hardness being less before aging. Probably, this behavior is due to the fact that the hardness of the bainitic and martensitic microstructure (of the as-received and re-heat treated samples, respectively) progressively becomes similar at increasing aging temperature, due to supplementary tempering phenomena superimposing on the aging precipitation. Overaging phenomena are not detected, except that in the re-heat treated sample aged at 550 °C, which exhibits a slight decrease in hardness for duration longer than 3 hours.

The FIMEC stress vs. displacement curves, obtained on the as-received and re-heat treated samples aged for the longest durations (about 440 min, Fig. 8), confirm the above results: the yield stress (like the hardness) increases from 890 MPa in the as-received condition, to 1230, 1275 and 1375 MPa, after aging at 470, 510 and 550 °C, respectively; the as-received and re-heat treated FIMEC curves become similar at increasing aging temperature, and those pertaining to the highest aging temperature are consistent with the slight overaging observed in the re-heat treated samples (Fig. 7). Charpy impact tests were carried out as a function of temperature on the as-received and re-heat-treated samples, the latter being either not aged, or aged at 520 °C for 2 h. (Fig. 9). The laboratory re-heat-treatment noticeably decreases the brittle to ductile transition temperature, which anyway remains above the room temperature (Fig. 9). The fracture surfaces appearance, in the center of the samples, is always brittle and consists mainly of cleavage zones.

The results of the tensile tests performed on the as-received and re-heat treated specimens after aging at 470, 510 and 550 °C for 2.5 h are displayed in Tab. 2 and in Fig. 10. As expected, aging at increasing temperatures yields higher yield and tensile strength, but lower uniform and fracture elongation. Consistently with the aforementioned hardness measurements (Fig. 7), aged as-received and aged re-heat-treated specimens have different uniform elongation, yield and tensile strength, for the same aging temperature, but these differences decrease at increasing aging temperature. On the contrary, the elongation-to-fracture difference increases; in particular, the aged as-received samples fail in a brittle manner, without appreciable necking. The lower toughness of the as-received and aged condition is confirmed by a significant reduction of the fracture toughness after aging, from 70 to 43 MPa/m (Tab. 3). The plane-strain fracture surface of the as-received material
Charpy-V impact tests: brittle-to-ductile transition curves of the as-received, re-heat-treated, and re-heat-treated and aged (at about 520°C for 2 h) metallurgical conditions.

Prove di resilienza Charpy-V: curve di transizione fragile-duttile dei campioni in condizioni metallurgiche di fornitura, r-­i-trattata, e r-­i-trattata ed invecchiata (a circa 520 °C per 2h).

shows mainly cleavage facets (Fig. 11a,b), with small ductile intergranular rupture areas (Fig. 11b); the latter morphology becomes prevalent in the as-received tensile fracture surface, together with some cleavage (Fig. 12).

Overall, the morphology of the tensile fracture surfaces of the different examined aged conditions depend mainly on the metallurgical state before the aging heat treatment. In fact, both the aged and not-aged as-received samples exhibit cleavage areas and ductile intergranular rupture areas (consistently with the lack of necking, Fig. 12a,c,e), whereas the fracture surfaces of the re-heat-treated and aged samples always show a cup-and-cone morphology, with mode-I coalesced micro­voids and mode-II shear areas (Fig. 12b,d).

**DISCUSSION AND CONCLUSIONS**

The microstructure of the examined positions inside the steel bloom consists almost completely of bainite modified by tempering. Therefore, the bloom fracture toughness is low in comparison to usual quenched and tempered steels, being about

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**Fig. 9**
Charpy-V impact tests: brittle-to-ductile transition curves of the as-received, re-heat-treated, and re-heat-treated and aged (at about 520°C for 2 h) metallurgical conditions.

**Fig. 10**
Tensile properties of aged samples (from initial as-received and re-heat treated, RHT, condition). Yield Strength (YS) and Ultimate Tensile Strength (UTS), elongation to fracture (El,) and uniform elongation (Elu).

**Fig. 11**
Plane-strain fracture surfaces in the as-received steel, at the onset of metastable crack propagation [a] and in the crack propagation region [b].

Superfici di frattura in deformazione piana nell'acciaio in stato di fornitura, all'inizio della propagazione instabile [a] e nella regione di propagazione instabile [b].
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70 MPa/m, but somewhat higher in respect to the largely used 1.2738 steel (40 MPa/m on average).

The as-received steel shows a precipitates layer at the previous austenitic grain boundaries, which may be tentatively related with the ductile intergranular fracture observed after the tensile tests in the as-received, both not-aged and aged, samples. Nevertheless, the aged re-heat-treated tensile specimens present fully ductile fracture surfaces, even if they also show a similar precipitates layer at the austenitic grain boundaries after the aging treatment (not after the re-heat-treatment). Therefore, the possible relationship among the grain-boundary precipitates and the intergranular rupture is not yet completely clear.

The as-received specimens are more sensitive to the aging heat treatment than the re-heat-treated ones, since they yield increasingly similar hardness values after aging at increasing temperatures, notwithstanding their lower hardness before aging. This fact may be partially explained by hypothesizing that the initial differences between the bainitic and martensitic matrixes (already tempered at low temperature) are progressively reduced due to coalescence phenomena occurring during tempering at increasing temperatures and interfering with the precipitation during aging.

The FIMEC and tensile tests of aged and not aged samples overall confirm the results of the hardness versus aging duration tests; moreover, the tensile tests evidence a difference in the fracture mode between the as-received and aged samples and those re-heat-treated and aged, the former generally showing brittle fracture surfaces without significant necking, and the latter ductile fracture surfaces and evident necking. Therefore, and also by considering the reduction of fracture toughness (43 MPa/m) of the as-received steel after aging at 525 °C, it is concluded that the aging treatment generally causes a relevant toughness reduction.

Overall, the reported results, and particularly the hardness curves as a function of the aging duration, outline the kinetics of the aging process and constitute a data set that could be employed for the choice of the more suitable parameters (duration and temperature) for the aging treatment of specific molds. In particular, the substantially asymptotic trend of the hardness, as a function of the aging duration, at the examined temperatures and for durations up to 8 h, may allow to obtain homogeneous result also in the aging of molds with large cross-section, for which the actual duration at temperature is necessarily differentiated from surface to core.

Nevertheless, the nature of the metallurgical transformations that originate the hardening process has not been completely determined yet and will be the subject of further studies. A first hint in this direction is given by the observation of the gradual disappearance of the previously existing carbides during the aging treatments, which may be related with the precipitation of different, finer carbides.

ACKNOWLEDGEMENTS

Italian Ministry for University and Research, for financial support by research grant PRIN 2005090102. Lucchini Sideromecanica steelwork, Lovere, Italy, for steel procurement and the CCT diagram.

REFERENCES


Fig. 12

Tensile fracture surface of as-received (a,c,e) and re-heat-treated (b,d) steel, after aging at 550 °C. Overviews (a,b) and details (c,d,e).
ABSTRACT

EFFETTO DEL TRATTAMENTO TERMICO SULLE PROPRIETÀ MECCANICHE DI UN ACCIAIO INDURENTE PER PRECIPITAZIONE PER GRANDI STAMPi PER MATERIE PLASTICHE

Parole chiave: acciaio, precipitazione

Gli stampi per particolari in materia plastica di grande dimensione, quali per esempio paraurti e crusotti, usati nell’industria automobilistica, sono solitamente lavorati per asportazione di truciolo da grandi blocchi di acciaio prebonificato. I blumi di acciaio ISO 1.2738 tradizionalmente usati presentano microstrutture disomogenee e tenacità ridotta ($K_{IC} \approx 40$ MPa√m e $KV \approx 10$ J a temperatura ambiente); inoltre questo acciaio è difficilmente saldabile ($C_{eq} \approx 1,16$). Numerosi acciai indurenti per precipitazione sono stati proposti come alternativa per superare questi limiti.

Siccome i processi di indurimento per precipitazione inducono deformazioni molto limitate, possono essere svolti dopo la lavorazione meccanica. Inoltre, questi processi possono produrre microstrutture e proprietà meccaniche omogenee in stampi grandi e geometricamente complessi.

È stato esaminato un acciaio induribile per precipitazione sviluppato per la fabbricazione di stampi per materie plastiche, con la composizione chimica esposta in Tabella I. Il ciclo produttivo consiste di colata, forgiatura, trattamento termico iniziale (svolto in acciaieria), lavorazione meccanica ed indurimento. Il trattamento termico iniziale consiste di un trattamento preliminare (includendo una de-idrogenazione) seguito da austenitizzazione/solubilizzazione a $1020 \, ^\circ\mathrm{C}$, tempra in aria e doppio rinvecimento a $400 \, ^\circ\mathrm{C}$ (Fig. 1); questo trattamento è rivolto ad ottenere una microstruttura bainitica con durezza compresa tra 310 e 350 HB.

Un blumo di dimensioni originali $500 \times 1500 \times 2400 \, \text{mm}$ è stato esaminato allo stato di fornitura, cioè dopo il trattamento termico iniziale sopra descritto. Inoltre, alcuni campioni sono stati ritrattati in laboratorio come segue: austenitizzazione/solubilizzazione a $1050 \, ^\circ\mathrm{C}$, tempra in acqua, doppio rinvecimento a $400 \, ^\circ\mathrm{C}$, ottenendo martensite rinvenuta. Il rafforzamento per precipitazione è stato studiato usando campioni inizialmente o allo stato di fornitura, o ritrattati in laboratorio, e poi invecchiati a $470$, $510$ o $550 \, ^\circ\mathrm{C}$ per durate fino ad $8 \, \text{h}$.

Le microstrutture sono illustrate nelle Fig. 2, 3 e 4. La dimensione del grano austenitico allo stato di fornitura è di $130 \, \mu\text{m}$. Carburi precipitati presso i bordi di grano austenitici sono presenti allo stato di fornitura, assenti allo stato ri-trattato, e nuovamente presenti allo stato ritrattato ed invecchiato. La quantità di carburi osservati mediante SEM decresce all’aumentare della temperatura e durata dell’invecchiamento (Fig. 5); si ipotizza che i carburi esistenti agli inizi degli invecchiamenti siano gradualmente solubilizzati, permettendo la precipitazione di altri carburi più fini, non osservabili, che causano il rafforzamento. L’analisi XRD dalle seconde fasi, estratte per via elettrochimica (da un campione ri-trattato ed invecchiato a $550 \, ^\circ\mathrm{C}$, Fig. 6), evidenzia la presenza di $\eta$-MoC e la possibile presenza di $V_7C_8$.

Allo stato di fornitura la durezza e le proprietà tensili sono paragonabili a quelle dell’acciaio ISO 1.2738, ma la tenacità a frattura è maggiore (Tab. II e III); la frattura in deformazione piana avviene per clivaggio (Fig. 11). Il ritrattamento termico riduce sensibilmente la temperatura di transizione fragile-duttile (Fig. 9). Gli invecchiamenti incrementano notevolmente la durezza (Fig. 7); l’incremento è maggiore per i campioni allo stato di fornitura, la cui durezza iniziale è minore; per le temperature e durate esaminate il sovrainvecchiamento è assente o trascurabile. Questi risultati sono confermati anche da prove di indentazione strumentata con penetratore cilindrico (FIMEC, Fig. 8). L’invecchiamento a temperature crescenti aumenta le tensioni di snervamento e rottura, coerentemente con l’aumento della durezza (Fig. 10); però i campioni di trazione allo stato di fornitura si rompono in modo fragile, senza apprezzabile strizione (contrariamente a quelli ritrattati ed invecchiati, Fig. 10 e 12). L’effetto avverso dell’invecchiamento sulla tenacità, nel caso di materiale inizialmente allo stato di fornitura, è confermato dalla riduzione della tenacità a frattura ($K_{IC} \approx 43$ MPa√m dopo invecchiamento a $525 \, ^\circ\mathrm{C}$).

Nel loro insieme, i risultati esposti illustrano la cinetica e gli effetti del processo di invecchiamento e costituiscono un insieme di dati utili per scegliere i parametri di invecchiamento più adeguati per specifici stampi.
HOW HEAT TREATMENT CAN GIVE BETTER PROPERTIES TO ELECTROLESS NICKEL-BORON COATINGS

V. Vitry, F. Delaunois, C. Dumortier

Electroless nickel-boron deposits were synthesized on mild steel and submitted to heat treatments under neutral and nitrogen based atmosphere. The properties obtained after these treatments were compared to as deposited nickel-boron coatings. The morphology and structure of the samples were investigated by XRD, SEM and optical microscopy; their composition was studied by ICP, GD-OES and SIMS analysis, and micro and nanoindentation tests were carried out to assess the coatings’ hardness. Scratch tests were used to determine the damage mechanisms of the coating.

KEYWORDS: electroless deposition – nickel-boron – nanoindentation – heat treatment

INTRODUCTION

Autocatalytic (Electroless) nickel plating was discovered by Brenner and Riddel in 1946 [1]. This process is based on the aqueous reduction of nickel salts by a chemical agent thus allowing deposition on non-conducting materials and leading to continuous coatings with a constant thickness [2-5]. Nickel boron coatings are obtained when a boron-based agent, such as sodium borohydride is used to reduce the nickel. Those coatings are of great interest and are extensively studied [6-12]. They present, in their as-deposited state, an hardness close to 750HV100 and are useful in many industries including automotive, electronic and chemical industries because of their good mechanical, chemical and tribological properties [2, 5, 11, 13, 14]. Depending on the amount of boron present, the coatings are considered amorphous, microcrystalline or a mix of the two, the amount of amorphous phases increasing with the amount of boron [2, 7, 15-18].

Heat treatments are often used to enhance the properties of nickel-boron coatings: they allow crystallisation of the amorphous part and, if well designed, lead to nano and microcrystalline structure which are harder than the as-deposited coatings and their hardness can reach 1200HV100 [2, 17, 19].

Much information can be obtained using nanoindentation: this technique is an instrumented indentation and the loading and unloading curves are recorded during each indent. Moreover, the loads are much smaller than in the case of microindentation (typically a few mN) [20-22]. This technique is often used with a Berkovitch indenter which has the same surface than the Vickers indenter while being easier to manufacture owing its triangle-based pyramid shape. Working with very low loads allows to get very small indents and thus to study the hardness evolution across a relatively thin coating.

Scratch test [23-29] can give information about the “practical adhesion” of coatings as well as the degradation modes of the coatings. It consists in the application of an increasing load to a coating. Modern investigation techniques are used to study the coating’s scratch test comportment: acoustic emission, friction coefficient and penetration depth measurements are recorded during the test and microscopic examination is carried out after the test. The critical load of a system which characterizes the adhesive strength of the coating/substrate system is determined from the first adhesive failure. The degradation modes can be identified from observation. However scratch tests cannot be used to predict quantitative wear rates of materials and coatings.

EXPERIMENTAL

Samples preparation

Steel and Aluminium alloy cylinders with a diameter of 25 ± 1 mm and a thickness of 10 ± 1 mm were plated with nickel-boron. Before plating, they were mechanically polished, degreased with acetone and etched in an acid solution. The aluminium samples were subjected to further pre-treatment by double-zincate conversion and acid nickel phosphorous flash deposition. The deposition bath is based on the reduction by sodium borohydride (NaBH4); the nickel ions source is nickel chloride (NiCl2.6H2O). The nickel ions are complexed by ethylene diamine (EN) and lead tungstate (PbWO4) is used as a stabilizer. The operation conditions and the installation have been described elsewhere [15].

Classical heat treatments were carried out under neutral gas flow (95%Ar – 5%H2) at 400°C for 1 hour for steel substrates and at 180°C for 4 hours for aluminium substrates (this temperature was proven by Delaunois et al. to offer a good compromise be-
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Fig. 1
GD-OES depth profile of an untreated nickel-boron coating on Steel.
Profilo GD-OES nello spessore di un rivestimento nichel-boro non trattato su acciaio.

Fig. 2
GD-OES depth profile of a nickel-boron coating on Steel after 1 hour heat treatment at 400°C under neutral atmosphere.
Profilo GD-OES nello spessore di un rivestimento nichel-boro su acciaio dopo 1 ora di trattamento termico a 400°C in atmosfera inerte.

Fig. 3
SEM micrograph of an untreated nickel-boron coating – left: cross section showing the columnar morphology – right: cauliflower-like surface.
Micrografia al SEM di un rivestimento nichel-boro non trattato – sinistra: sezione trasversale che mostra una morfologia colonnare – destra: superficie “a cavolfiore”.

tween coating hardening and substrate softening for aluminium substrates [15]). Preliminary test for other treatments were carried out under ammonia and nitrogen-based atmospheres. Samples prepared for SEM observation and hardness testing were first cut using a Leco Microtom cutting machine with a diamond cutting disk, then mounted in a non-retractable resin and mirror polished.

Samples analysis
Mean chemistry of the samples was investigated after dissolution in concentrated nitric acid by ICP analysis using a Jobin-Yvon apparatus. Profile analysis was carried out by GD-OES using Jobin-Yvon spectrometer and surface analysis by TOF-SIMS using an ION-TOF IV apparatus. A Siemens D500 X-rays \(\theta-\theta\) apparatus applying Cu K\(\alpha\) (1,54 \(\text{Å}\)) radiation was used to study the structure of the samples. Their morphology and thickness were observed using a Philips XL 20 Scanning Electron Microscope.

Microhardness measurements were carried out using a LECO M-400-A, mounted with a Vickers indenter for surface testing and with a Knoop (lozenge-shaped) indenter for cross section testing. A load of 100g was used for Vickers indentation while a load of 50g was used for Knoop hardness testing. The holding time was 20s for both techniques. Nanohardness was obtained with a MTS nano-indenter XP mounted with a Berkovitch (tetrahedron shaped), using depth controlled indentation in order to obtain indents of similar size. The hardness value at a load of 4000\(\mu\text{N}\) was chosen as nanindentation hardness value.

Scratch tests were performed on selected samples using the continuous load increase method up to 30N with a Microphotonics Micro Scratch Tester (MST), with a load rate of 19.17N/min and an advance rate of 9.58mm/min, resulting in a scratch of 15mm. The tip was a Rockwell C diamond stylus indenter with a radius of 200 \(\mu\text{m}\).

RESULTS AND DISCUSSION

Chemistry of deposits
ICP analysis of as-deposited coatings showed that their average composition is 93 wt. % nickel, 6 wt. % boron and 1 wt. % lead. Heat treatments do not modify the global composition of the coatings. GD-OES analysis, on steel substrate, allowed us to follow qualitatively the composition into the depth of the coating. In the as-plated state (Fig. 1), the boron and nickel content of the coating don’t vary with the depth while the lead content seems to be higher at near the substrate interface then decreases slightly before increasing once more. It means that more lead is deposited at the very beginning of the process and at the end that during the fast “regime” deposition of the coating.

Observation of the interface allows us to predict a good adhesion of the coating because there seems to be a certain amount of interdiffusion with the substrate. After heat treatment at 400°C for one hour under neutral atmosphere (Fig. 2), the nickel and boron content are quite unmodified while the lead content becomes higher near the free surface of the sample and decreases steeply with depth. The lead seems to diffuse outside the coating. However, the interface between the steel substrate and nickel-boron was not modified by this treatment. Previous work revealed important interdiffusion at the interface after heat treatment in the case of aluminium-silicon alloys (AS7G06) [15].
Positive and negative ions SIMS analysis was carried out on the untreated samples and revealed only the presence of the known constituent of the coating (Nickel, boron, lead) and of the classic surface contamination. The results obtained after neutral atmosphere heat treatment were similar, proving that the coating’s chemistry is not much influenced by those treatments.

Structure and morphology of the coatings

In the as-deposited state, the coatings present a columnar morphology and a cauliflower-like surface (which is characteristic of nickel-boron coatings [11,15,17]), as can be seen on Fig. 3. Neutral atmosphere heat treatments up to 400°C do not modify those properties. The structure of untreated samples and samples treated at 180°C revealed they were amorphous (Fig. 4) while crystallization occurred during heat treatment at 400°C. This is expected from the literature and our own previous results [2,15,19,30]. The effect of this crystallization on the mechanical properties of the coating will be discussed later.

Mechanical properties of the coating

Vickers hardness testing on the unprocessed surface of the sample is the standard method to measure hardness of nickel-boron coatings. However, we find it disputable because the surface is unprepared and its smoothness is unwarranted, and because the substrate hardness may significantly influence the results when the applied load is too high. We thus used other hardness testing methods to free ourselves from those potential problems: Knoop microindentation and Berkovitch nanoindentation were used on polished cross-sections. Nanoindentation were converted from GPa into Berkovitch hardness points (equivalent to Vickers values) to facilitate comparison. Hardness values for untreated samples were close to 825 for all techniques. After heat treatment at 180°C for 4 hours, those values reached 1000, and they were further increased after heat treatment at 400°C. The first increase is caused by short order rearrangement in the coating (the amorphous dome XRD intensity is slightly higher), while the crystallization observed between 180°C and 400°C causes a far greater hardness enhancement and reaches the maximum hardness value for nickel-boron coatings [2,17,19]. It is due to the generation of a high density of grain boundaries inside the coating. The hardness can thus be optimized by the grain-size control: if the grains are allowed to coalesce (i.e. if the heat treatment is too long or the temperature too high), the hardness of the coating will decrease [16,17,31].

It was not possible to obtain a reliable Knoop hardness value after treatment at 400°C because cracking of the coating occurred. This shows the importance of comparing values from different techniques: Nickel boron coatings have a very anisotropic structure, due to their synthesis mode. Vickers hardness is carried out in the growth direction of the coating and any damage occurring during this test will remain unseen because it will take place inside the coating. However, Knoop indentation is made perpendicular to the growth direction of the coating and the subsequent damage is easily observable (Fig. 5). Knoop indentation is thus more reliable. This may also explain why na-

<table>
<thead>
<tr>
<th></th>
<th>Untreated</th>
<th>4h ; 180°C</th>
<th>1h ; 400°C</th>
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</thead>
<tbody>
<tr>
<td>Knoop microhardness (hk50)</td>
<td>834 ±20</td>
<td>927 ± 30</td>
<td>-</td>
</tr>
<tr>
<td>Vickers microhardness (hv100)</td>
<td>854 ± 40</td>
<td>1014 ± 40</td>
<td>1302 ± 40</td>
</tr>
<tr>
<td>Berkovitch nanoindentation (4000μN)</td>
<td>823± 155</td>
<td>1140 ± 75</td>
<td>1584 ± 182</td>
</tr>
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Tab. 1

Vickers and Knoop and Berkovitch hardness values of nickel-boron coatings on aluminium alloys.

Valori di durezza Vickers, Knoop e Berkovitch di rivestimenti di nichel-boro su leghe di alluminio.

X-Ray diffraction patterns of Electroless nickel-boron coatings on aluminium substrates with and without heat treatments.

Spettri di diffrazione ai raggi X per i rivestimenti chimici di nichel-boro su substrati di alluminio con e senza trattamenti termici.
Trattamenti termici

noindentation values are so much higher than Vickers values for 400°C treatment: the load applied for the nanoindentation test is too low to generate damage.
The small size of nanoindentation indents allows measuring the hardness of the coating on several points across the coating without any interaction of the indents. We were thus able to draw hardness profiles for nickel-boron coatings (fig. 6). Those profiles showed that the hardness values are not homogeneous across the coating except after heat treatment at 180°C. This is probably due to the fact that as-deposited coatings have important internal stress. Heat treatment at 180°C releases a lot of this stress and the hardness becomes homogeneous. Heat treatment at 400°C however may generate new internal stresses because it is accompanied by important structural modifications. Moreover, as nanoindentation is a very sensitive experimental method it is possible hat part of the values scattering is due to external factors such as roughness of the sample (microscratches in the polished cross section), geometry of the indenter tip, thermal drift and creep.
The usual thickness used for nickel-boron coatings is at least 15-25 micrometers. For coatings of this thickness, scratch tests up to 25N did not reveal any significant damage. We decided thus to work with thinner coatings (with a thickness of 6μm) in order to determine the damage mechanisms observed during scratch test. These tests were only carried out without heat treatment and after heat treatment at 180°C.
The damage mechanisms were the same for both treated (Fig. 8) and untreated (Fig. 7) coatings which is not surprising knowing they present roughly the same structure and morphology. The first failure that was observed is longitudinal cracking on the edges of the coating. Chevron cracks were then observed. Those are cohesive damage. The first adhesive failure is discontinuous ductile perforation of the coating, which is followed, in the case of heat treated samples, by continuous ductile perforation of the coating. To observe this kind of failure is really encouraging because it is a proof of the good adhesion of the coating, which was first suggested by the interface observation by GD-OES. Moreover, this failure was only observed because the coating was so thin.

Effects of alternative post-treatment
Alternative heat treatments are now in their testing phase. They are mainly based on the use of reactive atmospheres. We present hereunder some interesting results we obtained using (i) a treatment under a reduced pressure in a nitrogen-based gas, which will be called “vacuum treatment” in further explanations and (ii) a treatment under an ammonia-based atmosphere, which will be called “ammonia treatment”.
SEM cross section micrographs of nickel-boron coatings after “vacuum” and “ammonia” treatments showed an important morphological modification of the coating: it becomes totally dense after “vacuum” treatment and the columnar morphology disappears completely (Fig. 9a), and the coating is composed

Fig. 6
Nanoindentation profiles on treated and untreated nickel-boron coatings.
Profili di nanodurezza su rivestimenti nichel-boro trattati e non trattati.

Fig. 7
Micrograph of scratches on 6μm thick nickel-boron coatings (aluminium alloy substrate) without heat treatment.
Micrografia di scalfitture su rivestimenti nichel-boro dello spessore di 6μm (substrato in lega di alluminio) senza trattamento termico.

Fig. 8
Micrograph of scratches on 6μm thick nickel-boron coatings (aluminium alloy substrate) after 4 hours at 180°C under 95% Ar + 5% H2 atmosphere.
Micrografia di scalfitture su rivestimenti nichel-boro dello spessore di 6μm (substrato in lega di alluminio) dopo 4 ore a 180°C in atmosfera 95% Ar + 5% H2.
of 2 distinct layers after “ammonia” treatment (Fig. 9b). The inner layer is dense and resembles the “vacuum” treated coating while the outer layer looks porous. Knoop hardness measurements were carried out on “vacuum” treated samples and on the dense part of “ammonia” treated samples. Values of 1570 ± 100 HK₁₀₀ and 1630 ± 100 HK₁₀₀ respectively were obtained instead of 1250 ± 100 HK₁₀₀ after treatment at 400°C. It shows that the hardness of nickel-boron coatings can be further enhanced by the use of modified heat treatments. Scratch tests were carried out on “ammonia” treated coatings. Those coatings, while they are plastically deformed, are nearly undamaged after the tests (Fig. 10) which is promising for wear applications.

CONCLUSIONS

As-deposited nickel-boron coatings possess several interesting features: high hardness (~825HV₁₀₀), good scratch comportment, amorphous structure, columnar morphology and cauliflower-like surface. Heat treatment influences some of those features, mostly in a positive way:
- The columnar morphology is unmodified by classical heat treatment up to 400°C but is transformed in a dense layer that can be accompanied by a porous outer layer after the alternative treatments we investigated.
- The amorphous structure undergoes crystallization during heat treatment if the temperature is high enough: there is no crystallization for 180°C treatments while crystallization is complete after treatment at 400°C.
- The hardness of the coating is very much influenced by its crystalline state: while low temperature treatment induces a slight increase and an homogenisation of the hardness, the treatment at 400°C leads to a hardness value of 1300HV₁₀₀. This high values is due to the important grain-boundaries density that is obtained after heat treatment.
- Alternative heat treatment allowed a further hardness increase by a still unidentified mechanism.
- The scratch comportment of nickel-boron coatings is quite unmodified by neutral heat treatments. The comportment after ammonia-based alternative treatment is mainly plastic deformation of the outer layer of the coating, which is very interesting for wear applications.

REFERENCES

COME IL TRATTAMENTO TERMICO PUÒ MIGLIORARE LE PROPRIETÀ DEI RIVESTIMENTI CHIMICI DI NICHEL-BORO.

Parole chiave: trattamenti termici, rivestimenti, acciaio

Deposizioni chimiche di nichel-boro sono state realizzate su un acciaio dolce e sono state poi sottoposte a trattamenti termici in atmosfera inerte o a base di azoto. Le proprietà ottenute dopo questi trattamenti sono state confrontate con quelle dei rivestimenti nichel-boro di partenza. La morfologia e la struttura dei campioni sono state esaminate mediante XRD, microscopia ottica e SEM; la loro composizione è stata studiata mediante analisi ICP, GD-OES e SIMS; infine sono state eseguite prove di micro-e nano-durezza per valutare la durezza dei rivestimenti. Per determinare i meccanismi di danneggiamento del rivestimento sono stati utilizzati prove di resistenza alla scalpittura.

IMPIEGO DI PROTOSSIDO D’AZOTO NEL TRATTAMENTO TERMOCHIMICO DI NITRURAZIONE GASSOSA: STUDIO DEI PROCESSI PRODUTTIVI E CARATTERIZZAZIONE METALLURGICA

M. Merlin, P. Camanzi, G. L. Garagnani

In questo lavoro sono presentati i risultati di uno studio effettuato sul trattamento termochimico di nitrurazione gassosa, in particolare sulla possibilità di ottenere un aumento della velocità di processo a livello industriale. È stato testato l’effetto dovuto all’introduzione, insieme all’ammoniaca anidra (NH₃), di protossido di azoto (N₂O) nell’atmosfera di processo. Inizialmente sono stati caratterizzati i normali cicli produttivi di nitrurazione gassosa in atmosfera di ammoniaca anidra, inserendo campioni identici di acciaio di tipo 42CrMo4 all’interno delle cariche di materiale da nitrurare nei forni a pozzo. I cicli produttivi di interesse sono stati quelli aventi come obiettivo finale l’ottenimento di uno strato di indurimento superficiale di 3 e 4 decimi di millimetro. Al termine della sperimentazione si è constatato che l’utilizzo dell’N₂O comporta un incremento della cinetica di processo, con una conseguente riduzione del 20÷30% dei tempi di processo rispetto al ciclo produttivo standard. Nonostante la riduzione dei tempi di processo, le caratteristiche meccanico-microstrutturali dei campioni trattati sono risultate confrontabili con il processo tradizionale, prospettando l’idoneità all’applicazione industriale della nitrurazione gassosa con impiego di protossido d’azoto. Alla sperimentazione e raccolta dei dati ottenuti segue una valutazione economica e di fattibilità per i cicli produttivi testati, sulla base di un confronto qualitativo con i processi già applicati industrialmente.

PAROLE CHIAVE: protossido d’azoto, indurimento superficiale, metallografia, nitrurazione gassosa

INTRODUZIONE

Le odierne esigenze produttive nel campo della meccanica portano a richieste sempre maggiori di elevate caratteristiche tecniche degli organi meccanici progettati. L’obiettivo infatti è quello di consentire a tali particolari un impiego in condizioni di esercizio particolarmente onerose in termini di tensioni meccaniche, di usura o di corrosione. Spesso la tecnologia produttiva che porta all’ottenimento dei particolari meccanici non consente di lavorare agevolmente metalli con determinate caratteristiche; basti pensare, ad esempio, alle lavorazioni per asportazione di truciolo eseguite su materiali ad elevata durezza. Inoltre, sono frequenti i casi in cui le caratteristiche richieste per l’utilizzo non sono nemmeno riscontrabili nei materiali di maggiore utilizzo, alle condizioni di normale fornitura. Pertanto si rende spesso necessaria l’esecuzione di trattamenti termici e termochimici per conferire al componente le dovute proprietà meccaniche. In particolare, i trattamenti termochimici, in modo tale da alterare superficialmente la composizione chimica, oltre che quella strutturale, del materiale iniziale. La nitrurazione gassosa è un trattamento termochimico di indurimento superficiale che viene condotto a temperature comprese tra i 480°C e i 570°C per un periodo di tempo variabile da qualche ora a parecchie decine di ore, in condizioni tali da consentire una diffusione di azoto nella superficie del particolare da trattare. Al termine del trattamento il pezzo viene raffreddato a temperatura ambiente mediante un’atmosfera gassosa di tipo inerte. Gli strati superficiali così generati sono caratterizzati da un ottima resistenza all’usura adesa, al grippaggio e all’abrasione meccanica. La nitrurazione può essere eseguita su numerosi tipi di acciaio, anche se le caratteristiche di durezza superficiale sono massime per gli acciai legati contenenti alluminio, cromo e vanadio. I più diffusi acciai
da nitrurazione in commercio sono, secondo la Norma UNI EN 10085, il 31CrMo12 e il 41CrAlMo7, a cui si aggiungono anche il 31CrMoV9 e il 34CrAlMo5. Non sempre durezze eccessivamente elevate sono desiderabili per impieghi costruttivi e pertanto il trattamento di nitrurazione gassosa può essere eseguito anche su acciai più basso legati [1-9]. Un esempio tipico è costituito dall’acciaio da bonifica basso legato 42CrMo4, che viene normalmente utilizzato per la realizzazione di molti organi meccanici in quanto presenta buone proprietà di tenacia e resistenza. Su tali componenti in 42CrMo4 spesso viene richiesto un trattamento superficiale di nitrurazione gassosa, pur non essendo un acciaio tipicamente “da nitrurazione”.

I normali cicli di nitrurazione gassosa per elevata profondità di indurimento comportano tempi di impegno impianto molto elevati. Pertanto, è stata valutata la possibilità di incrementare la velocità di processo tramite l’impiego di uno specifico gas di processo addizionale: il protossido di azoto (N₂O). L’effetto del protossido d’azoto è stato tradotto in una maggiore capacità nitrurante dell’atmosfera e quindi in un abbassamento dei tempi di processo. I risultati sperimentali di molteplici autori [10-24] mettono in evidenza questi effetti. In particolare, da [17, 23-24] si può concludere che una addizione di ossigeno all’atmosfera nitrurante porta ad una azione benefica, specialmente per acciai legati e contenenti cromo. Si è incominciato a sfruttare questi principi già dalla metà degli anni ottanta per acciai legati e contenenti cromo; si è successivamente dimostrato che l’impiego di protossido d’azoto può essere utilizzato per agevolare una formazione di eccessivo spessore di nitruri che non è desiderabile per applicazioni raffinate. In particolare, da [17, 23-24] si può concludere che una addizione di ossigeno all’atmosfera nitrurante porta ad una azione benefica, specialmente per acciai legati e contenenti cromo. Si è incominciato a sfruttare questi principi già dalla metà degli anni ottanta per acciai legati e contenenti cromo; si è successivamente dimostrato che l’impiego di protossido d’azoto può essere utilizzato per agevolare una formazione di eccessivo spessore di nitruri che non è desiderabile per applicazioni raffinate.

La scelta del materiale da utilizzare nell’indagine sperimentale è stata effettuata a seguito di una attenta analisi dei materiali più frequentemente trattati presso Siderit s.r.l.. È stato scelto l’acciaio 42CrMo4, la cui composizione nominale è riportata in Tab. 1. Tale acciaio, pur non essendo un tipico acciaio da nitrurazione, è comunque in grado di diffondere azoto fino a spessori efficaci elevati ed è ampiamente utilizzato per la costruzione di particolari meccanismi soggetti a sollecitazioni cicliche e a sfregamenti (mozzi, perni, alberi, camme, ecc.). Sono stati presi in considerazione due cicli produttivi standard, nominati NT3 ed NT4, relativi al trattamento di nitrurazione gassosa che consente di ottenere uno spessore efficace rispettivamente di 0.3 mm e 0.4 mm, con tolleranza ±0.1 mm. La prima fase del lavoro ha riguardato la campionatura sistematica di tali cicli produttivi, al fine di ricavarne la efficienza e la ripetibilità nelle diverse condizioni di processo. In Fig. 1. I cicli di nitrurazione gassosa standard sono composti da due fasi: la prima, detta di “arricchimento”, si svolge a 500°C, mentre la seconda, detta di “dissoluzione”, si svolge a 540°C. Nelle diverse fasi del trattamento sono stati analizzati sistematicamente i valori del grado di dissociazione dell’ammoniaca.

**MATERIALI E METODI**

La scelta del materiale da utilizzare nell’indagine sperimentale è stata effettuata a seguito di una attenta analisi dei materiali più frequentemente trattati presso Siderit s.r.l.. È stato scelto l’acciaio 42CrMo4, la cui composizione nominale è riportata in Tab. 1. Tale acciaio, pur non essendo un tipico acciaio da nitrurazione, è comunque in grado di diffondere azoto fino a spessori efficaci elevati ed è ampiamente utilizzato per la costruzione di particolari meccanismi soggetti a sollecitazioni cicliche e a sfregamenti (mozzi, perni, alberi, camme, ecc.).

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moniaca. I cicli standard sono stati modificati ed i cicli sperimentali sono stati nominati rispettivamente NT3-N₂O ed NT4-N₂O per metter in evidenza l’impiego del protossido d’azoto.

Le modifiche, rispetto ai cicli standard, hanno previsto un pre-riscaldamento del materiale a 380°C per uniformare il più possibile la temperatura dei pezzi, uno step di preossidazione del materiale, allo scopo di attivare la superficie preventivamente all’arricchimento, e l’inserimento del protossido d’azoto nella successiva fase di arricchimento. Si è deciso di mantenere una proporzione tra il protossido d’azoto e l’ammoniaca del 2-3%, lasciando inalterata l’atmosfera nitrurante nella fase di diffusione. L’effetto della preossidazione dell’acciaio in atmosfere nitruranti si esplica in un aumento della profondità di indurimento [14, 21]. La campionatura di ogni ciclo termochimico è stata effettuata inserendo provini cilindrici del materiale sopra descritto all’interno delle cariche di produzione. Le dimensioni dei provini sono di 20 mm di spessore e diametro di 35 mm, ricavati dal taglio di una barra cilindrica dello stesso diametro trattata termicamente con processo di bonifica. La durezza media del materiale base è di 310÷320 HV1. Prima di eseguire la nitrurazione, i pezzi sono stati accuratamente puliti per eliminare ossidi ed eventuali lubrificanti che possono alterare le condizioni di assorbimento di azoto sulla superficie; inoltre, sulle facce da nitrurare ogni campione è stato preventivamente trattato con operazioni di sgrossatura e successivamente finite con lapidellatura accurata. Tutti i campioni, trattati secondo i cicli standard e sperimentali con protossido d’azoto, sono stati successivamente sottoposti a caratterizzazione metallografica e a misure di durezza superficiale, di gradiente di durezza e di spessore della coltre bianca. Il parametro di confronto più importante che è stato preso in considerazione è lo “spessore efficace”, definito dalla Norma UNI 5478.

CARATTERIZZAZIONE MECCANICA E MICROSTRUTTURALE

I campioni, dopo essere stati estratti dai forni di trattamento, sono stati accuratamente sezionati secondo le sezioni longitudinali e trasversali, e preparati per le indagini di laboratorio. Tutte le successive analisi microstrutturali e meccaniche sono state eseguite con strumentazioni dedicate e seguendo i principali riferimenti normativi [27-28].

Prove di microdurezza

Un’accura indagine sclerometrica è stata eseguita sui campioni in modo da ricavare i dati utili alla caratterizzazione del trattamento termochimico. Sulle sezioni trasversali sono state effettuate numerose prove di durezza per definire il gradiente. I risultati ottenuti sono stati raccolti in fogli di calcolo ed espressi graficamente (Fig. 2). Tenendo in considerazione il valore medio della durezza a nucleo, è possibile individuare la profondità efficace ottenuta, definita dalla norme UNI 5478, come quel valore di durezza superiore di 100 unità HV rispetto alla durezza a nucleo.

L’indagine sclerometrica è stata estesa anche alla superficie esterna direttamente nitrurata, con l’analisi della durezza superficiale. Per ogni campione sono state eseguite le prove di durezza superficiale HV03, HV05, HV1, HV5, HV10, HV30. L’esecuzione di prove di durezza superficiale con carico di prova sempre maggiore è utilizzata per verificare a quale di
questi carichi e quindi indirettamente a quale pressione specifica, si abbia un cedimento per fragilità della coltre bianca, e quindi una possibile scheggatura. È stato riscontrato che l’influenza della coltre bianca sulla durezza risulta evidente per carichi fino al 1kg.

**Analisi metallografica**

Oltre alle prove sclerometriche è stata effettuata una indagine metallografica per valutare lo spessore della coltre bianca e la microstruttura; si è agito eseguendo un attacco chimico sulla superficie accuratamente lucidata dei provini inglobati con una soluzione di alcool puro e acido nitrico al 2% (NITAL 2). Dalla Fig. 3 si nota la presenza di nitruri di forma lamellare, la cui quantità è maggiore vicino al bordo esterno e diminuisce verso la zona interna. Non sono state rilevate strutture reticolari, particolarmente dannose per la resistenza meccanica del pezzo nitrurato.

**ANALISI DEI RISULTATI E DISCUSSIONE**

I risultati di interesse, accuratamente elaborati e discussi, riguardano i valori di durezza superficiale, il valore della profondità efficace e la quantità di coltre bianca conferita dal trattamento. I valori sono stati mediati sul numero di prove eseguite, cercando di tenere conto dei molteplici fattori di influenza sul trattamento.

**Campionatura cicli standard**

Seguendo le metodologie descritte nei precedenti paragrafi, sono stati campionati inizialmente i cicli produttivi già standardizzati dall’azienda. I cicli oggetto di analisi sono stati quelli aventi come obiettivo il raggiungimento di un valore di profondità efficace di indurimento di 0,3 mm (NT3) e di 0,4 mm (NT4). Questi cicli vengono differenziati in funzione dell’impianto osservato.

**Campionatura cicli con protossido d’azoto**

Durante l’esecuzione della campionatura dei cicli standard si sono affrontate anche le prime sperimentazioni relative al protossido di azoto. La sperimentazione è stata soggetta ad evoluzioni per quanto riguarda la conformazione dei cicli sperimentali applicati, in considerazione del fatto che il materia-
ta all’uso del programma (P.29) che garantisce una profondità efficace di 0.45 mm. Di queste considerazioni si terrà conto nel confronto tra i tempi ciclo, nel paragrafo dedicato a questo tipo di analisi.

Per l’impianto 2 i risultati ottenuti con i cicli standard NT4 e con quelli sperimentali NT4-N2O sono perfettamente comparabili (Fig. 5b). Il ciclo sperimentale (P.29) fornisce profondità efficaci di 0.45 mm comparabili con i cicli standard (P.15).

I cicli sperimentali per la classe di trattamento NT3 hanno anch’essi dato buoni risultati nel raggiungimento della profondità efficace, anche se la profondità efficace raggiunta dai cicli standard è leggermente più elevata per entrambi gli impianti (Fig. 6). Questi risultati possono essere legati al fatto che la diminuzione percentuale di tempo apportata ai cicli NT3 standard, per entrambi gli impianti, è leggermente superiore rispetto alla diminuzione apportata ai cicli NT4. Inoltre, la sperimentazione relativa ai cicli di trattamento tipo NT3 è stata più limitata per via del fatto che i due impianti oggetto della sperimentazione stessa sono prevalentemente impiegati per l’esecuzione di trattamenti di nitrurazione profonda NT4. È stato osservato che i trattamenti sperimentali hanno garantito una buona ripetibilità dei gradienti di durezza.

È stato osservato che il protossido di azoto, accelerando la cinetica di nitrurazione, ha una influenza sulla formazione e sulla crescita dei strati induriti. Si è verificato che un uso inadeguato dello stesso protossido porta a spessori della coltre bianca troppo elevati, e quindi indesiderabili, pertanto deve essere ben controllato per non portare a risultati finali non voluti. Per i due diversi impianti, si riporta di seguito la statistica relativa agli spessori della coltre bianca e dello strato poroso; sono messi in evidenza i valori medi ottenuti con i vari programmi di nitrurazione, e vengono confrontati con i cicli omologhi che

**Grafico di confronto della durezza superficiale ottenuta negli impianti 1 e 2:** (a) per i cicli NT4 ed NT4-N2O, (b) per i cicli NT3 ed NT3-N2O.

Surface hardness comparison obtained in implants 1 and 2: (a) NT4 and NT4-N2O cycles, (b) NT3 and NT3-N2O cycles.

**Confronto tra i gradienti di durezza medi per cicli NT4 ed NT4-N2O:** (a) impianto 1, (b) impianto 2.

Average hardness gradient comparison for NT4 and NT4-N2O cycles: (a) implant 1, (b) implant 2.

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Trattamenti termici

In Tab. 4 si può notare che i valori medi degli spessori delle coltri bianche ottenute con i cicli che prevedono l’impiego di protossido sono entro limiti più che accettabili, seppure leggermente superiori, ma del tutto confrontabili, con i rispettivi cicli omologhi che non impiegano protossido. In ogni caso gli spessori di coltre bianca rilevati non sono mai risultati superiori ai limiti di accettabilità imposti dalla norma UNI 5478. Nei grafici di Fig. 8 è riportata la dispersione dei dati relativi agli spessori delle coltri bianche e degli strati porosi rilevati nella sperimentazione per due cicli di nitrurazione NT4 ed NT4-N2O eseguiti nell’impianto 1.

Durata dei trattamenti – Confronto tra cicli omologhi
La sperimentazione ha evidenziato la possibilità di ridurre i tempi-ciclo per i trattamenti analizzati. Le progressive piccole variazioni dei cicli hanno permesso di ottimizzare i tempi-ciclo in funzione del risultato richiesto dalla produzione aziendale. Il confronto dei tempi-ciclo deve essere effettuato tenendo in considerazione il risultato finale ottenuto. In Fig. 9 si riportano

<table>
<thead>
<tr>
<th>TRATTAMENTO</th>
<th>NT4</th>
<th>NT4-N2O</th>
<th>NT4</th>
<th>NT4-N2O</th>
<th>NT3</th>
<th>NT3-N2O</th>
<th>NT3</th>
<th>NT3-N2O</th>
</tr>
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<tbody>
<tr>
<td>Impianto</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>1</td>
<td>2</td>
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</tr>
<tr>
<td>Programma</td>
<td>24</td>
<td>29</td>
<td>15</td>
<td>29</td>
<td>6</td>
<td>11</td>
<td>6</td>
<td>11</td>
</tr>
<tr>
<td>Coltre bianca [μm]</td>
<td>13.5</td>
<td>14.5</td>
<td>12.5</td>
<td>13</td>
<td>11</td>
<td>13.5</td>
<td>10</td>
<td>12.5</td>
</tr>
<tr>
<td>Strato poroso [μm]</td>
<td>7</td>
<td>7.5</td>
<td>6</td>
<td>7.5</td>
<td>4.5</td>
<td>7.5</td>
<td>5</td>
<td>6.5</td>
</tr>
</tbody>
</table>

Fig. 6 Confronto tra i gradienti di durezza medi per cicli NT3 ed NT3-N2O: (a) impianto 1, (b) impianto 2.
Average hardness gradient comparison for NT3 and NT3-N2O cycles: (a) implant 1, (b) implant 2.

Fig. 7 Ripetibilità dei gradienti di durezza per i trattamenti sperimentali NT4-N2O: (a) impianto 1, (b) impianto 2.
Repeatability of hardness gradients for NT4-N2O innovative treatments: (a) implant 1, (b) implant 2.

Tab. 4 Riassunto dei dati relativi agli spessori di coltre bianca ottenuta per i cicli di trattamento NT4 ed NT4-N2O, NT3 ed NT3-N2O.
Data referred to the white layer thicknesses in NT4, NT4-N2O, NT3 and NT3-N2O treatment cycles.
a confronto i tempi-ciclo per i programmi (P.24) relativo al trattamento NT4, e i programmi (P.28) e (P.29) per il trattamento NT4-N\textsubscript{2}O; si può notare che il ciclo derivato dal programma (P.29) riduce drasticamente il tempo di svolgimento del trattamento, facendo risparmiare 30 h; tuttavia la profondità efficace ottenibile con tale ciclo è inferiore rispetto al ciclo standard, da quanto rilevato in 4.3.2. Si riporta in Tab. 5 un confronto tra i tempi-ciclo, posti in relazione a parità di risultato ottenuto. Per i cicli NT4-N\textsubscript{2}O il risparmio di tempo-ciclo è evidente e può essere considerato attorno al 20÷25%, rispetto ai cicli standard, tenendo dovutamente conto degli errori sperimentali. Per quanto riguarda i cicli NT3-N\textsubscript{2}O anche in questo caso si riscontra un marcato risparmio di tempo, addirittura superiore rispetto ai cicli più lunghi. Questo potrebbe essere dato dal fatto che l’adsorbimento di azoto, e quindi la nitrurazione, non ha un andamento lineare con il procedere del trattamento; infatti, l’ispessimento della coltre bianca e l’allontanamento del fronte di adsorbimento di azoto rendono più difficoltoso il passaggio dell’azoto stesso. Dunque la capacità di adsorbimento di azoto cala con lo sviluppare del trattamento, e i cicli più corti risentono maggiormente del favorevole impiego del protossido di azoto. Da non sottovalutare è inoltre la possibile diversa risposta del forno all’impiego del protossido di azoto; la diversa capacità dissociativa è dipendente anche dalla geometria della storta, nonché dal moto di ricircolo al suo interno. È possibile che le diversità tra gli impianti portino a diverse efficienze a confronto i tempi-ciclo per i programmi (P.24) relativo al trattamento NT4, e i programmi (P.28) e (P.29) per il trattamento NT4-N\textsubscript{2}O; si può notare che il ciclo derivato dal programma (P.29) riduce drasticamente il tempo di svolgimento del trattamento, facendo risparmiare 30 h; tuttavia la profondità efficace ottenibile con tale ciclo è inferiore rispetto al ciclo standard, da quanto rilevato in 4.3.2. Si riporta in Tab. 5 un confronto tra i tempi-ciclo, posti in relazione a parità di risultato ottenuto. Per i cicli NT4-N\textsubscript{2}O il risparmio di tempo-ciclo è evidente e può essere considerato attorno al 20÷25%, rispetto ai cicli standard, tenendo dovutamente conto degli errori sperimentali. Per quanto riguarda i cicli NT3-N\textsubscript{2}O anche in questo caso si riscontra un marcato risparmio di tempo, addirittura superiore rispetto ai cicli più lunghi. Questo potrebbe essere dato dal fatto che l’adsorbimento di azoto, e quindi la nitrurazione, non ha un andamento lineare con il procedere del trattamento; infatti, l’ispessimento della coltre bianca e l’allontanamento del fronte di adsorbimento di azoto rendono più difficoltoso il passaggio dell’azoto stesso. Dunque la capacità di adsorbimento di azoto cala con lo sviluppare del trattamento, e i cicli più corti risentono maggiormente del favorevole impiego del protossido di azoto. Da non sottovalutare è inoltre la possibile diversa risposta del forno all’impiego del protossido di azoto; la diversa capacità dissociativa è dipendente anche dalla geometria della storta, nonché dal moto di ricircolo al suo interno. È possibile che le diversità tra gli impianti portino a diverse efficienze tab. 5
    ▲

<table>
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<tr>
<th>TRATTAMENTO</th>
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<th>NT4-N\textsubscript{2}O</th>
<th>NT4</th>
<th>NT4-N\textsubscript{2}O</th>
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<td>11</td>
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<td>1</td>
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<td>1</td>
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<tr>
<td>Tempo ciclo [h]</td>
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<td>62</td>
<td>65</td>
<td>52</td>
<td>45</td>
<td>32</td>
<td>45</td>
<td>32</td>
</tr>
<tr>
<td>Profondità efficace [mm]</td>
<td>0,5</td>
<td>0,5</td>
<td>0,45</td>
<td>0,45</td>
<td>0,4</td>
<td>0,38</td>
<td>0,4</td>
<td>0,38</td>
</tr>
<tr>
<td>Risparmio tempo</td>
<td>25%</td>
<td>20%</td>
<td>29%</td>
<td>27%</td>
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</tbody>
</table>

Fig. 8
    ▲

Spessori della coltre bianca e del relativo strato poroso per i cicli di nitrurazione a) NT4 e b) NT4-N\textsubscript{2}O in impianto 1.

Thicknesses of the white layer and of the corresponding nitrides substrate for nitriding cycles a) NT4 and b) NT4-N\textsubscript{2}O in implant 1.

Fig. 9
    ▲

Grafici temperatura-tempo di confronto per alcuni programmi di nitrurazione NT4 e NT4-N\textsubscript{2}O.

Temperature-time diagrams: comparison between NT4 and NT4-N\textsubscript{2}O nitriding programs.
Trattamenti termici

Dei cicli sperimentali. Nei grafici di Fig. 10 si pone in evidenza il risparmio sui tempi-ciclo, ottenuto con la sperimentazione, per i singoli impianti.

VALUTAZIONI ECONOMICHE

Analisi dei costi per i cicli produttivi

Questa analisi si basa essenzialmente sul confronto tra i costi di processo dei cicli per i trattamenti standard e per quelli che prevedono l’uso del protossido d’azoto, prendendo in considerazione, per quanto possibile, i fattori maggiormente incidenti. Vi sono fattori energetici, relativi al dispendio/contenimento dei gas tecnici di processo, e fattori legati alla pianificazione della produzione.

Costi dei gas di processo

La stima dei costi dei gas di processo nei cicli di trattamento è stata condotta calcolando la quantità di gas tecnici impiegati, almeno teoricamente, nei trattamenti omologhi. Sono stati presi in considerazione i parametri relativi ai costi e ai pesi specifici dei gas tecnici riportati in Tab. 6.

È stato sviluppato un algoritmo di calcolo che in funzione dei tempi e sulle portate dei gas di processo è in grado di valutare il risparmio di un ciclo rispetto all’altro. Analizzando con tale algoritmo il costo del ciclo NT4 standard secondo il programma (P.24) e del ciclo sperimentale NT4-N₂O secondo il programma (P.28), che hanno dato in fase di sperimentazione risultati operativi analoghi, è stato stimato un risparmio percentuale pari al 18% per il trattamento sperimentale. Nonostante, infatti, il maggiore costo specifico del protossido di azoto rispetto all’ammoniaca, il ciclo sperimentale ha un minore costo complessivo rispetto a quello standard grazie all’abbattimento dei tempi-ciclo.

Costi dell’energia elettrica

La riduzione dei tempi-ciclo porta anche ad un minor dispendio in termini di energia elettrica spesa. Sono stati presi in considerazione i costi energetici delle varie fasi di riscaldamento e di mantenimento per i cicli standard e per quelli sperimentali con protossido.

Ponendo a confronto i costi complessivi di energia elettrica, valutati per il programma (P.24) del ciclo standard NT4 e per il programma (P.28) del ciclo sperimentale NT4-N₂O, si è stimato un risparmio energetico pari a circa il 16%. È da osservare che il risparmio sui costi dovuti all’energia elettrica è comparabile con quelli ottenuto sul costo dei gas tecnici.

CONCLUSIONI

Nel presente lavoro, che ha costituito l’attività sperimentale di una tesi di Laurea in Ingegneria dei Materiali, è stato eseguito un confronto tra cicli standard di trattamento termochimico di nitrurazione gassosa e cicli sperimentali con protossido d’azoto, eseguiti sull’acciaio 42CrMo4. Dal confronto tra i cicli standard e quelli sperimentali è stato messo in evidenza come le caratteristiche tecniche dei campioni trattati e analizzati siano risultate del tutto comparabili per ciò che riguarda la durezza superficiale, gli spessori di coltre bianca ottenuti e la morfologia dei nitruri come rilevato delle analisi microstrutturali. Il vantaggio più evidente riguarda il risparmio di tempo-ciclo a parità di profondità efficace ottenuta, confermato ampiamente dall’utilizzo sistematico dei cicli testati anche nella produzione effettiva. Ulteriori vantaggi economici risultano evidenti dal confronto dei costi di produzione. Il maggior costo specifico del protossido di azoto è ammortizzato ampiamente dal risparmio complessivo sui gas grazie alla diminuzione dei tempi-ciclo e quindi ad un minore impiego complessivo di ammoniaca. Inoltre, il calo dei tempi-ciclo porta ad una non trascurabile diminuzione del consumi di energia elettrica per effetto di una conseguente minore necessità di mantenimento alle elevate temperature di processo. Infine, la diminuzione dei tempi di impegno impianto ne apporta una maggiore disponibilità, traducibile in una possibile migliore capacità di pianificazione della produzione.

RINGRAZIAMENTI

Si ringrazia l’azienda Siderit s.r.l di Zola Predosa (BO), tutto il personale operativo presente nel laboratorio metallurgico e nei reparti produttivi, in particolare il responsabile del progetto di
USE OF NITROGEN PROTOXIDE IN THE THERMOCHEMICAL GAS NITRIDING TREATMENT: STUDY OF PRODUCTION PROCESSES AND METALLURGICAL CHARACTERISATION

Keywords: nitrogen protoxide, surface hardening, metallography, gas nitriding

The gas nitriding treatment is usually carried out in an anhydrous ammonia environment inside retort furnaces at 500-550°C. The process times are variable according to the desired depth of surface hardening, but in general are very time consuming. This scenario motivated this research work, in particular studying the possibility to increase the industrial process rate. Many mechanical components in structural steels are subjected to this treatment in order to obtain an high surface hardening. The surface hardening is due to the deposition of α iron nitrides directly on the metal surface and to the precipitation of γ iron nitrides on the substrate. The research for new efficient and industrially applicable solutions led to use an additional gas in the process. In the present research the effect of the introduction of nitrogen protoxide within the anhydrous ammonia in the process environment has been tested. Tests have been performed inside of the Siderit s.r.l. establishments in Zola Predosa (Bologna-Italy). Initially, the standard nitriding processes in the anhydrous ammonia environment have been studied, introducing samples in 42CrMo4 steel within the production materials inside of the retort furnaces. The cycles able to guarantee surface hardening of 0.3 and 0.4 millimeter depths - namely NT3 and NT4 respectively - have been taken into account. Subsequently, the same samples have been realised introducing the protoxide of nitrogen as a processing gas and evaluating its quantity respect to ammonia, step by step. The mechanical and metallurgical characterisations of the standard processes and of the ones with the use of NO within the nitriding environment have been performed; Vickers hardness tests on the samples treated surfaces and also Vickers micro hardness profiles on the treated thicknesses have been carried out. Moreover, the quality of the nitriding layers, obtained by the different processing parameters, have been evaluated through a careful metallographic analysis. The use of nitrogen protoxide can improve the kinetic of the process, which leads to a reduction of the process times respect to the standard processes of about 20-30%. Despite that, the mechanical and metallurgical characteristics of the samples treated with the additional N,O gas are comparable with the ones treated by means of the traditional thermochemical process. This way, the use of nitrogen protoxide could be of great importance in industrial nitriding processes. Pros and cons in terms of feasibility and cost are discussed on the basis of qualitative comparisons with currently applied industrial processes.
INFLUENCE OF HEAT TREATMENT ON THE MICROSTRUCTURE AND TOUGHNESS OF BÖHLER M333 ISOPLAST STEEL

J. Perko, C. Redl, H. Leitner

In this work the through hardenability and the influence of the heat treatment parameters (austenitizing temperature, cooling parameter $\lambda$ and tempering temperature) on the microstructure and the achievable toughness level of Böhler M333 ISOPLAST are investigated. The results are compared to the standardized tool steel grade DIN 1.2083. The investigations showed that the cooling parameter $\lambda$ has a strong influence on the impact toughness of M333 ISOPLAST plastic mould steel. The toughness is reduced by pro-eutectoid precipitates and not by a lack of through hardenability. Furthermore, it was found out that depending on the cross section of the moulds appropriate heat treatments lead to a good combination of hardness and toughness.

KEYWORDS: plastic mould steel, heat treatment, toughness, through hardenability, pro-eutectoid precipitates

INTRODUCTION

Plastic mould steels which are currently available on the market, e.g. DIN 1.2083, 1.4028 and 1.2316, are often not able to fulfil the high requirements of the plastics processing industry. For that reason, Böhler Edelstahl developed the pressure-electro-slag remelted, nitrogen alloyed tool steel grade M333 ISOPLAST. Nitrogen has a lot of positive effects on martensitic chromium steels [1, 2]. The partial replacement of carbon by nitrogen leads to an increase in corrosion resistance and toughness. General corrosion is reduced as well as pitting and crevice corrosion. The improvement in toughness results primarily from the very homogeneous distribution of fine precipitates [3]. Thus, M333 ISOPLAST combines excellent mirror finish polishability with highest cleanliness and toughness levels and excellent corrosion properties.

However, as a consequence of the growing demand for large moulds, plastic mould steels must also exhibit an excellent through hardenability in order to avoid the formation of bainite or pearlite during quenching. Additionally, due to lower cooling rates a low tendency to form grain boundary precipitates, which cause grain boundary embrittlement, is required. Therefore, this work concentrates on the investigation of the through hardenability and on the influence of the heat treatment parameters on the mechanical properties and microstructure of M333 ISOPLAST.

MATERIALS AND INVESTIGATION METHODS

Tab. 1 shows the chemical composition of M333 in comparison to M310, which approximates the standardized tool steel grade DIN 1.2083. This steel was used as a reference steel grade for the dilatometer investigations. The samples for the dilatometer investigations of M333 and M310 were manufactured from a hot-rolled and soft-annealed bar with a diameter of 86 mm. For the dilatometer experiments a quenching dilatometer Bähr Dil 805 A/D was used. To follow the evolution of the hardness of the dilatometer samples, Vickers hardness values (HV10) were measured using microhardness tester supplied by Zwick.

For the samples for impact toughness testing of M333 two slices with a thickness of 60 mm were cut from the top of a forged and soft annealed bar with the dimension 603 x 303 mm². Then, the samples were cut in longitudinal direction at half radius and were heat treated with an oversize of 0.5 mm on every side. For the hardening of the samples a vacuum heat treatment furnace was used. Quenching was performed by using nitrogen as quenching gas.

The cooling parameter $\lambda$ is defined as the cooling time from 800 to 500 °C in seconds divided by 100. The selected cooling parameters $\lambda$ were adjusted with dummy samples exhibiting the same size as the test specimens. The temperature was controlled by mounting a thermocouple in a drilled hole in the centre of the dummy sample. The heat-treated samples were grinded to the final dimension of 7 x 10 x 55 mm³. The impact tests were performed with a 450 J pendulum Roell Amsler 101. Four samples of each heat treatment were tested and the average and standard deviation were calculated. Rockwell C hardness was measured on each specimen using a hardness tester Emco-Test M4R 025 G3. The fracture surface of all tested im-
Trattamenti termici

Fig. 1
Dilatometer curves of M310 and M333.
Curve dilatometriche degli acciai M310 e M333.

Tab. 2
Applied heat treatments for impact toughness testing.
Trattamenti termici applicati per la verifica della resilienza.

Results and Discussion

The aim of the dilatometer investigations was to find out how the austenitizing temperature and the cooling parameter \( \lambda \) influence the transformation behaviour of M333 in comparison to M310. During heating and cooling of a sample in the dilatometer, the change in length (\( \Delta L \)) as a function of time and temperature is recorded, from which \( \Delta L/L_0 \) versus temperature (T) is plotted. \( L_0 \) represents the original length of the sample. Representative plots of \( \Delta L/L_0 \) versus T of M310 and M333 at the two austenitizing temperatures selected (980 °C and 1020 °C) are shown in Fig. 1.

It can be seen that in case of M333 for both austenitizing temperatures only one transformation takes place during cooling. This is indicated by the distinct length change starting at about 250 °C. Microstructural investigations revealed that this length change is caused by the transformation austenite to martensite. However, the martensite start temperature (MS) is shifted to lower temperatures with increasing austenitizing temperature, which is due to the higher solution state of the austenite at higher temperatures. When M333 is austenitized at 980 °C, the remaining change in length after quenching is lower compared to 1020 °C. This indicates that the amount of retained austenite is less compared to austenitizing temperature 1020 °C, what was confirmed by XRD measurements.

In contrast, the steel grade M310 shows different transformation behaviour. At an austenitizing temperature of 980 °C and the highest investigated cooling parameter \( \lambda = 30 \) additional transformations can be observed, starting at about 750 °C and 400 °C. Microstructural examinations of this sample showed the presence of pearlite and bainite. Thus, an austenitizing temperature of 980 °C is not suitable for parts with large cross sections made of this steel grade. For an austenitizing temperature of 1020 °C, M310 shows neither a pearlitic nor a bainitic transformation at a cooling parameter \( \lambda = 30 \). However, for both steel grades the measured MS-temperatures are summarized in Tab. 3.

The experimentally determined hardness measurements of the dilatometer samples are also listed in Tab. 3. In case of M333 Tab. 3 reveals that while the hardness decreases with increasing \( \lambda \) values, the MS-temperature increases. This indicates a decreasing solid solution state with increasing \( \lambda \) values which might be due to the formation of pro-eutectoid phases such as carbides or carbonitrides.

The microstructure of selected dilatometer samples is shown in Fig. 2. In order to reveal the microstructure, Nital was used as etchant. In Fig. 2, the former austenite grain boundaries can

<table>
<thead>
<tr>
<th>Cooling parameter ( \lambda )</th>
<th>M333 ISOPLAST</th>
<th>M310 ISOPLAST</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.5</td>
<td>8</td>
</tr>
<tr>
<td>980 °C</td>
<td>HV10</td>
<td>593</td>
</tr>
<tr>
<td></td>
<td>MS [°C]</td>
<td>300</td>
</tr>
<tr>
<td>1020 °C</td>
<td>HV10</td>
<td>636</td>
</tr>
<tr>
<td></td>
<td>MS [°C]</td>
<td>278</td>
</tr>
</tbody>
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* BS … Bainite Start Temperature

Tab. 3
MS-temperature and hardness of M333 and M310 after various heat treatment cycles.
Temperatura MS e durezza degli acciai M333 e M310 dopo differenti cicli di trattamento termico.

Tab. 1
Chemical composition of the tool steels investigated.
Composizione chimica degli acciai da utensile investigati.

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<th>Cr</th>
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<td>0.70</td>
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<tr>
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<td>0.30</td>
<td>13.50</td>
<td>-</td>
<td>+</td>
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Tab. 2
Austenitizing temperature [°C] 980/1020
Holding time [min] 30
Cooling parameter \( \lambda \) [s x 10^{-2}] 0.5/5/8
Tempering temperature [°C] 250/300/510/520/530/540
Tempering time [min] 2 x 120
not be seen in both M333 samples quenched with $\lambda=0.5$. On the other hand, the grain boundaries of M310 at an austenitizing temperature of 1020 °C are weakly visible, indicating that the grain boundaries are decorated. Additionally, M310 contains some coarser M23C6 carbides, which is one of the reasons for the significantly lower toughness level in comparison to M333, as reported in [4, 5].

At a cooling parameter $\lambda=8$ both steels show a strong decoration of the grain boundaries, which is designated by the distinct appearance of the grain boundaries. However, the strongest decoration shows M333 when it is austenitized at 1020 °C. The M333 samples austenitized at 980 °C exhibit a finer grain and the decoration of the grain boundaries is less pronounced. The reason for the lower austenite grain size of the M333 samples austenitized at 980 °C is the higher content of undissolved precipitates, which stabilize the grain size during austenitizing. At 1020 °C their amount is strongly reduced, which causes grain growth.

The reason for the decoration of the former austenite grain boundaries seems to be due to the formation of pro-eutectoid, chromium-rich precipitates as reported in [6, 7]. Herzog [7] investigated oil and air quenched samples with SANS and found out that the amount of pro-eutectoid precipitates in the size range from 1 to 100 nm increases with increasing austenitizing temperature and cooling parameter, which correlates well with the results of M333.

The results of the dilatometer investigations indicated, that the interaction of pro-eutectoid precipitations and austenite grain size may have more impact on the toughness of M333 than a lack of through hardenability, as the grain boundary decoration occurred at $\lambda$-values below 8. Therefore, the impact toughness investigation of M333 concentrated on $\lambda$-values below 8.

However, the investigations are primarily focused on the heat treatment of larger cross sections. For that reason the tempering of most samples was carried out at temperatures between 510 and 540 °C, which are higher than the secondary hardening peak of M333. Temperatures above the secondary hardening peak are needed to reduce residual stresses and retained austenite as far as possible. However, for comparison the tempering temperatures 250 and 300 °C were included in the investigations.

Fig. 3 and 4 show the impact energy versus tempering temperature of the samples austenitized at 980 °C and 1020 °C, respectively. Additionally, the measured hardness values are plotted in the diagrams. The results reveal that the hardness at each tempering temperature is virtually independent of the cooling parameters up to $\lambda=8$. The hardness level of the samples hardened at 980 °C is continuous 1-2 HRc lower than the hardness of samples austenitized at 1020 °C. Nevertheless, a hardness of approximately 50 HRc, which is typical for the plastic moulding industry, is still achievable with an austenitizing temperature of 980 °C.

In contrast to the hardness, the achievable toughness level is strongly depending on the cooling parameter $\lambda$, especially for the samples austenitized at 1020 °C. As long as the cooling parameter $\lambda$ is low ($\lambda=0.5$), the samples austenitized at 1020 °C show high hardness and toughness values in all heat treatment cycles.
investigated. When the cooling parameter is increased to \( \lambda = 5 \), the toughness is significantly reduced, especially for the most relevant tempering temperatures 510 and 520 °C, which have to be applied to reach hardness levels of 50 HRc at temperatures above the secondary hardening peak.

For the most relevant tempering temperatures for industrial application, the impact energy as a function of the cooling parameter \( \lambda \) is illustrated in Fig. 5. It can be seen that the cooling parameter \( \lambda \) has only little influence on the impact toughness of samples austenitized at 980 °C, which is beneficial for the heat treatment of tools with large cross sections. In contrast to that, the negative influence of the cooling parameter \( \lambda \) on the impact toughness of samples austenitized at 1020 °C is more pronounced. For that reason, an austenitizing temperature of 1020 °C seems to be appropriate for tools with small cross sections only.

To investigate the microstructural mechanisms that cause embrittlement of M333, the fracture surface of the tested specimens was investigated macroscopically and by means of SEM (Fig. 6 and 7). Fig. 6 shows that the cooling parameter \( \lambda \) has only little influence on the fracture appearance of samples austenitized at 980°C. There is only a marginal reduction of ductility when samples with low and high cooling parameters are compared, which is in accordance with the measured impact toughness values. In contrast to that, the samples austenitized at 1020°C show a high loss of ductility with increasing \( \lambda \) values. This effect is extremely pronounced at tempering temperatures of 510 and 520 °C, where the fracture mode changes from ductile to mainly brittle already at \( \lambda = 5 \). This is also reflected in the low impact toughness values measured.

A detailed fracture analysis was conducted by SEM investigations. Fig. 7 shows the fracture surface of the samples austenitized at 980°C and 1020°C and tempered at 510°C in the region of the incipient crack.

It is obvious that both samples quenched with \( \lambda = 0.5 \) exhibit a ductile fracture, which corresponds well with the high impact toughness values measured. When the cooling parameter \( \lambda \) is increased up to 5 the fracture appearance changes significantly. The samples which were austenitized at 980°C still exhibit a ductile fracture, but the intergranular character increases. In contrast to that, the samples austenitized at 1020°C and cooled with \( \lambda = 5 \) exhibit an entire intergranular fracture with no ductile character at all, being the reason for the low impact toughness of these samples. When the cooling parameter is increased to \( \lambda = 8 \), the intergranular character of samples austenitized at 980°C increments, but there is still a high portion of ductile fracture visible.

A more detailed view on the surface of the fracture of the samples hardened at 1020°C with cooling parameter \( \lambda = 5 \) confirms this theory (see Fig. 8). However, the presence of pro-eutectoid precipitates could not be clearly confirmed with SEM-EDX, as the resolution of this method is too low. But it can be assumed that the formation of precipitates preferentially takes place at the grain boundaries, as the critical nucleation energy is lower there and because of the lower surface energy that needs to be brought up. Therefore, precipitations with a low coherency develop preferentially there [6].

However, the investigations revealed that a lack of through hardenability can be excluded; the embrittlement with increasing \( \lambda \) values can only be caused by a disadvantageous combination of austenite grain size and high solution state of the austenite. This encourages the formation of pro-eutectoid precipitates at the austenite grain boundaries at higher \( \lambda \) values, which causes the
observed embrittlement of M333, especially in the region of the secondary hardening peak. Additionally, heat treatment simulations were carried out in order to determine the temperature in the centre of a block during quenching. Nitrogen gas with a pressure of 5 bar was used as quenching medium. Fig. 9 depicts the temperature evolution in the centre of work pieces with different square cross sections. The results show that a cooling parameter $\lambda$ of approximately 8 is achievable in the centre of a block with a dimension between 150 x 150 mm$^2$ and 200 x 200 mm$^2$. However, Fig. 9 also reveals that cooling of real work pieces does not follow one $\lambda$-value. At the beginning cooling is always slower, which enables the formation of pro-eutectoid precipitates on grain boundaries. This cooling characteristic also applies for the quenching of the impact toughness samples investigated. Therefore, the measured impact toughness values reflect the behaviour of real work pieces.

CONCLUSIONS

The cooling parameter $\lambda$ has a strong influence on the impact toughness of M333 ISOPLAST plastic mould steel. It was found that the toughness is reduced by pro-eutectoid precipitates and not by a lack of through hardenability. If low cooling parameters $\lambda$ can be attained, e.g. during heat treatment of tools with small cross sections, austenitizing at 1020 °C results in the best combination of high hardness and impact toughness. On the other hand, the application of lower austenitizing temperatures, e.g. 980 °C, significantly reduces the negative influence of higher cooling parameters $\lambda$ on the impact toughness. This study showed that Böhler M333 ISOPLAST is a suitable steel grade for moulds with small and large cross sections, if an appropriate heat treatment is applied. However, additional work is required to gain detailed information on formation of the precipitates which cause embrittlement.

ACKNOWLEDGMENTS

The authors are grateful to Carlos Martinez, Böhler Edelstahl GmbH & Co KG, for performing the SEM studies.

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INFLUENZA DEL TRATTAMENTO TERMICO SU MICROSTRUTTURA E TENACITÀ DELL’ ACCIAIO BÖHLER M333 ISOPLAST

Parole chiave: trattamenti termici, proprietà, acciaio

In questo lavoro sono stati analizzati temprabilità e l’influenza dei parametri di trattamento termico (temperatura di austenitizzazione, parametro di raffreddamento $\lambda$, temperatura di rinnenimento) sulla microstruttura e sul livello di durezza ottenibile per l’acciaio Böhler M333 ISOPLAST. I risultati sono stati confrontati con il classico acciaio per utensili DIN 1.2083. Le indagini hanno evidenziato che il parametro di raffreddamento $\lambda$ ha una forte influenza sulla resistenza all’urto dell’ acciaio M333 ISOPLAST per stampi destinati all’industria delle materie plastiche. La tenacità risulta ridotta dai precipitati pro-eutettoidici e non dalla mancanza di temprabilità. Inoltre, si è accertato che a seconda della sezione trasversale degli stampi, i trattamenti termici appropriati portano ad un buon compromesso fra durezza e tenacità.

ABSTRACT

INFLUENCE OF HEAT TREATMENT ON MICROSTRUCTURE AND DUCTILITY OF BOHLER M333 ISOPLAST

Keywords: heat treatments, properties, steel

In this work, the temperability and the influence of the parameters of heat treatment (austenitization temperature, cooling parameter $\lambda$, tempering temperature) on the microstructure and the level of hardness achievable for the steel Böhler M333 ISOPLAST were studied. The results were compared to the classic tool steel DIN 1.2083. The investigations have shown that the cooling parameter $\lambda$ has a strong influence on the impact resistance of M333 ISOPLAST for dies used in the plastics industry. The ductility decreases due to the pro-eutectoid precipitates and not due to lack of temperability. In addition, it was ascertained that depending on the cross section of the dies, the appropriate heat treatments lead to a good compromise between hardness and ductility.
NEW CHALLENGES AND DIRECTIONS FOR HIGH PRESSURE DIE-CAST MAGNESIUM

L. Zaffaina, R. Alain, F. Bonollo, Z. Fan

Being the lightest structural metallic material, Magnesium is widely used by the automotive industry when searching for solutions to address energy consumption, emission reductions and vehicles handling. The need to use light-weight solutions is becoming stronger than it has been so far, as other technologies allowing to reduce emissions have already yield a lot of their potential.

This wider use of Magnesium requires to grow more knowledge on the material properties, corrosion protection techniques and improved production processes. However material R&D cannot be limited to property studies and potentials: directions for innovation implies to understand all the benefits of the material and process for each application and their values to the customer.

In this work, the components’ key attributes will be highlighted, together with the efforts to lower costs and the potential use of new technologies.

KEYWORDS: magnesium, optimisation, applications, automotive, CAE led design, innovation, value analysis, HPDC, melt conditioned HPDC

INTRODUCTION

Over the last decades, the demand for magnesium components in the automotive industry kept increasing. The key drivers have been the need to reduce fuel consumption and emissions and the need to improve vehicle handling. However there are other advantages to use cast magnesium solution such as improved tolerances, higher potential to contribute to vehicle stiffness, higher fit and finish, improved noise vibration and harshness performance; but these are difficult to evaluate and not often quantified in assessing the overall product values.

In order to plot the directions for innovation in terms of technology and applications, it would be useful to clearly understand the list of benefits of magnesium casting and their value to the customer.

A view of the market from Meridian’s point of view is shown here, together with the technologies whose have triggered new applications to appear in the market.

A MARKET VIEW

Fig. 1 shows the main cast magnesium components for automotive industry. Meridian’s key products in power-train applications are transfer cases and transmission housings with more than 2 million cast per year; steering column brackets and instrumentation panels are the main body component produced. (respectively 3 and 2,5 million parts per year).

New components are gaining growing interest at car makers, as indicate increased number of request for quotations and ongoing development studies; theses applications are Front End Structures, Engine Cradles, Centre Consoles and...
With regards to magnesium applications, the recent changes that have happened are as follows:

a) Availability of chromium-free corrosion prevention solutions helped to introduce exterior components with aesthetic requirements like Front End Carriers, door frames, roof mechanisms or rear window frames.

b) Low creep alloys with high ductility (AE44) led to the introduction of magnesium castings for engine applications, where both crash and high temperatures have to be met. Meridian produces the first example of engine cradle for the General Motors Corvette Z06 and more components are currently under development. In addition to the high temperature mechanical properties, AE44 shows better corrosion resistance than common used ductile alloys (AM60, AM50); the general corrosion resistance is even higher than the AZ91’s commonly used for transfer cases (see Fig. 2). As this part is not cosmetic, just like a transfer case, the Engine Cradle is not coated. [1]

c) Over the last two years, the cost of Aluminium alloys increased while magnesium alloys remained stable, hence Magnesium has become more attractive for power train components. This cost evolution and the increased need for weight saving is driving new interest and developments from costumers.

**UNDERTANDING WHAT CUSTOMERS VALUE**

The changes mentioned above are not sufficient to understand the growth of specific applications on the market and why for some costumers Magnesium has become core roots for defined applications, while others do not follow the trend.

Above weight saving, costumers see other interesting features in the use of Magnesium on specific applications: these are not always easy to capture by a tier 1 or tier 2 supplier. For a similar product, the functions to achieve can vary a lot depending on the customers or vehicle and the benefit of using Magnesium will vary. Each product been specific even within a family of products, general rules on reason to use Magnesium are difficult to extract.

More focus has been put to try to understand drivers to chose Magnesium, what influences costs and where the costumer will find more interest in the technology. In other words where will the costumer find values. According to J.C. Anderson and J. A. Naurus “Value is the worth in monetary terms of the technical, economic, service and social benefits a customer company receives in exchange for the price it pays for a marketing offer” [3].

It is important to keep in mind that the customer does not really want materials or services. He wants Use and/or Aesthetic functions to be accomplished. [4]

On products two kind of functions can be individuated: pre-requisites and attributes.

The pre-requisites are the functions for which there is a target to reach and the customer is not willing to see value in an extra-performance.

At the opposite, for attributes, the customer is willing to value an increased performance. By extension the costumer could also review his requirements at the system level and allow a reduction in performance in exchange for a price reduction.

**WORK TO LOWER THE COST OF FUNCTIONS**

As all automotive suppliers, Meridian works to reduce costs, and different approach are used. The usual one, based on product cost breakdown, is not sufficient to identify actions; it has been found that more ways to reduce cost could be yield by doing the analysis operating on the cost per function. An extension of this approach is the identification of other markets i.e. new applications for which current technology could reach other values at an interesting cost.

In an ideal world, it would be useful to plot for each function, the state of the market, i.e. for each function, what level is reached, at which costs, where Magnesium technologies are present. A successive step should be to assess if different values could be reached, at which costs and with which technology improvements. Unfortunately such approach is not feasible as it would require to grab a high amount of information from the customer.

For a single component, the system is complex, and it is actually sometime not even obvious for the customer to know that he has the information about component/system functions and their value: in fact the data may lay in different functional areas [3].

Considering the complexity of the component and the system, a simple approach has to be developed so that it can be reapplied to other products. The investigation has then to focus on:

- identify functions
- understand attributes and values
- assess the cost for attributes and values
- reduce cost to achieve attributes and values
- understand costumers sensitivity to different values

In this work, the component’s key attributes will be highlighted, together with the efforts to lower costs and the potential use of new technologies.
NEW COMPONENTS

a) Front End Carrier (FEC)

The features that customers value for front ends are:
- weight saving (40 to 50%), i.e. 3 to 9 kg depending on the type of vehicle [5]
- induced weight saving: the reduced weight at the front of the vehicle reduces load on the body in white
- better fit and finish performance compared to steel when requirements increase
- high stiffness achievable, hence potential to redistribute stiffness target at the system level
- better air flow management offered by foundry shape
- higher integration offered by the foundry compared to welded technologies

In the past, the biggest issue facing the use of magnesium for exterior applications has been corrosion prevention and in particular galvanic corrosion. Today galvanic and cosmetic corrosion requirements are achieved by proper design of component and fixings and proper choice of joint materials. Two principles are to respect. The first one is to insure a good drainage in fixing areas to avoid that mud and humidity get trapped. The second is to separate Magnesium from steel by the introduction of an Aluminium spacer, respecting a simple design rule that is to have a minimum distance of 10 mm between Magnesium and steel.

Cosmetic targets have to be achieved, hence parts have to be coated. Current solution consist of an acid etch to remove surface iron particles picked up during casting process, a chemical conversion coating to insure the adhesion of the final top coat that is an epoxy base powder coating. This solution is capable of 1000 hours salt spray tests and of 12 weeks humidity salt spray tests (see Fig. 3).

To reduce cost, new development consider top plastic cover so that the magnesium part is not visible anymore and coating can be removed.

Over last six years, the use of magnesium for FEC extended from light truck to SUV and finally to passenger car where component weight decreased from 5 to 3 kg, whilst increasing crash requirements were met. (Fig. 4)

The potential of Front End Structure is strongly dependant from the type of Front End and the functions it has to achieve. The number of part in production in Europe has increased from 1 to 4 from 2004 to 2007 and 7 will be in production in 2009. Components under development and physical validation increase coincidently, as shown in Fig. 5.

The first development was carried out in North America and led to development and production of the Corvette Z06 Engine Cradle (Fig. 6). The project’s aim was to achieve significant weight savings and it led to the development of a new alloy capable of high to moderate strength, ductile behaviour to function in cyclic loading, elevated temperature and corrosive environment resistance.

The magnesium solution led to a 32% weight saving in comparison with aluminium which means 4.8kg. The new alloy is capable of working at 125°C, has improved elongation (9-10% with 7mm-thick walls) and good corrosion resistance. Proper alloy properties together with an accurate fixings design allowed to avoid any surface treatment. For this sport car the key attribute
Magnesio e leghe

is an important weight saving associated with the size of the part in an area where Magnesium had not been used before.

Alloys development has allowed new chassis and power train application to be considered: productions processes in conjunction with material properties and Meridian design approach [6] will lead to more engine cradles, engine brackets, power train components.

c) Centre Console Structure (CCS)

The Centre Console Structure is one of these products for which requirement can differ strongly for one application to another. Key attributes are:

- weight saving of 50% compared to steel
- high integration in a small mass of Magnesium, hence very cost efficient
- increased potential to offer storage volume offered by casting potential
- improved assembly precision due to one-piece solutions, which improves visible perception of quality
- can be structural and transfer load in lateral impact in specific vehicle configurations
- can be cosmetic with a coating: an example of that is the Ford GT40 console

Costumers show more and more interest in Centre Console Structure, and the amount of development is growing significantly.

INCOMING APPLICATIONS AND TECHNOLOGIES

Some interesting opportunities and projects under development are briefly mentioned here.

a) Increased use of optimisers

CAE-led design approach [7] gives interesting advantages such a significant reduction of time to deliver the concept, possibility to provide to customer the CAE model in advance to verify structural performance and a mid-surface model to check the package.

In addition to that, the use of optimisers since the early concept-design stage makes that geometry can be design to maximise the value to customer by using optimisers to lower the cost for functions or to minimise the weight-saving premium price.

Ongoing and future developments will extend this approach with increased use of optimisers on process and design.

b) MC-HPDC process

Melt conditioned high pressure diecasting (MC-HPDC) process, previously named rheo-diecasting (RDC) process, is a combination of a MCAST (melt conditioning by advanced shear Technology) machine for melt conditioning with a standard high pressure diecasting (HPDC) machine for component shaping [8].

Laboratory trials show that the MC-HPDC process is capable of producing close to zero porosity samples with a fine and uniform microstructure throughout the entire component (Fig. 7). MC-HPDC process provides samples with improved strength and ductility in the as-cast condition, compared with HPDC or other available semisolid processing techniques. Initial tensile test have shown elongations up to 20% on AM50 test sample [9].

Extended work on this process has shown that the MCAST machine is actually mostly acting as a grain refining device and that it could be used with 100% liquid, meaning that all process parameters can remain unchanged compared to conventional HPDC process. The addition of this single part
pianta equipment to an existing HPDC machine would limit investment and permits new applications. Potential applications could be in the suspension area, together with A644 showing high temperature resistance, very good corrosion performance and increased ductility.

Another interesting characteristic of the MCAST machine is the fact that it breaks up oxides in small round-shaped particles well dispersed through the entire components. This characteristic opens another potential that is the possibility to use scrap parts and runners systems for specific internal applications, leading to interesting cost savings [10]. Fig. 8 compares the mechanical properties of AZ91 alloy produced by the MC-HPDC process using different amount of scraps.

c) New trend on approaching cosmetic corrosion As market grows, cost for coating has significantly decreased; another consideration is the fact that the coating is acting just to respect visual requirements, while the structural-galvanic corrosion resistance is obtained through spacers and proper design of components. Hence in some cases where part would not be visible coating could be removed: it could be the case on FEC by masking the top surface with a plastic cover.

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ABSTRACT

**NUOVE SFIDE E DIREZIONI DI SVILUPPO PER IL MAGNESIO PRESSOCOLATO**

Parole chiave: magnesio e leghe, corrosione, pressocolata, tixoforatura, impieghi alta/bassa temperatura, processi, produzione, proprietà

È noto come il magnesio sia il più leggero tra i materiali strutturali: è questo il principale motivo per cui è utilizzato in maniera estensiva nell’industria automobilistica (fig. 1) al fine di minimizzare i consumi energetici e le emissioni inquinanti e nel caso in cui si voglia migliorare la maneggevolezza dei veicoli.

La spinta verso l’alleggerimento ha visto un’accelerazione negli ultimi anni dovuta probabilmente al fatto che altre tecnologie tese a ridurre le emissioni hanno già raggiunto gran parte del loro potenziale. Un utilizzo più ampio del Magnesio comporta la necessità di aumentare la conoscenza delle proprietà del materiale, delle tecniche di protezione dalla corrosione (figg. 2, 3) e di processi produttivi migliorati.

Tuttavia le attività di R&S non possono essere limitate allo studio delle proprietà e dei potenziali del materiale: indirizzare l’innovazione in termini di tecnologie e applicazioni implica comprendere tutti i benefici del materiale e del processo per ogni applicazione e il loro valore per il cliente.

Oltre alla riduzione di peso ci sono infatti altri vantaggi nell’uso di soluzioni in magnesio pressocolato quali: riduzione delle tolleranze, potenzialità di contribuire alla rigidità del veicolo, miglioramento dell’assorbimento delle vibrazioni; purtroppo questi benefici sono difficili da valutare e spesso non quantificati nella stima del valore complessivo del prodotto da parte del cliente.

In questo lavoro sono analizzati alcuni dei cambiamenti tecnologici e di mercato che hanno permesso l’estensione del magnesio pressocolato a nuove applicazioni (fig. 6) e il loro impatto su specifici componenti.

Si è evidenziato come i cambiamenti citati non siano sufficienti a spiegare la crescita di specifiche applicazioni (fig. 5) e come sia difficile estrarre regole generali che spieghino le ragioni per cui si scelga di utilizzare o meno soluzioni in magnesio pressocolato.

Diventa quindi importante lavorare al fine di comprendere quali siano le funzioni del componente e i loro costi e cercare di indirizzare le attività di R&S alla riduzione del costo delle specifiche funzioni. A titolo esemplificativo sono sottolineati in questo lavoro gli attributi chiave per alcuni componenti (fig. 4), gli sforzi tesi a ridurlo il costo e il potenziale d’uso di alcune nuove tecnologie (figg. 7, 8).