

# INFLUENCE OF DEEP CRYOGENIC TREATMENT ON THE PROPERTIES OF CONVENTIONAL AND PM HIGH SPEED STEELS

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*The influence of Deep Cryogenic Treatment (DCT) on the properties of four wrought and PM high speed steels was investigated. Hardness and (apparent) fracture toughness  $K_a$  were measured to highlight the possible influence of DCT carried out before and after tempering. Dry sliding wear tests were carried using a block on disc configuration. The properties of the two wrought steels, HS6-5-2 (AISI M2) and HS6-5-2-5 (AISI M35), highlight a well defined influence of DCT. HS6-5-2 shows a remarkable improvement in abrasive wear resistance without any hardness increase. The most promising result is obtained by carrying out DCT before tempering. The opposite occurs by HS6-5-2-5, containing about 4.8%Co, whose wear resistance decreases in any treatment condition including DCT. PM steels show a less significant change in properties, since these are mainly controlled by the high amount of evenly distributed primary carbides which are not influenced by DCT. A slight increase in wear resistance was observed just for HS6-5-3. A general worsening was observed for HS6-5-3-8, namely HS6-5-3 plus 8%Co. In the light of results here presented cobalt seems to play a negative effect with respect to the low temperature conditioning of martensite.*

**KEYWORDS:** steel, phase transformation, powder metallurgy, heat treatment, mechanical tests, tribology, properties

## INTRODUCTION

High speed steels (HSS's), taking the name from their ability to retain high hardness during cutting at high speeds are known for over a century [1]. Their properties are the result of the complex interaction between hard primary carbides and a tougher metallic matrix, which is given by tempered martensite after heat treatment.

The chemical composition has a prominent role in determining hardness, toughness and wear resistance. It primarily affects the solidification structure in terms of volume, type, morphology and distribution of carbides. It also affects the final hardness after quenching by acting on the partitioning of alloying between carbides and metallic matrix and on the amount of retained austenite. Finally it affects the secondary hardening response of the steel during tempering at about 550°C. A typical composition for HSS is that of AISI M2, HS 6-5-2, containing 6% tungsten, 5% molybdenum, 2% vanadium

and about 4% chromium. Most tungsten-molybdenum grades (AISI M) were developed starting from this base composition. The use of cobalt in high speed steels is very important for hot hardness. Co does not act as carbide former, but enters in solid solution into the matrix. It raises the solidus temperature, allowing higher hardening temperature to be used and promoting carbide solution. This would promote higher amount of retained austenite after quenching, but Co decreases its stability allowing reduced amount of this phase by increasing %Co [2]. Negative results of cobalt addition are claimed to be reduced toughness and wear resistance [1].

The high speed steel properties are also dependent on the manufacturing process. Due to the high content in carbon and alloying such steels tend to form a highly interconnected eutectic carbide network during solidification, which can be only partially destroyed during following hot forging and rolling process. Large carbides strings oriented along the direction of thermomechanical processing are quite common in wrought steels. This poor carbide distribution, along with the relative big size of these hard particles, is reflected in limited values for fracture toughness, so that their maximum hardness must be kept below a critical level to avoid tools breakage or even microchipping phenomena during cutting [3]. The driving force towards an improved carbide distribution allowed PM High

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Material	HS	AISI	C	Cr	W	Mo	V	Co
<b>W</b>	6-5-2	M2	0.87	4.3	6.4	5.0	1.9	-
<b>W</b>	6-5-2-5	M35	0.93	4.2	6.4	5.0	1.8	4.8
<b>PM</b>	6-5-3	M3:2	1.28	4.0	6.4	5.0	3.1	-
<b>PM</b>	6-5-3-8	-	1.30	4.2	6.3	5.0	3.0	8.4

▲  
Tab. 1

**Nominal composition of the steels investigated (W=wrought, PM=Powder Metallurgy).**

Composizione nominale degli acciai studiati (W=forgiati, PM=Metallurgia delle Polveri).

3T	Q + 3T
3TC	Q + 3T + C
C3T	Q + C + 3T
C2T	Q + C + 2T

▲  
Tab. 2

**List of the heat treatments (Q=quenching,**

**T=tempering, C=Deep Cryogenic treatment).**

Elenco dei trattamenti termici (Q=tempra,  
T=rinvenimento, C=trattamento criogenico).

Speed Steels to appear in the early 1970's. Since then several different processes have been developed such as several new alloys suitable for PM production techniques. The small droplets produced during rapid solidification give rise to a very uniform carbide microstructure, which is retained even after Hot Isostatic Pressing (HIP) and hot working operations. The conventional coarse carbides segregation in conventional cast and forged high speed steels are thus avoided in PM HSS's [4].

Heat treatment of quenching and tempering contribute in the definition of the final properties of HSS tools. Austenitizing time and temperature are key parameters during quenching, because they control the final partitioning of carbon and alloying between the matrix and carbides and the grain size, as well. Proper combination of tempering temperature and time must be selected in order to achieve optimum secondary hardness, accompanied by a sufficient toughness. Secondary alloy carbides precipitate during heating, because of the diffusion of substitutional carbide forming elements. Fresh martensite forms from destabilized retained austenite on following cooling to room temperature.

In the past the use of subzero cooling of tool was proposed as a way to improve hardness and wear resistance. Subzero cooling of tool steel parts has been originally introduced in order to remove the retained austenite usually present after quenching. This transformation involves a hardness increase accompanied by a certain loss of toughness. However, the wear resistance, shows a slight improvement only, if compared to that obtained after a deep cryogenic treatment (DCT) carried out at lower temperatures, i.e., in liquid nitrogen (-196°C) [5,6]. This claims for a different strengthening mechanism operating in this temperature range. The most reliable theory ascribes this experimental evidence to a low temperature martensite conditioning phenomenon involving the formation of carbon clusters, which represent preferential nucleation sites for carbides precipitation during tempering [7]. As a consequence, the microstructure after tempering is more homogeneous, because secondary carbides result smaller and more evenly distributed.

In this work the influence of DCT on the properties of four wrought and PM high speed steels is investigated. A standard subzero cooling was introduced in the conventional heat treatment cycle before and after tempering. The influence of Co was also considered with respect to hardness, fracture toughness and wear resistance.

### EXPERIMENTALS

The composition of the high speed steels investigated is reported in Tab. 1. Two wrought steels, namely an HS6-5-2 (AISI M2) and HS6-5-2-5 (AISI M35) and two PM steels, HS6-5-3 and HS6-5-3-8 were selected. The first grade was already considered in a previous publication of the present author [8] and the results will be recalled in the frame of the present paper for purpose of comparison with the new ones. HS6-5-2-5 represents a variant of the base M2 containing about 5%Co, the element promoting hot hardness. PM HS6-5-3 can be considered as the Powder Metallurgical variant of base M2, added by a small V addition. Finally, HS6-5-3-8 is the variant of the former grade containing about 8%Co.

Prismatic samples (10x10x55 mm<sup>3</sup>) were extracted in the longitudinal direction from annealed bars. Standard vacuum heat treatment (3T, Tab. 2) followed, comprising austenitizing, gas quenching in 5bars nitrogen (Q) and triple tempering (3T), 2 hours each. Slow cooling down to about -180°C and soaking for 24 hours was performed by using a cryogenic processor placed at the University of Trento. Samples were cryogenically treated after and before tempering. In the first case (3TC) DCT followed standard heat treatment. In the latter case the treatment was carried out between conventional hardening and tempering (C3T) and reducing the number of tempering cycles (C2T).

An apparent fracture toughness  $K_{Ic}$  was determined using a procedure proposed by Lee et.al. to overcome the difficulties in introducing fatigue precrack to small fracture toughness specimens [11]. By high speed steels the major difficulty in introducing a fatigue precrack is given by the high brittleness of these materials. Notches depth with root radii  $\rho$  of 50µm were electro discharge machined in standard Charpy (10x10x55mm<sup>3</sup>) specimens. The ratio of notch depth to the specimen width ( $a/w$ ) was set at 0.2. Static fracture toughness testing was performed using 10 ton capacity universal tester (Instron). The specimens were loaded in three-point bending at a crosshead speed of 0.5 mm min<sup>-1</sup> according to the ASTM E399. Dry sliding tests were carried out using a block on disc configuration. A counterpart disc (40mm external diameter, 10mm width) of ASP2052 salt bath quenched and tempered to 70HRC was selected. The sliding speed was 0.68m/sec and a load of 200N was applied for a total sliding distance of 3000m. Each test was interrupted each

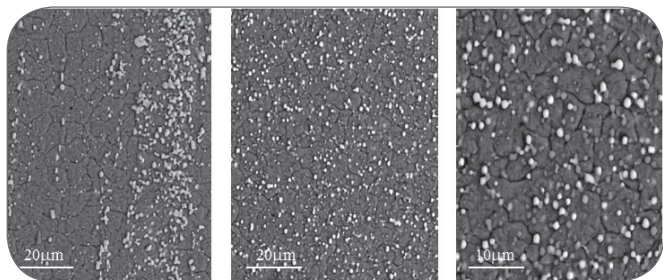


Fig. 1

Microstructure of a) HS6-5-2-5 and b,c) HS 6-5-3 (Nital 2%) (SEM, BSE).

Microstruttura dell'acciaio a) HS6-5-2-5 e b,c) HS 6-5-3 (Nital 2%) (SEM, BSE).

1000m to weight the sample by means of a precision balance (0.0001g).

## RESULTS

In Figure 1 the microstructure of HS6-5-2-5 (Fig.1a) is compared to that of the PM HS6-5-3 (Fig.1b-c). In this last case the higher amount and the better distribution of primary carbides can be appreciated. A quantitative analysis was carried out by analyzing XRD diffraction patterns using the Rietveld method. The relative amount of MC (gray in Fig.1) and M<sub>6</sub>C (white in Fig.1) carbides could be determined (Tab. 3). As expected, the highly V-alloyed grades display a higher MC content and a higher %vol of primary carbides, as well.

The hardness values of the steels after conventional heat treatment (3T Tab. 2) evidence the influence of Co, allowing higher HRC and HV30 to be observed. In order to evaluate the influence of DCT on the tempering behaviour the tempering curves for the four steels were determined. As already demonstrated for HS6-5-2 [8] DCT shifts the secondary peak hardness to lower temperature (see Fig.2 and Tab. 4). Hence, in order to

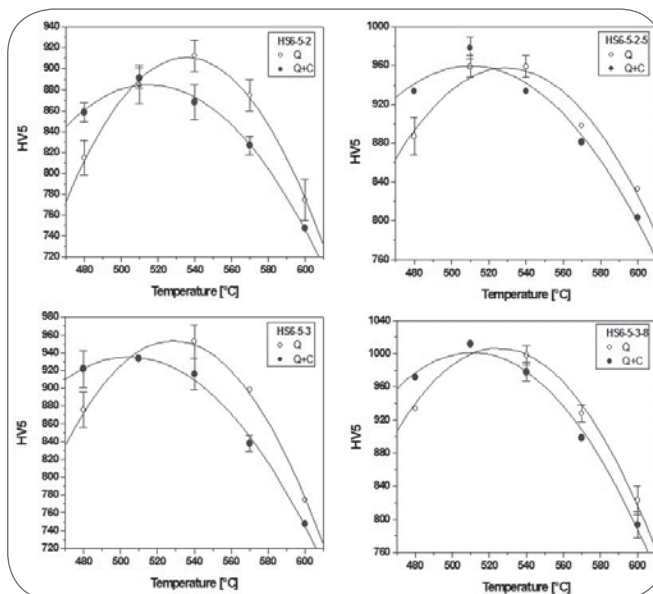


Fig. 2

The influence of DCT on the tempering curve of the investigated HSS's.

Influenza del trattamento criogenico sulla curva di rinvenimento degli acciai rapidi studiati.

avoid a loss of hardness due to overtempering, cryogenically treated steels should be tempered at a lower temperature. This also explains the lower hardness displayed by the steels DC treated following cycles C3T and even C2T in Tab. 2.

### Apparent fracture toughness

The toughness test of HSS material determined using EDM notched specimens always evidenced a brittle fracture process. In any case no plastic deformation could be appreciated during the 3-point bending tests carried out until fracture.

HS	Hardness				Primary carbides		
	HRC	sd	HV30	sd	MC	M <sub>6</sub> C	CVP
6-5-2	64.7	0.8	819	9	3.2	6.1	9.3
6-5-2-5	65.6	0.4	861	8	3.8	5.0	8.8
6-5-3	65.2	0.4	855	7	5.4	4.9	10.3
6-5-3-8	67.1	0.5	909	7	7.4	5.0	12.4

Tab. 3

HRC, HV30 and %vol primary carbides after conventional heat treatment 3T.

Durezza HRC, HV30 e %vol di carburi primari dopo trattamento termico convenzionale 3T.

Material	T <sub>p</sub> (Q)	T <sub>p</sub> (Q+C)	T <sub>p</sub> (Q) - T <sub>p</sub> (Q+C)
6-5-2	535°C	515°C	-20°C
6-5-2-5	525°C	510°C	-15°C
6-5-3	530°C	500°C	-30°C
6-5-3-8	525°C	510°C	-15°C

Tab. 4

Temperature of the secondary hardness peak for quenched (Q) quenched and deep cryogenically treated (Q+C) steels.

Temperature del picco di durezza secondaria per l'acciaio temprato (Q) e sottoposto ad ulteriore trattamento criogenico (Q+C).



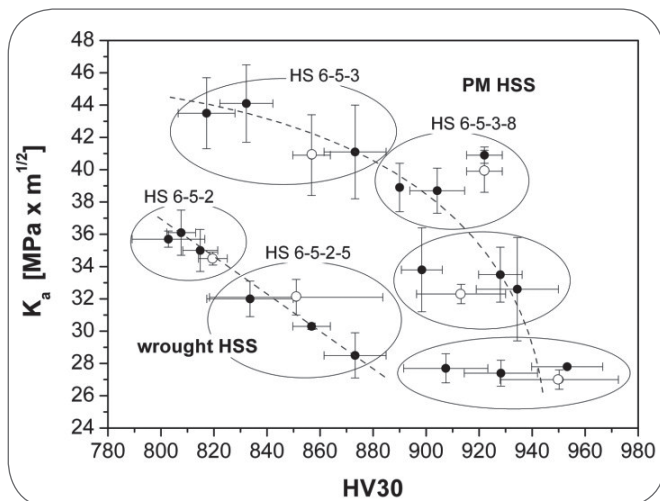


Fig. 3

**Apparent fracture toughness  $K_a$  vs. HV30 hardness.**

Tenacità a frattura apparente  $K_a$  in funzione della durezza HV30.

The apparent fracture toughness displays a general decrease by increasing hardness (Figure 3). However, given a specific hardness level lower  $K_a$  values were shown by wrought steels compared to those produced via Powder Metallurgy. This result highlights the marked influence of primary carbides distribution contrarily to previous investigations aimed at establishing a correlation between KIC and steel properties.[ ] The decrease in stress triaxiality with increasing  $\rho$  causes a decrease in the stress ( $\sigma_{yy}$ ) that would initiate fracture, so that the  $K_a$  value is generally higher than that of KIC measured on pre-cracked sample.[ ] Furthermore, since the radius of the plastic zone ahead of the notch, i.e.,  $r_p$ , according to Eq.1 increases with notch radius, the plasticized region is higher in the case of U-notched samples ( $\rho=50\mu\text{m}$ ) compared to the pre-cracked ones ( $\rho=0$ ).

$$\text{Eq. 1} \quad r_p = \frac{1}{6\pi} \left( \frac{K_I}{\sigma_{ys}} \right)^2 \quad \text{for plane strain}$$

$$\text{Eq. 2} \quad K_I = \lambda(a_{\text{eff}}) \cdot \sigma_{nom} \sqrt{\pi a_{\text{eff}}} \quad \text{stress intensity factor}$$

$a_{\text{eff}} = a + r_p$  = effective crack length

$a$  = original crack length (notch depth, 2mm)

$\lambda(a_{\text{eff}})$  = parameter depending on specimen and crack geometry

$\sigma_{ys}$  = yield strength

As a consequence the influence of primary carbides on crack nucleation is more likely to be observed in U-notched specimens, as demonstrated by the lower  $K_a$  values exhibited by the two wrought steels, showing large carbides strings (Fig.1a). The discussed relationship between  $K_a$ , hardness and microstructure is verified by conventionally treated steels (red points in Fig. 3) but, within the experimental scatter, also by all DC treated samples (dark points). With minor exceptions, the experimental points allow to conclude that DCT causes a general increase in toughness in Co-free grades, a decrease being always observed for the other two Co-containing gra-

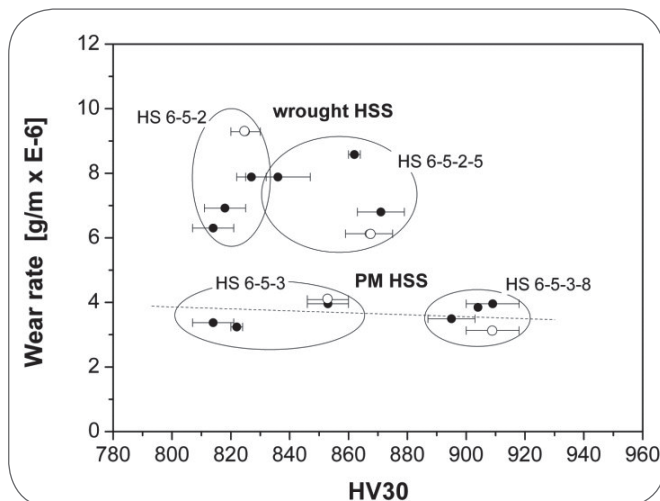


Fig. 4

**Wear rate vs. HV30 hardness.**

Tasso di usura in funzione della durezza HV30.

des here investigated. Even if the influence of Co seems to be mostly correlated to its influence on hardness, it is believed that it would also exert an influence of microstructure. This assumption is confirmed by HS6-5-3-8, the grade containing the highest %Co, where a marked drop of  $K_a$  is displayed in correspondence of hardness values lower than that attained with conventional heat treatment 3T (red point). The same, but to a lower extent, could be also concluded for HS6-5-2-5, the steel containing about 5%Co, showing constant  $K_a$  by hardness values lower than that of 3T.

## Wear resistance

Figure 4 displays the influence of hardness on the wear rate of various steels for all heat treatment variants considered. Even if a strict correlation cannot be established, the experimental points of steels not subjected to DCT (red points in Fig.4) evidence a general decrease of the wear rate by increasing hardness. This is in agreement with the higher resistance versus abrasion, the main damage mechanism operating during the tests. [8] However, higher wear rates are displayed by the two wrought steels compared to PM steels at a given hardness level (Fig.4). Furthermore, considering the experimental point of DC treated samples the above correlation between wear resistance and hardness tend to become less marked. It can be concluded that the microstructure also influences the tribological behaviour of high speed steels.

The most remarkable result is that of HS6-5-3 showing considerably lower wear rate compared to HS6-5-2, having very similar hardness and carbide content (Table 3). It is believed that this result can be explained looking at the finer microstructure of the PM grade. The more homogeneous primary carbides distribution is reflected in a lower carbides mean spacing, causing a more efficient protection of the softer matrix from the abrasion caused by the primary carbides in the ASP2052 counterpart material (Figure 5a) containing a big amount of hard MC.

The present explanation is supported by the SEM analysis of the worn surface. Fig.5b and 5c show that the abrasion scratches are mostly localized within the metallic matrix. The damage is more extended in wrought HS6-5-2 (Fig.5c,d), where big scratches can be appreciated even at low magnification. In this material, and similarly also in HS6-5-2-5, the uneven carbi-

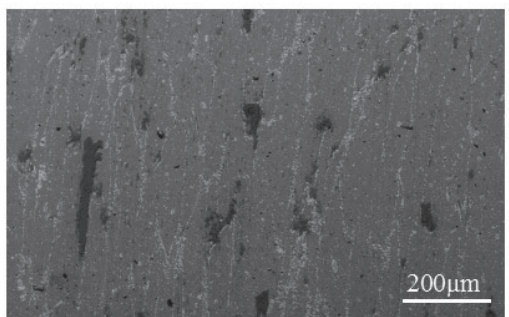
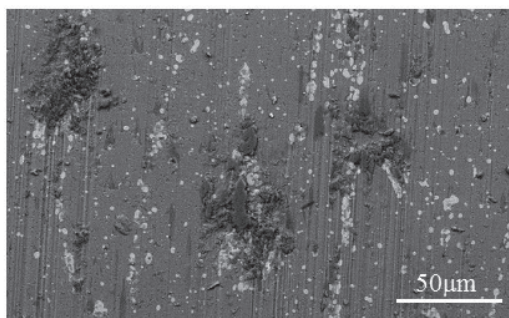
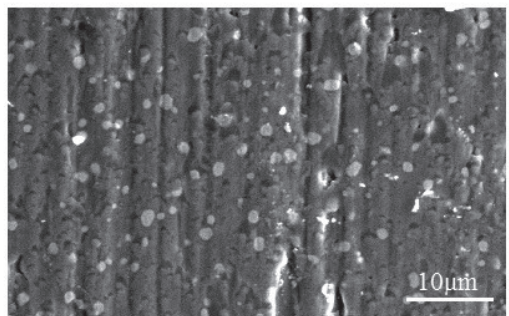
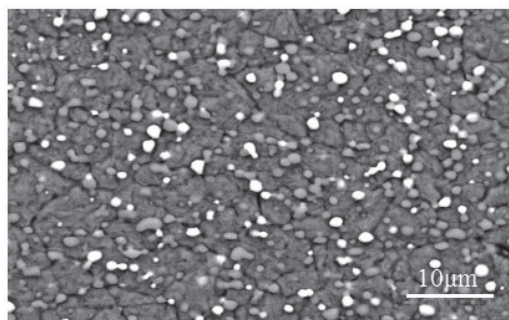


Fig. 5

**Microstructure of the a) counterpart ASP2052 HSS b) worn surface of PM HS6-5-3 c,d) worn surface of wrought HS 6-5-2.**

*Microstruttura a) dell'antagonista ASP2052 b) dell'acciaio PM HS6-5-3 usurato c,d) dell'acciaio forgiato HS 6-5-2 usurato.*

des distribution exposes the metallic matrix to heavy abrasion. This is confirmed by the formation of quite big oxide patches on the worn matrix regions, due to the local entrapment and sintering of wear debris.

Indeed, the closely spaced carbides in HS6-5-3 promote a very efficient protection of the matrix (Fig.5b). Hence, the wear ra-

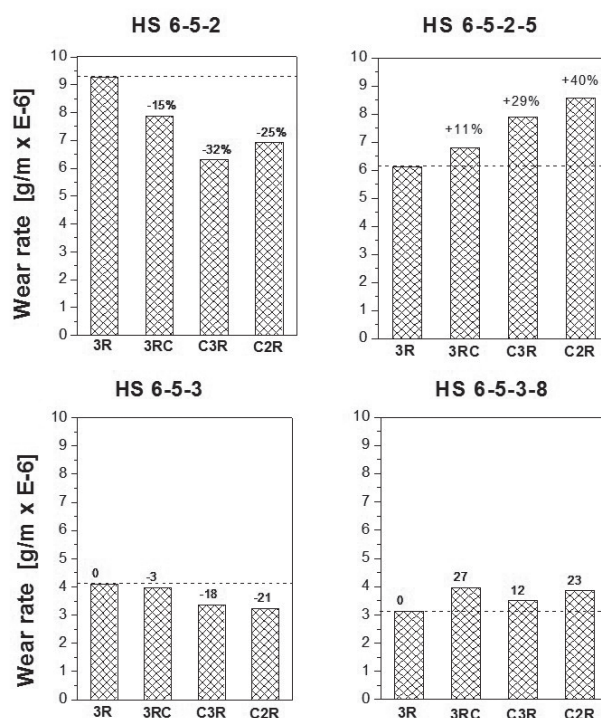


Fig. 6

**Influence of different DCT cycles on the wear rate of HSS's investigated.**

*Influenza dei differenti cicli di trattamento criogenico sulla resistenza ad usura degli acciai studiati.*

tes reported in Fig. 4 are closely related to the size of abrading particles, i.e., the fine hard carbides of the counterpart HSS (Fig.1a)

Looking in more detail at the influence of DCT it can be stated that the wear resistance is always increased in the case of Co-free HSS's (Fig.6). This result was already known for HS6-5-2 [8], where the improvement was ascribed to the low temperature conditioning of martensite, giving rise to a very fine precipitation of secondary carbides in the metallic matrix during tempering. The result is now confirmed by the PM HS6-5-3, as well. However, the prominent role of primary carbides in the tribological contact and the lower exposition of the metallic matrix leave us to appreciate just a lower increase in wear resistance for this PM grade with respect to HS6-5-2.

In any case, both Co grades show lowered wear resistance after DCT. The most deleterious effect is displayed by HS6-5-2-5, containing the lower Co content but showing the poorest carbides distribution, i.e., the largest influence of the metallic matrix to the wear behaviour. In the worst situation (C2R) the wear rate is increased of 40% with respect to the conventional heat treatment.

## CONCLUSIONS

The influence of deep cryogenic treatment (DCT) on the properties of four high speed steels has been evaluated in this work. Two of them, namely HS6-5-2 and HS 6-5-2-5, were selected among wrought grades, whereas the other two, HS6-5-3 and HS 6-5-3-8 among PM grades. The steels were heat treated including DCT before and after tempering. DCT does not significantly alter the hardness when carried out after conven-



tional tempering. Indeed, when carried out immediately after quenching, it always causes an anticipation of the secondary peak hardness, so that a lower hardness is generally observed. Lower tempering temperature must be selected to avoid over-tempering caused by carbides coarsening. The measured apparent fracture toughness  $K_a$  displayed a significant influence of hardness and primary carbides distribution. A general drop in  $K_a$  is observed by increasing hardness. Wrought grades displayed lower  $K_a$  than PM grades at a certain hardness due to the uneven distribution of primary carbides, arranged in strings. DCT causes a general increase in toughness in Co-free grades, a decrease being always observed for the two Co-containing grades here investigated. A general decrease of the wear rate by increasing hardness was shown by conventionally treated HSS. The decisive role of primary carbides distribution could be verified in protecting the softer metallic matrix from the abrasion caused by the hard carbides embedded in the counterpart high speed steel. From this viewpoint the more homogenous distribution in PM grades resulted in a remarkably higher wear resistance than wrought grades, even at comparable hardness level. DCT significantly influences the wear resistance of wrought steels, because of the prominent role played by the martensitic matrix. A marked increase was shown by HS6-5-2, whilst a general worsening was displayed by HS6-5-2-5, thus highlighting the negative role of cobalt. The result is confirmed by the two PM grades. While the Co-free grade HS6-5-3 displayed a contained but defined increase in wear resistance, HS6-5-3-8 showed a general increase caused by DCT. In the light of results here presented cobalt seems to play a negative effect with respect to the low temperature conditioning of martensite, even if an experimental support to this assumption was not reported in this paper.

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## ABSTRACT

### EFFETTO DEL TRATTAMENTO CRIOGENICO SULLE PROPRIETA' DI ACCIAI RAPIDI FORGIATI E DA POLVERI

**Parole chiave:** acciaio, trasform. di fase, met. delle polveri, trattamenti termici, metallografia, prove meccaniche, tribologia, proprietà

Nel presente lavoro si propone uno studio dell'influenza del trattamento criogenico (Deep Cryogenic Treatment, DCT) sulle proprietà di quattro acciai rapidi, due dei quali (HS6-5-2 E HS 6-5-2-5) forgiati, gli altri due (HS6-5-3 AND HS 6-5-3-8) ottenuti da polveri. Le analisi HS6-5-2-5 e HS 6-5-3-8 costituiscono una variante delle HS6-5-2 e HS6-5-3 con l'aggiunta del 5% ed 8%Co, rispettivamente (Tab.1). Si è considerato l'effetto del trattamento criogenico (-180°C per 24 ore) eseguito sia prima (C3T, C2T) che al termine dei cicli di rinvenimento (3TC) (Tab.2). I trattamenti criogenici sono stati eseguiti impiegando un impianto frigorifero in grado di controllare la velocità di raffreddamento attraverso PLC. La variante di trattamento con raffreddamento criogenico finale non modifica la durezza dei materiali in modo significativo. Realizzato subito dopo tempra invece il trattamento causa un anticipo del picco di durezza secondaria (Fig. 2) per cui è necessario adottare temperature di rinvenimento inferiori a quelle impiegate convenzionalmente per evitare il calo di durezza dato da fenomeni di coalescenza dei carburi. La tenacità a frattura apparente ( $K_a$ ) evidenzia una dipendenza

significativa dalla durezza e dalla distribuzione dei carburi primari. Il valore di  $K_a$  diminuisce all'aumentare della durezza (Fig. 3). Gli acciai forgiati, a pari durezza, evidenziano valori di tenacità inferiori a quelli per polveri a causa della peggior distribuzione dei carburi primari allineati in stringhe (Fig.1). Il trattamento criogenico produce un incremento generale di tenacità nei due acciai privi di cobalto, mentre un si osserva un calo nelle leghe contenenti tale elemento.

La resistenza all'usura, misurata attraverso prove di strisciamento a secco contro un'antagonista in acciaio rapido ASP2052 (70HRC), aumenta all'aumentare della durezza nei materiali trattati convenzionalmente (Fig.4). La distribuzione dei carburi primari si dimostra fondamentale per la protezione della matrice metallica relativamente più tenera dall'abrasione dei carburi primari dell'antagonista (Fig.5). Da questo punto di vista la distribuzione più fine ed omogenea negli acciai da polveri si riflette, a pari durezza, in una miglior resistenza all'usura rispetto agli acciai forgiati. Il trattamento criogenico, dal canto suo, evidenzia un'influenza significativa proprio sugli acciai forgiati, laddove la matrice metallica esercita un ruolo prevalente. Si riscontra un notevole incremento della resistenza all'usura nell'acciaio HS6-5-2, mentre una diminuzione è stata osservata nel HS6-5-2-5, ribadendo il ruolo negativo del cobalto (Fig.6). Il risultato è confermato dagli acciai da polveri. Mentre la lega PM HS 6-5-3 evidenzia un contenuto ma definito incremento della resistenza all'usura, HS 5-5-3-8 denota un generale deterioramento delle proprietà antiusura in seguito a trattamento criogenico.