
A study of the strengthening mechanisms in the novel precipitation-hardening KeyLos[®] 2001 steel

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Abstract: KeyLos[®] 2001 is a new precipitation-hardening steel especially developed for plastic moulds. In this study the precipitation stage of KeyLos[®] 2001 steel has been investigated and compared to the results obtained with 17-4 PH steel. Precipitation-hardening has been carried out at three different temperatures and the stages of hardening and overageing have been studied in order to clarify the hardening mechanisms.

It has been found that hardening and softening mechanism during the precipitation-hardening treatment occur at higher temperatures and in correspondence with more prolonged treatment times than those typical for the best known 17-4 PH steel; hardness is then expected to remain stable also for very extended mould lives.

Microstructural investigations by means of Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) have also been carried out and the microstructural parameters responsible for the hardening and overageing have been pointed out.

Keywords: plastic mould steels; strengthening mechanisms; precipitation-hardening steel; age-hardening treatment; primary carbides.

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1 Introduction

The major problems encountered during the fabrication of large plastic moulds using hardening steels are represented by the risk of distortion or even fracture during quenching, the high costs of finishing the premachined and hardened mould and the difficulty of achieving uniform characteristics throughout the quenched and tempered block.

KeyLos[®] 2001 is a new precipitation-hardening steel especially developed for plastic moulds. The final mechanical properties are obtained by means of an age-hardening treatment that can be carried out at relatively low temperatures on the semifinished mould. The use of a precipitation-hardening steel results in the advantage that null or minimum deformation is encountered during the preliminary solution-hardening treatment, with no risk of cracking; the mould is also easily machined out of the solution-treated and stress-relieved block, and high and homogeneous mechanical properties are achieved throughout the whole mould, regardless of its size and complexity.

In the present research work the hardening and overageing stages of the precipitation-hardening treatment have been studied in order to investigate the microstructure evolution and the related hardening and softening mechanism. This subject has been thoroughly studied in the literature; in relatively recent papers a precipitation-hardening model has been developed for maraging steels (Sha, 2000;

Wilson, 1997; 1998). The model, described in detail in the experimental part of the present paper, estimates the process activation energy starting from Vickers hardness versus age-hardening treatment time curves. Sha (2000) showed that the age-hardening behaviour in maraging steels and nickel-base superalloys followed the classical growth and coarsening theories, and could be quantified using just a few accurately measured hardness points.

Hsiao *et al.* (2002) studied microstructural development and ageing reactions in a commercial 17-4 PH stainless steel. He found that after solution treatment, the steel produced a uniform matrix, which consisted chiefly of lath martensite with micro twins. Transmission Electron Microscopy (TEM) revealed copper precipitates in the specimen aged at 480°C for one hour, *i.e.*, the peak ageing condition. However, at a higher ageing temperature, 620°C, in addition to the formation of copper precipitates, the reformation of austenite was noticed.

2 Experiment details

In the present study two steels have been investigated: 17-4 PH, a well-known precipitation-hardening martensitic steel, and KeyLos[®] 2001, a new precipitation-hardening steel. KeyLos[®] 2001 steel is obtained through a special 'super clean' production process, which allows the achievement of a high level of micro purity; it is supplied in the solution-treated condition and it is stress-relieved, in order to reach hardness values between 310 and 350 HB, which are typical of a prehardened steel. If high mechanical properties are required and a homogeneous hardness is needed throughout the whole mould, KeyLos[®] 2001 steel can be further hardened up to 350–450 HB, by means of an age-hardening process. Since the hardening treatment occurs below the phase transformation points, this process can also be carried out on a semifinished mould, causing minimal deformation and no cracking during the hardening phase. The mechanical characteristics of this steel are adaptable to a wide range of applications that are more widespread than those covered by other grades that are normally used in this field. The chemical composition of the investigated steels is shown in Table 1.

Table 1 The chemical composition of the investigated steels (weight percent)

<i>Steel</i>	<i>C</i>	<i>Mn</i>	<i>Cr</i>	<i>Si</i>	<i>Ni</i>	<i>V</i>	<i>Mo</i>	<i>Cu</i>	<i>Nb</i>
17-4 PH	0.03	0.60	15.33	0.44	4.00	–	0.09	3.28	0.34
KeyLos [®] 2001	0.15	0.72	0.16	0.31	3.26	0.09	3.07	0.10	–

The specimens have been machined out of the solution-treated forged blocks in the form of prismatic samples 5 × 10 × 15 mm and then precipitation hardened at different temperatures for various times, followed by water cooling to room temperature.

As reported in *High temperature property data: Ferrous Alloys* (ASM International Metals Park, 1988; Davis, 1994), 17-4 PH steel has been heat treated at 480°C, 565°C and 620°C for up to eight hours; ageing was not extended beyond eight hours since the overageing phase with progressive hardness decrease was already reached. KeyLos[®] 2001 steel has been heat-treated at 550°C, 590°C and 630°C for times up to 20 hours, in

order to investigate the overageing phase also for the lowest ageing temperature. Vickers hardness measurements have been carried out with a load of 100 g and each value is the mean of at least three measures.

Microstructural investigations by means of SEM and TEM have also been carried out and the microstructural constituents responsible for the hardening and overageing have been pointed out.

The strengthening mechanisms in 17-4 PH steel and in the novel precipitation-hardening KeyLos[®] 2001 steel have been studied according to a precipitation-hardening model originally developed for maraging steels (Sha, 2000; Wilson, 1997; 1998). This model estimates the process activation energy starting from Vickers hardness versus age-hardening treatment time curves. Strengthening phenomena have been studied separating the initial precipitation phase and the following overageing phase of the age-hardening treatment.

The increase in hardness in the initial phase is described by the following equation derived by Wilson (1997):

$$\Delta H = (Kt)^n \quad (1)$$

where:

ΔH = represents the hardness increase owing to the precipitation phenomenon with reference to H_0 (as quenched hardness)

K = a parameter depending on temperature

t = age-hardening treatment time

n = a time exponent slightly dependent on temperature.

The decrease in hardness in the overageing phase is described by a different equation, again derived by Wilson (1997):

$$\left(\frac{1}{\Delta H}\right)^3 = \left(\frac{1}{\Delta H_0}\right)^3 + M(t - t_0) \quad (2)$$

where:

ΔH_0 = represents the hardness increase at the beginning of the coalescence time t_0 , that is, the difference between H_{max} (peak hardness) and H_0 (as quenched hardness)

M = a parameter depending on temperature.

The theoretical derivation of Equations (1) and (2) is hereafter reported, in accordance with the Wilson development (Wilson, 1997).

During the early stages of ageing, hardening is due to coherency strains. The equation relating the increase in shear stress $\Delta\tau$ to precipitate size r and their volume fraction f has the form:

$$\Delta\tau = (\kappa\varepsilon)^2 \mu \left(\frac{3}{2\pi b}\right)^{\frac{1}{2}} r^{\frac{1}{2}} f^{\frac{1}{2}} \quad (3)$$

where:

k = a constant

μ = matrix shear modulus

b = matrix Burgers vector

ε = specific difference in the lattice parameters between the precipitates and the matrix.

If the strains are isotropic, the increase in yielding stress $\Delta\sigma_y$ has the form:

$$\Delta\sigma_y = 2\Delta\tau = q\Delta H. \quad (4)$$

Combining Equations (3) with (4), it is possible to obtain:

$$\Delta H = A r^{\frac{1}{2}} f^{\frac{1}{2}} \quad (5)$$

where:

$$A = \frac{2}{q} (\kappa\varepsilon)^{\frac{3}{2}} \mu \left(\frac{3}{2\pi b} \right)^{\frac{1}{2}} r^{\frac{1}{2}} f^{\frac{1}{2}}. \quad (6)$$

It is further assumed that precipitates are spherical, and that their radius r with time t is given by the Zener parabolic relationship (Zener, 1949):

$$r = \alpha \sqrt{(D \cdot t)} \quad (7)$$

where:

D = diffusion coefficient of elements forming the precipitates in the matrix at the temperature T in Kelvin degrees

α = a constant related to their concentration in the alloy and to solid solubilities of precipitates in the matrix.

The Burke equation (Burke, 1965) relating the transformed fraction y to ageing time t , is given by:

$$y = 1 - \exp[-(kt)^m] \cong f / f_0 \quad (8)$$

where f_0 is the precipitate volume fraction in the equilibrium condition.

In the early stages of ageing, when $kt \ll 1$, this equation reduces to:

$$y = (kt)^m. \quad (9)$$

This is tantamount to saying that precipitates do not impinge and that diffusion fields do not overlap, which is reasonable for the early stages of ageing. During this period, in fact, hardening is due to coherency strains.

Using this approximation it is possible to write:

$$f = f_0 \cdot (kt)^m. \quad (10)$$

Combining Equations (3), (4), (7) and (10) it is possible to obtain:

$$\Delta H = \frac{2}{q} (\alpha f_0)^{\frac{1}{2}} (k\varepsilon)^{\frac{3}{2}} \mu \left(\frac{3}{2\pi} \right)^{\frac{3}{2}} D^{\frac{1}{4}} k^{\frac{m}{2}} t^{\left(\frac{m+1}{2} \right)} = (Kt)^n. \quad (11)$$

Combining this equation with Equation (1) it is possible to obtain:

$$n = 1/4 + m/2 \quad (12)$$

$$K^n = B D^{\frac{1}{4}} k^{\frac{m}{2}} \quad (13)$$

where:

$$B = \frac{2}{q} (\alpha f_0)^{\frac{1}{2}} (k\varepsilon)^{\frac{3}{2}} \mu \left(\frac{3}{2\pi} \right)^{\frac{3}{2}} = A (\alpha f_0)^{\frac{1}{2}}. \quad (14)$$

Now it is possible to show that K is an ageing temperature-dependent parameter; starting from the Equation (13):

$$K^n = B D^{1/4} k^{m/2} \\ = B (D_0 \exp -(Q_h/RT))^{1/4} [k_0 \exp -(Q_h/RT)]^{m/2} \quad (15)$$

$$= (K_0 \exp -(Q_h/RT))^n \quad (16)$$

it is possible to assert that the diffusion coefficient D , k and K are related by an Arrhenius equation, where Q_h is the process activation energy for the initial hardening stage.

Applying the logarithm to Equations (15) and (16) it is possible to obtain:

$$n \cdot \ln K = \ln B + \frac{1}{4} \ln D_0 + \frac{m}{2} \ln k_0 - \frac{1}{RT} \left(\frac{Q_h}{4} + \frac{mQ_h}{2} \right) = n \ln K_0 - \frac{Q_h n}{RT} \quad (17)$$

and

$$n \ln K = -Q_h n / RT + \ln B + \frac{1}{4} \ln D_0 + \frac{m}{2} \ln k_0 \quad (18)$$

where R is the gas constant (8,31 J/mol °K).

A plot of $n \ln K$ versus n/T gives a straight line, whose slope is equal to $-Q_h/R$ and whose ordinate axis intercept I is given by:

$$I = \ln B + \frac{1}{4} \ln D_0 + \frac{m}{2} \ln k_0 = n \ln K_0. \quad (19)$$

It is then possible to calculate the process activation energy for the initial hardening (Q_h) phase.

The derivation of Equation (2) assumes that precipitation hardening is due to Orowan bowing (Orowan, 1947) and that yielding is isotropic; the increase in shear stress $\Delta\tau$ on overageing then has the form:

$$\Delta\tau = \frac{\mu b}{\lambda} = \frac{\Delta\sigma_y}{2} = \frac{q\Delta H}{2} \quad (20)$$

where:

- q = a proportionality constant relating the increase in yield strength $\Delta\sigma_y$ to the increase in hardness ΔH
- λ = interparticle spacing (*i.e.*, the surface-to-surface spacing of precipitates)
- μ = matrix shear modulus
- b = matrix Burgers vector.

In addition to the Orowan bowing of dislocations, the model assumes that the final complete volume fraction of precipitate f_∞ occurs at the onset of coarsening. Assuming that the precipitates are spherical and have radius r , then the relationship between f_∞ , λ and r is given by Martin (1980; 1996):

$$\lambda = \left\{ 1.18 \sqrt{\left(\frac{2\pi}{3f_\infty}\right)} - 2 \sqrt{\left(\frac{2}{3}\right)} \right\} r = pr. \quad (21)$$

On overageing incoherent precipitates form with negligible strain energy and particles coarsen. Large precipitates grow at the expense of small ones, and the spacing between precipitates λ increases with ageing time. The relationship between precipitate size r and ageing time t is given by the coarsening theory developed by Greenwood (1956):

$$r^3 = (r_0)^3 + \frac{8}{9} \frac{D}{kT} \sigma \cdot c_\alpha \cdot \Omega \cdot (t - t_0) \quad (22)$$

where:

- k = Boltzmann constant ($1,38 \cdot 10^{-23}$ J°K)
- T = temperature (Kelvin)
- D = diffusion coefficient for solute in the matrix at temperature T
- σ = surface energy of incoherent precipitates
- c_α = solid solubility of precipitates in the matrix
- Ω = atomic volume of precipitates
- r_0 = radius of precipitates at the onset of coarsening time t_0 .

Combining Equations (20), (21) and (22) it is possible to obtain Equation (2):

$$\left(\frac{1}{\Delta H}\right)^3 = \left(\frac{1}{\Delta H_0}\right)^3 + M(t - t_0) \quad (23)$$

where:

- ΔH_0 = represents the hardness increase at the beginning of the coalescence time t_0 , that is, the difference between H_{max} (*peak hardness*) and H_0 (*as quenched hardness*)
- M = a parameter depending on temperature

$$M = \frac{8}{9} \left(\frac{pq}{2\mu b} \right)^3 \sigma \cdot c_\alpha \Omega \frac{D}{kT} \quad (24)$$

$$M = \frac{8}{9} \left(\frac{pq}{2\mu b} \right)^3 \sigma \cdot c_\alpha \Omega \frac{D_0}{kT} \exp\left(-\frac{Q_{oa}}{RT}\right). \quad (25)$$

M is the slope of the line in the graph $(1/\Delta H)^3$ versus ageing time t .
 Rearranging and taking logarithms of Equation (25) gives:

$$\ln MkT = 3 \ln \left(\frac{pq}{2\mu b} \right) + \ln \frac{8}{9} \sigma \cdot c_\alpha \Omega D_0 - \frac{Q_{oa}}{RT}. \quad (26)$$

A plot of $\ln (MkT)$ versus $1/T$ therefore gives a straight line whose slope is equal to $-Q_{oa}/R$; from this plot it is then possible to calculate the activation energy for the final overageing (Q_{oa}) phase.

3 Discussion

Vickers hardness versus age-hardening treatment time curves for 17-4 PH steel and KeyLos[®] 2001 steels, at the investigated precipitation-hardening temperatures, are reported in Figure 1 and in Figure 2, respectively.

Although a similar behaviour can be observed in the effect of ageing temperature and times, compared with 17-4 PH steel, ageing of KeyLos[®] 2001 steel occurs at higher temperatures; peak hardness is obtained for more prolonged ageing times for KeyLos[®] 2001 steel, and starting of overageing is delayed towards longer ageing times.

Figure 1 Hardness versus age-hardening treatment time curves for 17-4 PH steel

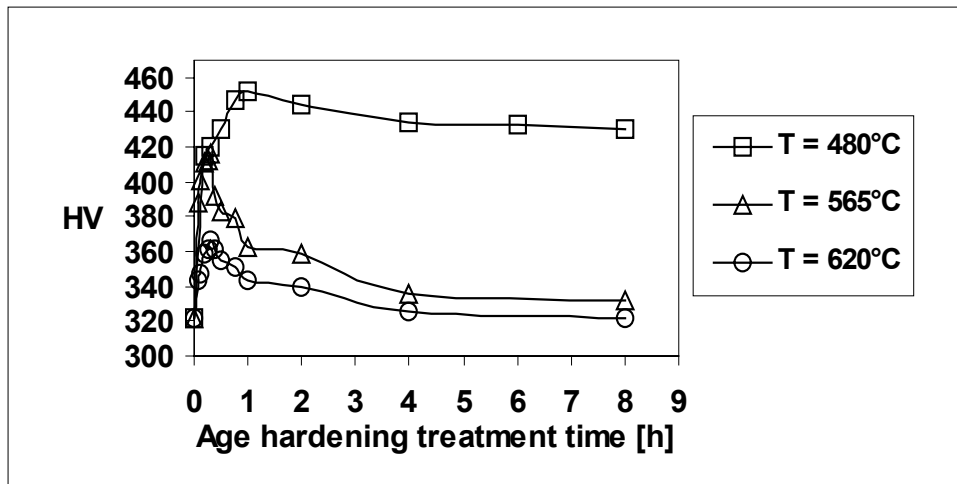
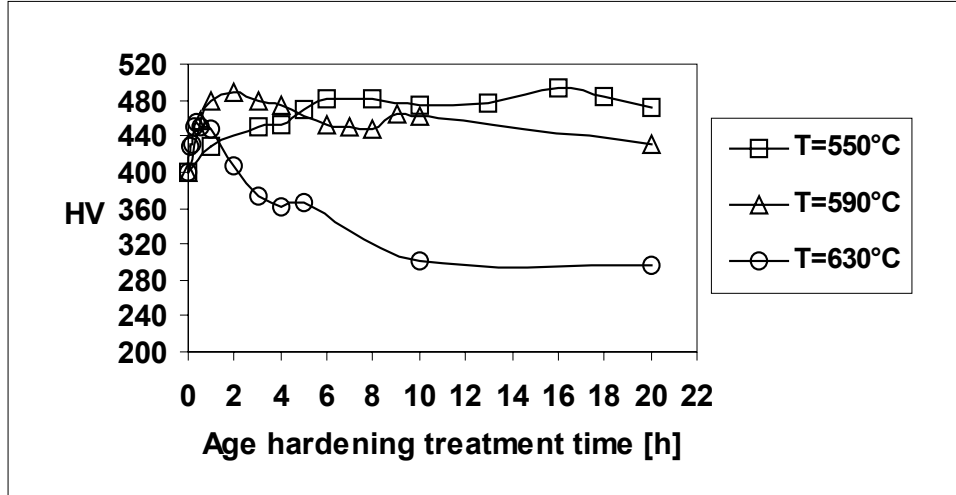


Figure 2 Hardness versus age-hardening treatment time curves for KeyLos[®] 2001 steel

The parameters n and K along with the process activation energy Q_h for the initial hardening phase, as well as the parameters k , M and the process activation energy Q_{oa} for the overageing phase for 17-4 PH steel are reported in Table 2 and in Table 3, respectively.

Table 2 n , K and Q_h for the initial phase for 17-4 PH steel

T [$^{\circ}\text{C}$]	n	K [sec^{-1}]	Q_h [kJ/mol]
480	0,2174	1,4724E+06	80.95
565	0,2390	1,7519E+05	
620	0,5152	1,3631E+00	

Table 3 k , M and Q_{oa} for the over-ageing phase for 17-4 PH steel

T [$^{\circ}\text{C}$]	M [h^{-1}]	k [J/K]	Q_{oa} [kJ/mol]
480	4,4866E-08	1,38E-23	340
565	1,2366E-04	1,38E-23	
620	1,0893E-04	1,38E-23	

The same parameters for KeyLos[®] 2001 steel are reported in Table 4 and in Table 5, respectively.

Table 4 n , K and Q_h for the initial phase for KeyLos[®] 2001 steel

T [$^{\circ}\text{C}$]	n	K [sec^{-1}]	Q_h [kJ/mol]
550	0,5344	1,4414E-01	76.21
590	0,2998	4,8986E+02	
630	0,4978	2,4828E+00	

Table 5 k, M and Q_{oa} for the over-ageing phase for KeyLos[®] 2001 steel

$T [^{\circ}\text{C}]$	$M [\text{h}^{-1}]$	$k [\text{J/K}]$	$Q_{oa} [\text{kJ/mol}]$
550	5,757E-08	1,38E-23	
590	1,632E-06	1,38E-23	314
630	2,9358E-06	1,38E-23	

The process activation energy Q_h in the initial phase of the age-hardening treatment shows quite similar values for both 17-4 PH steel and KeyLos[®] 2001 steel. Roughly similar values are observed for the process activation energy Q_{oa} for the overageing phase of the treatment.

In the initial phase the process activation energy Q_h has values definitely lower than those which are typical for the diffusion of substitutional elements responsible for hardening in these alloys (Robino *et al.*, 1994). The time exponent n presents lower values than those expected for the nucleation and growth processes (Robino *et al.*, 1994).

Robino studied the hardening response of 13-8 PH Mo stainless steel by hardness measurements following ageing in the temperature range normally specified for this alloy (510°C to 590°C). According to Robino *et al.* (1994), low values of Q_h and n in many maraging steels are due to the effect of the intermetallic particles nucleation along or near dislocations and the subsequent growth of these precipitates via enhanced dislocation pipe diffusion. This behaviour appears physically reasonable because of the high dislocation density of the martensitic matrix.

In analysing hardness versus age-hardening treatment time curves for both steels under investigation, notwithstanding the different effects of temperature and time previously pointed out, it is evident that the hardness increases more rapidly at high temperatures to the peak value and that the time needed to achieve the peak hardness decreases at increasing temperature. Finally, beyond the peak hardness, hardness decreases more rapidly at high temperatures compared with low temperatures. These characteristics are in agreement with previous investigations (Robino *et al.*, 1994) and with the well-known effect of both temperature and time on nucleation, growth and coalescence of hardening phases.

The absence of an identifiable incubation period during the age-hardening treatment suggests that intermetallic particles nucleation occurs during the initial phase of the treatment.

The initial hardening phase is due to the precipitates nucleation and growth from solid solution, while the following softening phase may correspond not only to the coarsening of the precipitates to form the overaged structure, but also either to the recovery of martensite, to the reversion of martensite to austenite, or to a mixture of these three reactions (Suzuki *et al.*, 2001).

It is reasonable to assert that, in 17-4 PH steel and in KeyLos[®] 2001 steel, precipitates nucleation occurs along dislocations that are copiously present in the matrix because of the fast cooling rate from the solubilisation temperature. Thus, in the initial phase of the age-hardening treatment, the steels studied in this article appear to behave similarly compared to those reported in Robino *et al.* (1994).

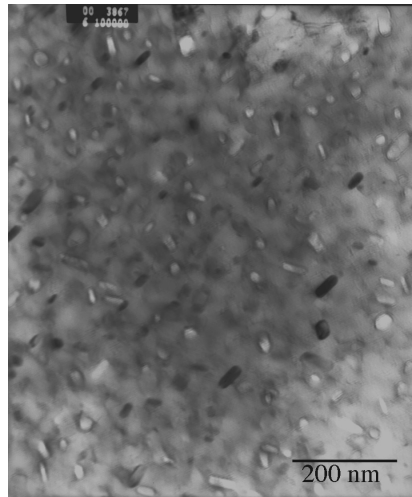
In the overageing phase, the process activation energy Q_{oa} for both 17-4 PH steel and KeyLos[®] 2001 steel results higher than that obtained in Suzuki *et al.* (2001) for maraging steel.

Since the hardness versus age-hardening treatment time curves for 17-4 PH steel have comparable results with those obtained in Hsiao *et al.* (2002), it is possible to suppose that hardening mechanisms are also the same with those described in Hsiao *et al.* (2002). In order to confirm such a hypothesis, microstructural investigations by means of TEM have been carried out on 17-4 PH steel on the sample that has been treated at 620°C for four hours. More precisely, possible undesirable retained austenite presence, as a result of tempering, has been looked for and copper-rich precipitates presence responsible for hardening has been investigated, such as those reported in Hsiao *et al.* (2002).

The absence of retained austenite has been confirmed by TEM analysis.

A detail of this sample is shown in Figure 3: small, thinly dispersed incoherent particles are evident, whose morphology and distribution are typical of copper-rich clusters.

Figure 3 TEM micrograph: detail of the 17-4 PH steel sample kept at 620°C for four hours



Therefore, the alloy hardening is really due to these precipitates, which are coherent with the matrix (GP zones) in the initial phase of the treatment and may behave as obstacles for dislocation movements, producing a considerable increase of the hardness. These precipitates grow and become incoherent with the matrix in the overageing phase of the treatment, causing the decrease of mechanical properties.

TEM and SEM analyses on KeyLos[®] 2001 steel have been carried out on the as-quenched condition sample and on the sample heat-treated at 630°C for 20 minutes (peak-hardness condition).

A SEM micrograph of the as-quenched condition sample is shown in Figure 4. The presence of dispersed particles in the matrix (the light, largely spaced particles in the figure) can be easily observed. The quantitative chemical analysis of these particles shows that these are primary molybdenum carbides (Figure 5) not completely solubilised during austenitising. In the as-quenched condition, in fact, provided that every preexistent carbide is solubilised, only a martensite matrix is expected to be found. Molybdenum carbides, on the other hand, have a very high-solution temperature, and in case their size

is not small enough in the starting material (according to the thermal treatment in the course of the bar production in the steel work), they are not completely dissolved during the austenitising treatment.

Figure 4 SEM micrograph: detail of the KeyLos® 2001 steel as-quenched condition sample

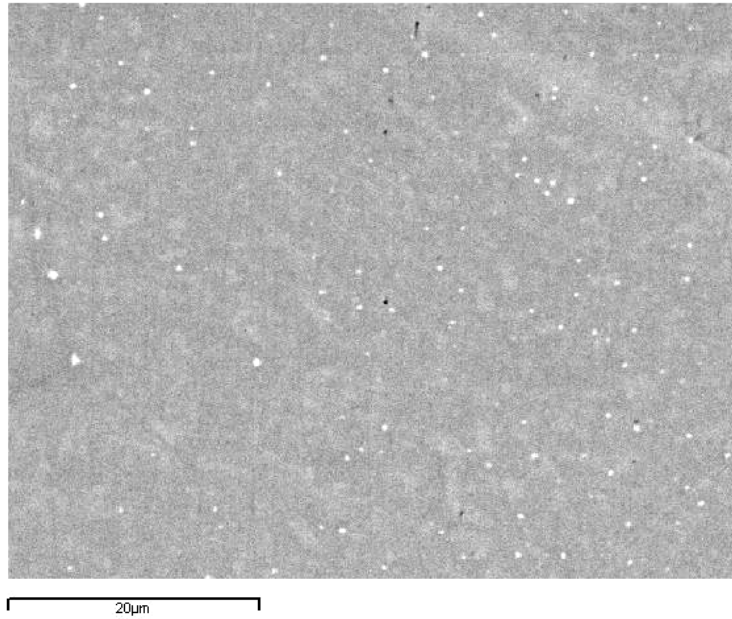
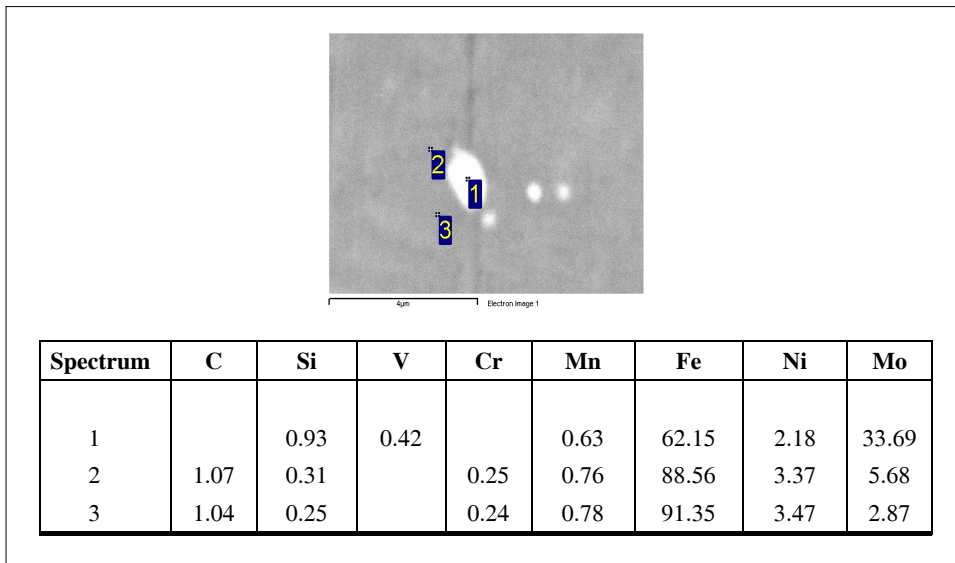
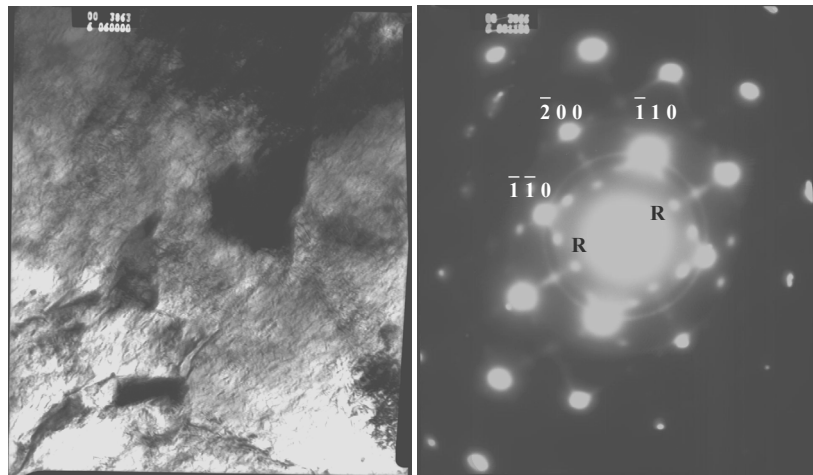


Figure 5 SEM micrograph of a detail of the KeyLos® 2001 steel as-quenched condition sample and quantitative analysis of zones 1, 2 and 3 (all results in weight percent)



TEM micrographs of KeyLos[®] 2001 steel sample aged at 630°C for 20 minutes are shown in Figure 6. Besides primary molybdenum carbides (dark zones in the centre of the left image), thin precipitation of particles in the form of parallel tags couples is evident. The Selected Area Diffraction Pattern (SADP) in this figure shows principal spots corresponding to the ferritic matrix; from the chamber constant and the R value an a_0 body centred cubic cell parameter equal to 2,90 Å is in fact obtained. In the same SADP thin elongated streaks around the principal spots are observed; their occurrence confirms the presence of precipitates in the form of parallel tag couples, coherent with the matrix, which may correspond to metastable precipitates such as GP zone (Edington, 1994). It is possible to suppose that these precipitates are responsible for the alloy hardening.

Figure 6 TEM micrograph: detail of the sample of KeyLos[®] 2001 steel kept at 630°C for 20 minutes and the corresponding diffraction pattern



4 Conclusions

The research work aimed at investigating the microstructure evolution and the related hardening and softening mechanism during the precipitation-hardening treatment of the new precipitation-hardening KeyLos[®] 2001 steel especially developed for plastic moulds.

The final mechanical properties, which are obtained by means of an age-hardening treatment carried out on semifinished moulds at relatively low temperatures, comply with the needs of plastic moulds; high and homogeneous mechanical properties are achieved throughout the whole mould, regardless of its size and complexity, and this makes KeyLos[®] 2001 steel particularly suitable for large-size moulds, as null or minimum deformation is encountered during the preliminary solution treatment, with no risk of cracking, and then the mould is easily machined out of the solution-treated and stress-relieved block.

It has been found that hardening and softening mechanisms during the precipitation-hardening treatment occur at higher temperatures and in correspondence with more prolonged treatment times than those typical for the best known 17-4 PH steel; hardness is then expected to remain stable also for very extended mould lives.

It has been found that the process activation energy Q_h in the initial phase of the age-hardening treatment shows quite similar values for both 17-4 PH steel and KeyLos[®] 2001 steel. Roughly similar values are observed for the process activation energy Q_{oa} for the overageing phase of the treatment.

The low value of Q_h for both 17-4 PH steel and KeyLos[®] 2001 steel has been due to the effect of the intermetallic particles nucleation along or near dislocations and the subsequent growth of these precipitates via enhanced dislocation pipe diffusion.

Primary molybdenum carbides and thin precipitation of particles in the form of parallel tags couples have been observed in the microstructure using TEM and SEM analyses.

Selected area diffraction pattern has shown thin streaks that confirm the presence of precipitates in the form of parallel tag couples, coherent with the matrix, which may correspond to metastable precipitates such as GP zone.

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