LOW ALLOYED Cr-Mo SINTERED STEELS UPDATE

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Abstract
Chromium and molybdenum are alloying elements well known in the ingot steel industry due to their positive influence in hardenability and mechanical properties. In spite of the fact that molybdenum was introduced in PM steels in 70's, chromium remains in a second plane due to its peculiarities for sintering: its high affinity for oxygen requires a well controlled atmosphere, and only recently industry is capable to use it in a reliable way. In this paper a review of the combined use of chromium and molybdenum is made, from their early use in laboratories till today, where it can be found for structural parts sintered in mesh belt furnaces.
Keywords: powder metallurgy, chromium and molybdenum steels, sintering atmosphere

INTRODUCTION

In conventional ingot steels for heat treatments, chromium and molybdenum are widely used as alloying elements [1]. Both are carbide formers and alpha phase stabilizers, and both improve greatly the hardenability and the hardening effect due to quenching in the presence of carbon. Moreover, molybdenum advances the bainitic transformation in time, with the chance to obtain bainitic steels with high toughness, over a wide range of cooling rates. Like most of the alloying elements in steels, due to solid solution hardening effect, they improve tensile and yield strength as well as hardness. As a result, low-alloyed Cr-Mo steels are used when a carbon steel (for the same amount of carbon) requires hardenability, tensile strength and good wear behaviour. The steel can be effectively oil quenched, which minimizes the risk of crack formation encountered by water quenching. Molybdenum additions compensate for the low ductility and avoid the brittleness produced by high tempering temperatures. The high affinity of chromium for carbon allows it to replace iron in cementite structure, producing the complex carbide $(Fe,Cr)C_3$ [2, 3].

Due to the peculiarities of chromium for sintering, its use during the 60's was limited to research at the laboratory field [4-6]. Nevertheless, low chromium alloyed steels fulfil all the requirements of most structural components, and satisfy the latest demands of the market.

In the mid 80's, several authors [7-11] began to work with low chromium content prealloyed powders, water or oil-atomised. These first experiences were related to processing conditions far from those conventionally used in industry, and using an extremely precise dew point of sintering atmosphere to avoid oxidation during sintering. But although the market was demanding high performance materials, industry was not ready for the particularities of these materials.

Additional advantages of chromium as an alloying element for PM steels are its low price, and easy recycling as compared to copper. This kind of alloy can be directly used as scrap in pig iron manufacture [12, 13].

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Together with chromium and molybdenum, nickel is the third alloying element most used in the ingot steel industry, and one of the elements most used in the PM manufacture of steels. Nickel alloyed steels can be recycled as scrap, but after its being classified as a dangerous substance by the European Union[1] [14-16], several studies and investigations were made in Europe to replace nickel in the PM industry, bringing in new families of nickel free alloys with high performance.

LOW CHROMIUM PM STEELS AND THEIR DEVELOPMENT

The most widely used elements in the PM industry, concerning low alloy steels (copper, molybdenum, nickel and phosphorus), fulfil enough of the requirements regarding mechanical properties, and when combined with the proper amount of carbon, they provide good dimensional behaviour. They have, moreover, low affinity for oxygen, which allows their use in industry with certain reliability from the point of view of atmosphere control. However, they are expensive materials, they could be difficult for recycling (as copper is) or have other related disadvantages (such as the difficult handling of nickel).

Alloying elements such as chromium (or manganese, and in a lesser sense silicon) have a powerful effect on hardenability and the increase of mechanical properties due to solid solution hardening. For a successful sintering process of low chromium alloyed sintered steels, the partial oxygen pressure in sintering atmosphere must be taken into account, so efforts are focused on solving and controlling the oxidation problem during sintering [17]. From the beginning, the main problem when these elements were used, is the oxide layer that covers the donor particle surface. Diffusion is completely prevented and the oxide layer acts as a barrier that avoids good homogenization and wider particle contacts. Using ferroalloys as alloying agents can reduce those effects.

During the 60's decade, and due to the complexity of sintering steels with chromium, its use was limited to the laboratory [4, 18]. Motooka et al. [19] studied the effect of oxygen on the final properties using different atmospheres: vacuum, hydrogen, and dissociated ammonia. Their results show how oxygen content in these sintered low chromium alloyed steels determines the toughness. Studies of chromium additions have been running parallel to those of manganese additions, both elements have similar oxygen affinity, therefore, their sintering conditions are comparable [6]. Studied at the beginning was the effect of chromium on iron for manganese fixed amount. It was obtained that, jointly with Cr, the effect of Mn on strength is positive. This makes sense if it is considered that manganese is usual in the ingot metallurgy of steel, and presents similar problems regarding oxygen affinity. In 1977, Dudrová and Šalak [20] investigated chromium additions in the range of 2-5 wt.%. In form of ferrochromium and despite the low sintering temperature used, 1100ºC in dissociated ammonia, they improved the ultimate tensile strength. The incomplete reduction of the oxide layer in the ferrochromium particles at such low temperatures prevents good microstructural homogenisation. This has been the main problem that has marked the investigations of this kind of material till now.

Many of the conclusions obtained for manganese could be applied to low chromium sintered steels but chromium does not exhibit the mass transport mechanism that singularises manganese, that is, evaporation-condensation which promotes a sintering process in which coexist solid and gaseous states [21-25]. In chromium mixed steels, the sintering takes place through the conventional solid-solid mass transfer, and this will affect the microstructure and the final properties.

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[1] The EU has classified Ni as carcinogenic, level 3. This category refers to the possible carcinogenic effect on the human body.

At this stage of the investigations, chromium was added in the form of C_3Cr_2 carbides, but it demanded higher sintering temperatures, up to 1250ºC, and longer sintering times to allow the complete dissolution of the carbides in the austenite. After five hours of sintering, the measured tensile strength was comparable to that obtained when chromium was added as ferrochromium, although the homogenisation of the microstructure was higher [26].

When fully prealloyed Fe-Cr powders began to be used, the main problem was the high oxygen content (>0.5%), even after H_2 annealing. Its production requires higher sintering temperatures, but it gives a homogenised microstructure.

In the mid 80's, several authors began to work with low-alloyed chromium prealloyed powders, both water and oil-atomised. Tengzelius et al. [7, 8] selected the Fe-1Cr-1Mn-C system, and performed sintering process at 1250ºC in N_2-$5H_2$ atmosphere, using a close box with the aim to avoid the oxidation during the process. In this work, manganese and chromium were added as ferroalloys. Through a good and controlled sintering process, it can be seen how mechanical properties increase with these alloying elements present. Ichidate et al. [9] and Karasumo et al. [10] performed a deep analysis of oil-atomised prealloyed powder properties with different compositions: 1% Cr (4100S), 2% Cr (2CrMS) and 3% Cr (3CrMVS) with very low oxygen content (<0.08%) thanks to the oil-atomisation process. They characterized not only the fatigue or wear behaviour, but also the effect of nitriding and carburising as well the heat treatment effect, or the influence of steam treatment on the properties. By sintering at 1250ºC, with good control of the dew point, or sintering in a vacuum, good results can be obtained, but the powder production process is too expensive. Water atomisation of the powder would efficiently reduce the price, but the problem is to reduce the superficial chromium oxide layer associated to the process (Fig.1).

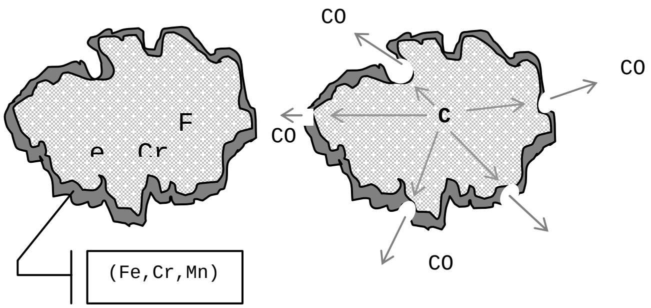

Fig.1. Diagram of vacuum reductant process in carbon presence [11].

Prealloyed powders are usually annealed in H_2, but in the presence of elements such as chromium or manganese, it is difficult to avoid oxygen contents bellow <0.2%. Looking for an alternative to the annealing after the water atomising process, the work of Ogura et al. [11], succeeded in annealing water-atomised powders with Fe-0.7Mn-1.1Cr-0.3Mo in a vacuum by using carbon dissolved in the powders (Fig.1). In 1992, the same company developed a prealloyed powder Fe-1Cr-0.3Mn which, mixed with 0.9% C, offered the best mechanical properties balance sintered at 1200ºC. Afterwards, a new grade Fe-1Cr-1Mo was introduced with better compressibility performance than conventional nickel pre-alloyed powders [27].

The feasibility of reducing oxides from chromium and manganese depends on process temperature and admixed carbon content. So they calculated the needed carbon to reduce the Cr_2O_3 from the carbon activity of the dissolved carbon in austenite (a_c), which produces the equilibrium for a certain partial pressure of oxygen:

$$K_1\sqrt{P_{O_2}} + K_2 P_{O_2} - P\cdot a_c = 0 \qquad (1)$$

Where K_1 and K_2 are the equilibrium constants of the reactions (1) and P is the pressure of the annealing atmosphere.

$$C + \tfrac{1}{2}O_2 \Leftrightarrow CO \qquad C + O_2 \Leftrightarrow CO_2 \qquad (1)$$

Since a_c depends on the carbon content in the austenite, and P_{O2} is a function of the temperature, the carbon content needed to reduce the oxides is a function of the pressure and temperature, as shown in Fig.2.

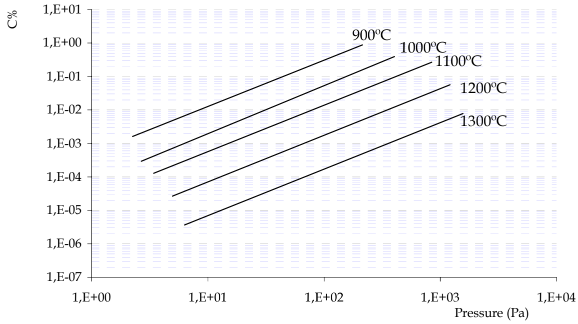

Fig.2. Carbon content needed in the powder particles for reducing Cr_2O_3 at different pressures [11].

The main problem with this reduction is the final control of the carbon content, and the decrease in the compressibility of the powder.

The results obtained by Ogura et. al. [11], were interesting from the point of view of the mechanical behaviour, but the sintering conditions were still far from those employed by industry (1250ºC, in dissociated ammonia atmosphere for 1 hour).

Ramana et al. [28], studied the alloying system of Fe-3Cr alloyed with 1% Mo and 0.9% C additions. The chromium, in this case, was added by using prealloyed Fe-Cr powders. Sintering was done in hydrogen at 1200ºC, with an exhaustive oxygen control in the process. These results showed how molybdenum improves the densification and hardness in the sintered material, allowing more favourable heat treatments. These materials however, did not yet posses the robustness required by the industrial conditions of that period. Moreover, the addition of chromium in the form of a ferrochromium produced heterogeneous microstructures in which the amount of martensite or bainite after quenching is conditioned by the amount of chromium present locally, and it promised a poor steel reliability. More recent studies have compared fully prealloyed powders with an equivalent mix in composition, and sintering in the same strict conditions. These show that the mix with iron powder will never compete with a fully prealloyed powder, mainly on account of the microstructural heterogeneity [29].

Danninger et al. [30] studied, in different steps, the low chromium alloyed sintered steels presenting a comparative study of chromium, molybdenum and tungsten alloyed steels. The best-evaluated material was the molybdenum (1.5Mo) steel, due to the better microstructural homogeneity promoted by liquid phase sintering. In all the materials studied, sintering conditions were still far from those used at the (1240ºC). The same work concluded that chromium alloyed steels can be sintered at this temperature due to the liquid phase formed by the chromium and carbon reaction. In [31] the author continued with special focus on chromium and the Fe-Cr interphase where, due to the different inter-diffusion rate between chromium and iron chromium rich carbides, different chromium contents were detected. In [32] it was remarked for the first time the role that can play carbon in these steels, reducing the importance of the atmosphere, as admixed carbon is acting as reducing agent. In this work there were compared, moreover, steels obtained by mixing elemental powders (either iron or chromium with graphite) or master alloy powders Fe-6Cr.

In 1996, Šalak [12] published a wide study of chromium, manganese and molybdenum low alloyed sintered steels, in which two things were stated with the benefit of avoiding the nickel additions: the synergic effect of alloying with chromium, manganese and molybdenum, together with high temperature sintering, allows the production of components with properties impossible to obtain under conventional conditions of pressing and sintering. At the end of this work, Šalak predicted "the future belongs to manganese, chromium and molybdenum as alloying elements, with the following hypothesis: no dimensional changes, a total amount of alloying elements under 4%, and values for tensile strength after sintering of 1000 MPa".

LOW CHROMIUM SINTERED STEELS AND THEIR IMPLEMENTATION IN THE INDUSTRY

As stated in the introduction, one important factor that has driven the development of chromium as a possible substitute for other alloying elements is the change in environmental policies, for improving health and quality in the work environment, and extending the recycling policy to all steps of the production cycle during the 90s. In Europe, this trend produced a strengthening of the laws regarding hazardous materials. With this aim, in the 90s the industry tried to reintroduce safe elements to improve and increase hardenability and mechanical properties through hardening by solid solution. This is the case of chromium.

In 1994, under the framework of a Brite-Euram project (Vth Framework Programme), different research centres and industries (powders and parts producers) began to work on development nickel free steels for gears production [33]. They concluded with the benefits of these new alloys which could replace those containing nickel without new tool design criteria, based on spring-back measurements on green compacts. From the economic point of view, these new alloys could reduce the cost of raw materials for mass production of parts by about 20%, below that of nickel.

In the same sense, in an EU COPERNICUS Project in 1994 [34], different manganese additions were studied, sintering in hydrogen or rich hydrogen atmospheres, in the range of temperatures of 1120-1300ºC. In [35] Link studied fatigue behaviour in molybdenum alloyed steels sintered at high temperature and, after quenching, as a possible substitution for components subjected to alternating stresses.

Höganäs AB (Sweden), in 1998, introduced chromium as an alloying element through the prealloyed water-atomised powder of composition Fe-3Cr-0.5Mo (commercially Astaloy CrM), which offers an interesting price/performance relationship [36]. This material was presented as an alternative to existing Fe-Cu-Ni-Mo partially prealloyed powder by diffusion bonding.

At this stage, Lindberg [17] and Advirdsson [37] have designed the suitable oxygen partial pressure control process at the three zones in large scale sintering furnaces.

By taking into account Ellingham's diagrams, and keeping oxygen partial pressure below $5x10^{-13}$ atm. it is possible to sinter Astaloy CrM correctly.

At the time of the developments of the above projects, and boosted by the appearance in the market of different low chromium prealloyed powders, several papers appeared related to these materials. In [38], Linqvist compares the prealloyed Astaloy CrM grade properties with those of the well known steel obtained from the pre-diffusion alloyed powder Distaloy AE (pre-diffused powder widely used during the 70's, Fe-2Cu-4Ni-0.5Mo). The work compares both materials sintered at 1120ºC and in atmosphere $90N_2$-$10H_2$, with no such evident difference in properties, but with sintering at 1250ºC, the properties of the new grade were better. If value dispersion appears, it is mainly due to an inadequate atmosphere control. The conclusion is a critical study of the effect of the sintering atmosphere on the properties.

The increasing interest in this alloying element encouraged the study of new compositions of the prealloyed powder. Thus, Lewenhagen in [39], with the aim to make easier the sintering process, has studied a prealloyed powder with lower chromium content (Fe-1.5Cr-0.2Mo), obtaining results that can be compared with those obtained from conventional prealloyed powders obtained by diffusion bonding with copper, nickel and molybdenum. This new prealloyed grade was launched in [40].

All the alloying elements, and especially chromium, can be sintered by observing the following conditions: either the atmosphere is reductant enough, considering the particular balance oxide/metal in Ellingham's diagram, or the oxygen content is low enough to avoid any significant oxide formation. As reported in [7, 8] the oxide reduction not only can be produced at high temperature, or at low temperature in a vacuum, but at low temperatures it is also possible with an adequate control of the atmosphere.

The growth importance of chromium alloyed PM steels, is demonstrated by several recent papers [41-55]. Some of these works [41, 43, 54] combined the feasibility of improving the chromium effect by the addition of manganese or other elements that could activate the sintering. Karlson et al. [45] has studied particle size influence on the weight of the oxide layer. Maroli et al. [46] has evaluated the capability of the chromium as an alloying element for sinter-hardening over other elements used in pre-diffusion powders. In the same sense, and considering the bainitic microstructure that these steels have even for low cooling rates, Molinari et al. [56] demonstrate how this material possesses high levels for impact and wear behaviour. Yu [51] studied the kinetic and thermodynamic behaviour of the sintered steels obtained from the ferrous prealloyed powder with 3% Cr and 0.5% Mo. Lindberg et al. [42] and Campos et al. [53], among others, used warm compaction and high sintering temperature in order to improve all the potential capabilities of these materials by a combination of all the influencing factors. The susceptibility of these steels to thermochemical treatments has also been studied [55, 57]. And finally, some works have studied the possibility of using a smaller amount of chromium in order to improve the sintering cycle, even obtaining lower performance levels [39, 27].

In the World Congress on Powder Metallurgy recently held in Orlando, several works were related to chromium-molybdenum alloyed steels. Some of these works deal with their capability for thermo chemical treatments. Molinari et al. [58], has considered plasma carburising and plasma nitriding and their positive influence on properties such as fatigue or wear behaviour. This work improves on the knowledge about the reaction of these steels to the heat and thermochemical treatments, previously studied in [57]. The big improvements that can be obtained by using simultaneously high density techniques, such as warm compaction or high temperature sintering are shown in Ref. [59, 60]. In Figure 3, this effect can be seen in terms of impact strength and hardness. In this line of high temperature processing, where all the potential can be exploited in order to get the best performance, it is the work of Kano et al. [61], with some ideas for improving the quality of the furnaces at temperatures close to 1250ºC, by sintering in mesh belt furnaces of carbon-carbon composites that assure higher productivity, longer belt life, and less oxygen, hydrocarbon and humidity.

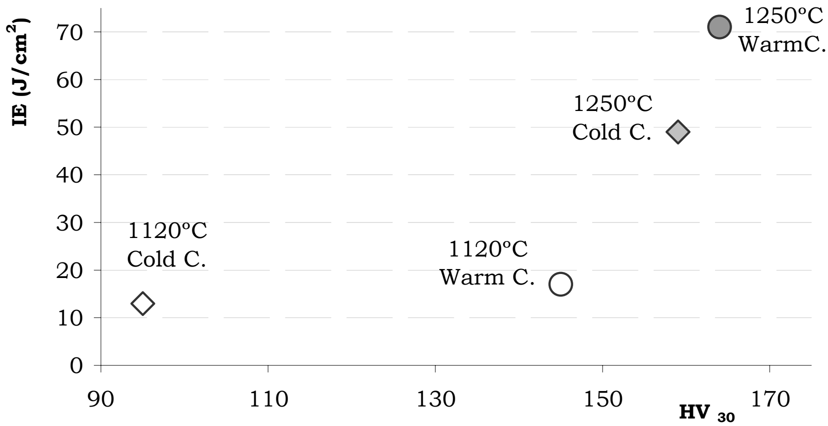

Fig.3. Impact energy absorbed values for Astaloy CrM+0.30% C_c vs. hardness [59].

It must not be forgotten that the most important challenge in these materials is to sinter properly without oxidation, hence the importance of controlling the sintering parameters [62, 63]. In this respect, the work of Danninger et al. [63] has studied the degassing curves coupled with differential dilatometry. Figure 4 shows one of these curves, in which a single test can correlate changes produced by the effect of the temperature, due to material-atmosphere interactions. A comparison of this curve (Fig.4) with those in earlier studies carried out with unalloyed steels, shows the different behaviour of prealloyed powder [64-67]. Three main peaks were determined in the above-mentioned works: a small one at around 350-400ºC, a second one a little below the transition temperature $\alpha \rightarrow \gamma$, and the third at about 1000ºC. In Ref. [65], the generated gases were analysed and it was found that the first peak was due to CO_2, which indicates a low-temperature reduction of the adsorbed oxygen. The second peak was mainly due to the CO, with a small amount of CO_2 from the surface reduction of the iron particles. The third, also of CO, may be that of the oxygen in the volume of the particle. In the steel obtained from prealloyed powder, the first peak appeared as expected at 370ºC, but the second is seen displaced a little up to 700-800ºC on account of the alphagenous elements, though it is smaller than expected. The third peak at 1020ºC is sharper than those of the unalloyed steels, which indicates a notable reduction of the oxide on the surface of the particles. In addition, a new peak appeared just before the isothermal zone at 1250ºC, an indication that the degassing is incomplete after sintering at 1120ºC, which explains the high oxygen content in sintering processes at that temperature. The considerable carbon losses and the low oxygen content found after sintering at 1250ºC, confirm the assumption that at high temperatures the real reducing agent is the carbon in the mixture.

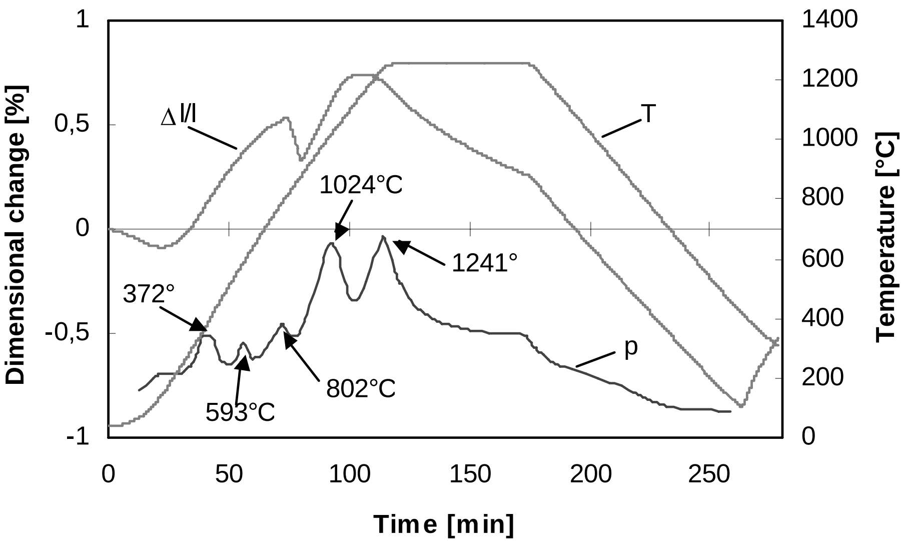

Fig.4. Degassing curve of Astaloy CrM + 0.5% C admixed [63].

CONCLUSIONS

To judge by the different work within recent works, it seems that chromium is an alloying element that will be used more and more by the PM industry of low alloyed steels, for higher robustness and reliability.

From the reviewed works it can be concluded:

- Chromium is an efficient alloying element for PM steels, taking into account hardenability and mechanical properties.
- Chromium alloyed steels can be sintered in industrial atmospheres, when the oxygen content in the atmosphere is controlled carefully.
- Chromium effect on performance of the steels could be improved by using high compacting pressures or/and high sintering temperatures, or by using special processes such as warm compaction.
- The effect of chromium can also be improved by the use of other alloying elements that can produce an activation of the sintering, such as transient liquid phase sintering.
- As expected, chromium is a good additive for steels that will be heat-treated or thermo chemically treated, as used in wrought steels.

It could be concluded with the prediction, previously mentioned, by Professor Šalak: “The future belongs to manganese, chromium and molybdenum as alloying elements” [12].

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