# Technical requirements of an "ideal" sinter-hardening furnace

# A. Bressan, A. Scipione

**T**he physical and metallurgical grounds on which sinter-hardening process is based are shortly surveyed. To achieve the required hardenability, by forced gas cooling, rather highly alloyed materials are required. These materials impose controlled processing conditions.

The parameters to be controlled are: effective lubricant removal, correct carbon distribution and average content, favourable pore shape, amount of diffusion of alloying elements, either added by mixing or by diffusion bonding, high and even cooling speed, final stress relieving.

The latter step is needed to remove any risk of brittleness attributable to a freshly formed martensitic structure. The splitting of a complete sintering cycle into unit stages enables us to analyze the technical requirements of the corresponding zones of any industrial furnace.

The equipment pattern depends on the type of controlled atmosphere. Gas-solid exchanges and equilibria allow to understand why some current technical solutions may be unapt to a precise control of carbon content immediately before the fast cooling on which the final microstructure depends. Available literature data are used to point out the importance of a narrow tolerance on carbon content that is required on properly processed sinter-hardened steels.

A typical scheme of an "ideal" sinter-hardening furnace is presented; pilot equipment, suitable even to research activities or small scale and pilot productions, is finally described.

Parole chiave: acciaio, metallurgia delle polveri, sinterizzazione

## INTRODUCTION

Sinter-hardening represents one of the most recent novelties in ferrous powder metallurgy. Shortly, it is an innovative process where the natural high hardenability of a ferrous alloy is exploited to get hard microconstituents immediately after cooling inside a suitable sintering furnace. Presumably, the first papers describing positive results of tests aimed at achieving hardened structures immediately after an improved sintering were published about 11 years ago (for instance, [1]).



Figure 1 – Influence of alloying additions on compressibility of iron powders. The reference density varies between 6.8 and 7.0  $g/cm^3$ .

Figura 1- Influenza delle aggiunte di lega sulla compressibilità delle Polveri di Ferro. La densità di riferimento varia tra 6.8 e 7.0 g/cm<sup>3</sup>.

Andrea Bressan, Angelo Scipione G. ELF Powder Metallurgy Equipment - Belt Furnaces, Treviso, Italy. Paper presented at the 7<sup>th</sup> European Conference EUROMAT 2001, Rimini, 10-14 June 2001, or-ganised by AIM



*Figure 2 – Influence of alloying additions on hardenability of steels.* 

Figura 2- Influenza delle aggiunte di lega sulla temprabilità degli acciai.

It should be clear that high hardenability is required. In addition, the alloy additions should be compatible with the most diffuse sintering conditions and atmospheres and should affect only in a minor extent the powder behavior under pressure, namely compressibility, to get high density directly by simple compaction, without being obliged to replace existing atmosphere generation facilities. The basics of the process have been described by W. B. James, [2], while the various influences of possible alloy addition have been analyzed by Julien and L'Espérance [3]. The plots they published, redrawn, are shown in figures 1 and 2. Unfortunately, no information is available on the effect of copper on hardenability. In a first approximation, we can admit that the Cu multiplying factor should not differ from that of Ni. The strong affinity toward oxygen, that is typical of Mn, Cr and Si, in comparison with the other alloying additions of figures 1 and 2, as it is well known, prevents the use of the first metals, un-

less H<sub>2</sub>/N<sub>2</sub> atmosphere, with low dewpoint, are used. According to this fast survey, the sintered steels suitable to sinterhardening should contain higher amounts of the same alloy elements, which are commonly used for PM steels to be heat treated after sintering, in separate equipment.

To avoid misunderstanding, it must be stated that indications of previous figures are valid for completely homogeneous alloys, i.e. for atomized powders. When powder mixes or diffusion-bonded powders are used, the influence of alloy additions on compressibility is usually trifling, while the hardenability improvement is not a constant value and depends on the intensity of sintering. To complete this paragraph, it is interesting to observe the positive balance between adverse and positive influences of molybdenum, which, furthermore, does not require low dewpoint atmospheres.

#### **POWDERS FOR SINTER-HARDENING**

The starting point to design the "ideal" sinter-hardening furnace is represented by existing standards and useful literature issued by reliable powder suppliers. Sinter-hardening materials have been included into MPIF Standard No. 35 [4] since some years.

The list is reported in table I. A list of powders suitable to sinter-hardening is presented in table II. In all grades, if no indication is given, Mo, Mn, and Cr are completely diffused already on atomization.

The suggested copper additions permit to reach the highest properties after sinter-hardening. Of course, since copper particles, on melting during sintering, originate pores, which replicate their shape, the Cu powder used for pre-paring the

Material	Chemical composition, weight %									
designation	Fe		С		Ni		Mo		Cu	
designation	min	max	min	max	min	max	min	max	min	max
FLN2-4408	93.15	97.75	0.6	0.9	1.00	3.00	0.65	0.95		
FLN4-4408	91.15	95.75	0.6	0.9	3.00	5.00	0.65	0.95		
FLN6-4408	89,15	93,75	0.6	0.9	5.00	7 00	0.65	0.95		
FLN-4608	91.00	93.41	0.6	0.9	3.6(a)	5 0(a)	0.39	1.10		
FLC-4608	91.00	96.41	0.6	0.9	1.60	2 00	0.39	1.10	10	3.0
FLC-4908	92.40	95.10	0.6	0.9			1.30	1.70	1.0	3.0
FLC-4408	90.15	96.75	0.6	0.9	1.00	3.00	0.65	0.95	1.0	3.0

composition of some hybrid low-alloy steels listed in MPIF Standard 35 as sinter hardening materials

Table I - Chemical

Tabella 1- Composizione chimica di alcuni acciai bassolegati "ibridi" elencati nella norma MPIF 35 come materiali da sinterotempra.

Other elements: Total by difference equals 2.0 wt% maximum, which may include other minor elements added for specific purposes. (a) At least 1 wt% of the nickel is admixed as elemental powder.

Commercial	Chemical composition, typical, wt %								
designation	Fe (a)	Ni(b)	Ni(c)	Mo	Cr	Mn	Cu(b)	Cu(d)	
Ancorsteel 737 SH	96.95		1.40	1.25		0.4		1.0	
Atomet 4601	97.45		1.80	0.55		0.20		1.0	
Atomet 4701	97.20		0.90	1.00	0.45	0.45		1.0	
Atomet 4801	95.30		4.00	0.50		0.20		1.0	
Distaloy DH-1	96.50			1.50			2.0		
Distaloy HP-1	92.50	4.0		1.50			2.0		
Domfer MP 55 HD	96.90		1,85	0.75		0.50		2.0	
Domfer MP 56 HD	96.55		1.85	1.10		0.50		1.5	
Kobe 46 F3H	97,5	1.5		1.0				2.0	
Mannesmann MSP 2	97,45		1.85	0.5		0.20		1.0	
Mannesmann MSP 4	95.25	4.0		0.55		0.20		1.0	

(a) before copper, graphite and lubricant addition; (b) added by diffusion-bonding; (c) added before atomization; (d) suggested addition.

Stage No.	Unit stage	Temperature range, °C	Typical time, min.
1	Lubricant removal, in the liquid state	150 ÷ 200	25 ÷ 30
2	Lubricant removal, by gaseous decomposition	300 ÷ 600	5 ÷10
3	Refining (reduction of surface oxides)	750 ÷ 1000	15 ÷ 25
4	Carbon diffusion	900 ÷ 1050	10 ÷ 20
5	Diffusion of alloy additions (mixes or diffusion-bonded powders)	1050 ÷ 1150	30 ÷ 35
6	Pore rounding, especially in case of fully prealloyed materials	1100 ÷ 1150	25 ÷ 35
7	Carbon pick-up from the atmosphere	> 1000	15 ÷ 25
8	Cooling without microstructure changes	1150 ÷ 950	15 ÷ 25
9	Carbon restoration	950 ÷ 800	25 ÷ 30
10	Fast cooling, to get martensite	800 ÷ 300	< 3
	Slow cooling, to try to complete the martensitic transformation	300 ÷ 50	45 ÷ 60

Stage No.	Additional unit stage	Temperature range, °C	Typical time, min.
-11	Slow cooling, to try to complete the martensitic transformation	300 ÷ 50	30 ÷ 45
12	Reheating, to prepare for stress relieving	50 ÷ 250	15 ÷ 25
- 13	Stress relieving	160 ÷ 250	40 ÷ 60
14	Slow cooling, to a low enough temperature to avoid oxidation	250 ÷ 50	5 ÷ 10

Table II – Typical chemical compositions of sinterhardening powders

Tabella 2- Composizioni chimiche tipiche per acciai da sinterotempra.

Table III – Basic unit stages of sinter-hardening, in endogas, in a "normal" belt conveyor furnace.

Tabella 3- Singoli stadi del procedimento di sinterotempra in un forno continuo convenzionale con atmosfera di Endogas.

Table IV – Additional stages of sinter-hardening, in endogas, in a "complete" belt conveyor furnace. Stages from 1 to 11 are the same of table III.

Tabella 4- Stadi addizionali del processo di sinterotempra in un forno continuo "completo" con atmosfera di endogas. Gli stadi da 1 a 11 sono uguali a quelli di tab. 3.

mix should be definitely finer than the iron base. As a matter of fact, the typical pore size affects mechanical behavior, especially fatigue strength.

## **IDEAL STAGES OF A CORRECT SINTER-HARDENING PROCESS**

The sinter-hardening process is an improved version of the correct sintering of carbon steels. The "unit" stages are listed in table III, based on a paper by Bocchini et alii, [5]. The presence of a car-bon restoration zone is necessary only if endogas is the protective atmosphere. Some addition of carbon-containing gas is required in case of  $H_2/N_2$  mixes. Since a stress-relieving step is required after martensite forming, or other heat treatments can be planned, the end portions of sinter-hardening dedicated furnaces could be modified according to processing steps of table IV.

For some specific application, the standard sinter-hardening process could be properly modified, to get austempering, usually described in iron metallurgy textbooks, such as [6]. In this case, a soaking timed above  $M_s$ , the starting temperature of martensite formation, (indicatively, between 300 and 400°C) should be added, before the final cooling.

To be complete, the carbon restoration zone could be utilized to increase the carbon content of surface zones of parts compacted from a low-graphite mix. In this way, sinter-hardened materials with controlled carbon gradients could be manufactured. A controlled carbon gradient may combine high surface hardness and strength with relatively high core toughness.

To get consistently optimum properties from sinter-hardened steels, some comments to previous tables may be useful. In particular:

- lubricant removal should be carried out without originating grain-border cementite or altering the right carbon content. The first problem may derive from zinc stearate addition, whereas the second one can be a consequence of uncontrolled oxidation promoted by RBO systems;
- the diffusion of alloy additions should reach an extent apt to generate a correct distribution of hard micro-constituents after fast cooling. In case of not-prealloyed powders it is necessary to remember that nickel diffusion is a rather slow process. The diffusivity of nickel into iron, at various temperatures, is plotted in figure 3, based on Smithells data [7];

The curve of figure 3 shows that a temperature increase of 25 °C, between 1100 and 1150 °C, enhances the Ni diffusion by about 50%. The inadequate nickel diffusion may be the reason of too high austenite fraction after fast cooling. At least partially, this austenite cannot be considered as "residual", because it survives any fast cooling. In other words, the too Ni-rich areas correspond to incoercible austenite on cooling. If nickel diffusion in sintering is improved, [8], it is possible to enhance, dramatically, the microstructure distribution and the apparent hardness and mechanical properties after fast cooling;

in case of completely prealloyed powders, the sintering conditions should originate a good pore rounding, to avoid the typical brittleness of a high strength material locally weakened by not rounded enough porosities. According to German [9]: "... porosity featuring sharp re-entrant corners, a possibility for marginally equilibrium-sintered powder particle boundaries, may be more accurately viewed as crack precursors. Pore geometry can be altered by modifications to the sintering cycle, such as a longer hold time or higher temperature, wherein the smoother pores improve strength, fatigue life and fracture resistance". This statement agrees with Esper and Sonsino view [10], who assert: "it should be noted that with increasing hardness of a material the notch sensitivity increases and con-



Figure 3 – Diffusivity of nickel into iron versus temperature, from Smithells, [7]. Plot prepared by G. F. Bocchini, private communication.

Figura 3- Diffusività del Nichel nel Ferro in funzione della temperatura.



Figure 4 – Influence of sintering conditions on the shape factor of pores. From [8].

Figura 4- Influenza delle condizioni di sinterizzazione sul fattore di forma dei pori.

sequently the fatigue strength is reduced by a notch". Esper and Sonsino consider the stress-raising effect due to external notches, but according to theory of elasticity, the ratio between typical pore radius and average distance between adjacent pores can be taken as a measure of internal notch effect due to pores. The latter remark un-doubtedly indicates the basic importance of a suitable sintering degree when sinter-hardening materials are processed. Just as an example, the influence of sintering condition on pore round-ness is plotted in figure 4;

• the carbon restoration zone is required to give back the carbon previously removed by the endogas atmosphere, at high temperature, from the surface zones. At equal processing conditions, the depth of carbon variations, in both steps, namely on depletion and on restoration, is a function of part porosity and surface "permeability" or "perviousness". As far as a precise carbon content we may assume that sinter-hardening steels are more demanding than customary high strength steels, because the microstructure distribution cannot be adjusted by a subsequent heat treatment. It may be interesting to observe that the interactions between atmosphere and solid where analyzed and discussed even more than half a century ago by Koebel [11]. Subsequently, a comprehensive review on sintering atmospheres was prepared by Durdaller [12], whereas Kaufmann [13] described clearly the changes to a standard fur-

2002

nace pattern needed for the correct sintering of carbon steels. More recently, the thermodynamics of sintering atmospheres has been surveyed by Mosca [14] and Beiss [15], who published updated versions of the classical diagrams wherein the carbon potential of ferrous materials are plotted versus dew point, or versus CO<sup>2</sup> percentage or, eventually, versus FEM of oxygen probe;

- the cooling speed to get martensite on cooling depends on alloy content and diffusion extent, in case of not-preal-loyed powders. According to Ferguson [16], "an optimal microstructure for a sinter-hardened material would show more than 90% martensite at the surface of the part with no less than 70% martensite in the core." To get consistently these features, the cooling speed should exceed at least 1 °C per second. This lower limit seems confirmed by Ferguson et alii [17]. The limited extent of experimental results authorizes to some allowance, so that it seems safer to design a sinter-hardening equipment with a cooling speed at least equal to 2.5 ÷ 3 °C/s;
- the temperature to be reached after fast cooling should be fixed in order to achieve at least 90% martensite. From the experience made on fully dense steels, it is known that any alloy addition lowers Ms. According to a formula proposed by Andrews [18], 1% Ni lowers Ms by 17,7 °C, while 1% Mo lowers Ms by 7.5 °C. (Manganese and carbon are even more effective, because 1% Mn lowers Ms by 30.3 °C and 0.1% C lowers Ms by 42.3 °C). Just as an example, applying the formula proposed by Andrews to a steel containing 0.65 % C, 1% Mo and 2% Ni we get Ms = 221 °C. Since Mf is usually about 200 °C lower than Ms, to get the desired microstructure the parts must be cooled at least to 50 °C before stress relieving;
- as far as stress relieving, the conditions for fully dense steels can be directly transferred to P/M ones. In case of stress relieving after quenching, the only difference may derive from possible ignition problems due to quenching oil, heated in air at more than 200 °C. This limitation does not exist in case of stress relieving after sinter-hardening and the temperature may be fixed only on the basis of the required properties. D'Armas et alii, [19], have shown that the choice of the most suitable stress relieving temperature changes if fatigue strength instead of static strength is the de-sign parameter. The importance of stress relieving has been pointed out by many research workers.

#### POSSIBLE PATTERN OF AN "IDEAL" SINTER-HARDENING FURNACE

A typical continuous production furnace for sintering of P/M compacts is normally composed of three different zones:

- · a delubrication zone for removal of compacting lubricants,
- a sintering zone, in which the parts are sintered,
- a slow cooling zone, where the parts are cooled to below their oxidizing temperature before being discharged from the protective atmosphere of the furnace.

Given the previous considerations regarding the influence of controlled atmospheres, in case of use of endogas the furnace should contain an additional zone namely the carbon restoration zone. This pattern apply to a furnace for traditional sintering processes, whereas, obviously, in case of sinterhardening cycle, the equipment must contain a rapid cooling zone where the cooling rate of the pieces is such that a martensitic structure is obtained. Usually the rapid cooling chamber operates on the principle of high convection, using the furnace atmosphere as convection medium.

The dimensions of the various zones are linked to the length of the sintering chamber. The latter, in turn, must ensure the pieces, for a given belt speed, a permanence of at least 20 minutes at the sintering temperature. As a rule of thumb, the length of the delubrication zone should be at least 70 % of the sintering zone, but 100 % is the most desirable length to achieve a complete elimination of any compacting lubricants. It is necessary to remember that the lubricant removal should be slow below 300 °C and as fast as possible between 400 and 600 °C. This need for different heating speeds, before reaching 600 °C, often neglected by equipment manufacturers, leads to the necessity of dividing the delubrication chamber into a minimum of three controlled heating zones.

The length of the carbon restoration zone could be evaluated by calculations, considering diffusion kinetics and physical properties of different gaseous substances. As a matter of fact, positive experiences have been made on length of carbon restoration zone at least equal to two/thirds the length of sintering zone. As already explained, this additional chamber is not required in case of atmospheres not containing carbon. As for the rapid cooling chamber, its length must be calculated considering belt speed, expected cooling rate and required temperature drop of the pieces. The rapid cooling de-vice is usually designed to achieve a cooling rate of at least 3°C/s in the temperature range from 850°C to 300°C. The presence of a rapid cooling section could lead to a reduction of the slow cooling zone, which is typically two to two and one half times the effective length of the sintering zone. However, it is preferable to keep the length of the slow cooling zone at its standard: with this configuration, the furnace can also be used for traditional sintering cycles. In this case the rapid cooling device is simply turned off.

Sinter-hardening is often referred to as a one step process. In reality, because either a partially or fully martensitic structure is obtained after sintering, tempering is usually required, to avoid brittleness problems or too high scattering of mechanical properties. With this in mind, a further devel-opment for a sinter-hardening furnace is represented by the presence of a stress-relieving zone. This thermostatic section, equipped with a number of heating elements, should be placed after the rapid cooling zone and after a sufficient temperature drop below Ms. Its length should be calculated in order to keep the pieces at about 200 °C for one hour. The addition of a stress relieving section implies the presence of a second rapid cooling device, to bring the temperature of the pieces from 200°C to less than 50°C at the end of the stress-relieving zone.

## **DESCRIPTION OF A PILOT EQUIPMENT**

The equipment illustrated in figure 5 is a pilot sinter-hardening furnace suitable to research activities or small scale productions. Its configuration is similar to that of a traditional belt conveyor sintering furnace, with the addition of a carbon restoration zone and a rapid cooling section.

On the same figure, also the temperature profile along the furnace tunnel is plotted.

The furnace has a 150 mm wide conveyor belt and a passage useful height of 60 mm; the conveyor belt speed is variable between 20 and 300 mm/min. The length of the sintering zone is 1000 mm and it is equal to those of the delubrication and the carbon restoration zones.

Keeping the length of the delubrication zone at 100 % of the sintering zone, like mentioned before, ensure a complete elimination of every compacting lubricant. The length of the carbon restoration zone is similarly kept at 100 % of the sintering zone to give optimum control over combined carbon.

The equipment has an electrical heating system composed of six heating element units, two for every chamber, independently controlled trough thermocouples and thermo-regulators. This configuration allows better control and flexi-

Figure 5 – Pilot equipment and ideal thermal profile.

Figura 5- Equipaggiamento pilota e profilo termico "ideale".



bility over the thermal profiles of the furnace. Maximum operating temperature of the equipment is 1150 °C, while the protective atmosphere is endogas and is provided by an external generator.

The muffle is a pressed-bent sheet metal building made of refractory steel and is connected to the rapid cooling section trough a water-cooled flange permitting a perfect gas-tight. The rapid cooling section is of stainless steel construction and it is designed to allow the impingement on the pieces by a stream of protective atmosphere. The protective atmosphere is circulated in the water-jacketed rapid cooling section using combined inlet and extract fans. After the impingement on the pieces, the protective atmosphere is refrigerated in an external heat exchanger.

After the rapid cooling section there is the slow cooling channel, also water-jacketed, still of stainless steel construction. The length of the slow cooling channel is 3000 mm.

The furnace is equipped with an oxygen probe, that can be fed by three different inlets, to control the carbon potential of the atmosphere inside any separately heated chamber.

Moreover, the equipment has been designed with a modular concept, to allow for further develop-ments. For this reason, the closing plate of the heating zone is flanged, to permit the possible modification of the rapid cooling section. Besides, the rapid cooling channel could be easily substituted by the thermostatic stress relieving section mentioned in the previous paragraph.

## ACKNOWLEDGMENTS

The authors thank Mr. G. F. Bocchini for the useful indications given to prepare this report.

#### REFERENCES

V. C. POTTER, W. B. JAMES, T. F. MURPHY: "Im-1. proved Dimensional Control and Elimina-tion of Heat Treatment for Automotive Parts", Advances in Powder Metallurgy, Vol. 3., L. F. Pease III and R. J. Sansoucy, Editors, Metal Powder Industries Federation, 1990, p 33-48

- W. B. JAMES: "What is Sinter-Hardening?", Hoega-2. naes Technical Data, Cinnaminson, N.J., USA, 1998
- 3. B. JULIEN and G. L'ESPERANCE: "Evaluating Hardenability and Compressibility for the Development of an Optimum Sinter Hardening Powder", 1996 World Conference on Powder Metallurgy and Particulate Materials, Metal Powder Industries Federation, Washington, D.C., June 26-21, 1996
- 4 METAL POWDERS INDUSTRIES FEDERATION: "Materials Standards for P/M STRUC-TURAL PARTS", 1997 Edition, MPIF, Princeton, New Jersey, USA, 1997
- 5. G. F. BOCCHINI, R. CESARI, M. R. PINASCO, E. STAGNO: "Sintering of Carbon Steels: Controlled Atmospheres, Equipment, Practical Results", PM International Conference, Pune, (India), February 1997
- D. T. LLEWELLYN & R. C. HUDD: "Steels. Metal-6. lurgy and Applications", Third Edition, Butterworth Heinemann, Oxford, UK, 1998
- E. A. BRANDES, G. A. BROOK (Editors): "Smithells 7. Metals Reference Book", Butterworth Heinemann, Oxford, UK, 1998
- G. F. BOCCHINI, M. G. IENCO, A. NAVAZIO, M. R. 8. RINASCO, G. SILVA, B. RIVOLTA, E. STAGNO: "Physical and Mechanical Properties, Microstructure and Fractography of a P/M Steel from Diffusion-Bonded Powder, Differently Sintered or Heat Treated", Advances in Pow-der Metallurgy & Particulate Materials -2000, 2000 PMPM Int. Conference, MPIF, New York, N.Y, June 2000
- R. M. GERMAN & R. A. QUENEY: "Fatigue and Frac-9. ture Control for Powder Metallurgy Components", in Powder Metal Technologies and Applications, Metals Handbook, Volume 7, Tenth Edition, ASM International, Materials Park, Ohio, 1998, p. 959
- 10. F. J. ESPER and C. M. SONSINO: "Fatigue Design for PM Components", European Powder Metallurgy association, Shrewsbury, England, 1994, p. 22
- 11. N. K. KOEBEL: "Sintering Atmospheres Used in Pow-

METALLURGIA DELLE POLVERI

der Metallurgy", Proc. 4th Annual Meeting of Metal Powder Association, USA, 1948

- C. DURDALLER: "Furnace Atmospheres", Technical Bulletin from Hoeganaes Corporation, Riverton, N. J., 1972
- S. M KAUFMAN: "Carbon Control During the Sintering of Iron", Powder Metallurgy Interna-tional, Vol. 5, No. 1, 1973
- 14. E. MOSCA: "Controlled Atmospheres in Powder Metallurgy", Sintering, Theory and Practice, Intensive short course, EPMA, Torino, Italy, October 1996
- P. BEISS: "Thermodynamics of Protective Atmosphere Control", in Advances in Powder Met-allurgy and Particulate Materials – 1996, Volume 3, Edited by T. M. Cadle and K. S. Narasim-han, 1996 World Congress on Powder Metallurgy and Particulate Materials, MPIF, Washington, D.C., June 1996

- H. A. FERGUSON: "Heat Treatment of Ferrous Powder Metallurgy Parts", in Powder Metal Technologies and Applications, Metals Handbook, Volume 7, Tenth Edition, ASM International, Materials Park, Ohio, 1998, p. 652
- 17. H. A. FERGUSON, G. L'ESPERANCE, E. DUCHES-SE, A. dE REGE : " Effect of Mass/Cross Sectional Thickness of Sinter Hardening of Two Prealloyed Steels of Different Hardenability", in Advances in Powder Metallurgy and Particulate Materials – 1997, MPIF, Princeton, N. J.
- K. W. ANDREWS, Journal of Iron and Steel Institute, 203, 721 (1965)
- H. D'ARMAS, LO. LLANES, J. PENAFIEL, J. A. BAS, M. ANGLADA : "Tempering effects on the tensile response and fatigue life behaviour of a sinter-hardened steel", Material Science and Engineering, A277 (2000), Elsevier Science, p. 291-296

# ABSIRA

## REQUISITI TECNICI DEL FORNO "IDEALE " PER SINTEROTEMPRA

La sinterotempra rappresenta una delle novità più recenti nel campo della metallurgia delle polveri per materiali a base ferro. Il termine sinterotempra indica un processo in cui l'alta temprabilità di una lega a base ferro viene sfruttata per ottenere dei microcostituenti di elevata durezza immediatamente dopo il raffreddamento in un forno continuo a nastro per sinterizzazione dotato di opportune caratteristiche. Ovviamente le aggiunte di lega devono garantire un'elevata temprabilità per dare luogo alla formazione di martensite nelle condizioni di raffreddamento che si verificano all'interno del forno. Inoltre, queste aggiunte di lega devono essere compatibili con le comuni condizioni di sinterizzazione e non devono compromettere in misura eccessiva la compressibilità delle polveri. In letteratura sono disponibili vari diagrammi che indicano gli effetti dei vari elementi di lega sulla temprabilità e sulla compressibilità delle polveri. L'uso di questi diagrammi, combinato con la co-noscenza della compatibilità delle varie aggiunte di lega con le atmosfere di protezione comunemente usate nei processi di sinterizzazione, permette di definire la composizione ideale per l'ottenimento di determinate caratteristiche meccaniche dopo sinterotempra.

In definitiva si può affermare che le polveri per sinterotempra dovrebbero contenere maggiori con-centrazioni delle stesse aggiunte di lega utilizzate negli acciai per metallurgia delle polveri che ven-gono temprati dopo sinterizzazione in un equipaggiamento separato.

Fondamentalmente il procedimento di sinterotempra è una versione migliorata del processo di sinterizzazione per acciai al carbonio.

La differenza principale consiste nella presenza di una zona del forno dove il materiale viene raf-freddato rapidamente nell'intervallo di temperatura dagli 800 ai 300 °C in modo da ottenere una microstruttura prevalentemente martensitica. La velocità di raffreddamento necessaria per ottenere la formazione di martensite dipende dalla concentrazione degli elementi di lega e, nel caso di polveri non prelegate, dalla diffusione degli stessi nel ferro. Per quanto riguarda quest'ultimo aspetto, è necessario prestare particolare attenzione nel caso di aggiunte di Nichel la cui diffusione nel Ferro è piuttosto lenta.

I dati reperibili in letteratura suggeriscono che per ottenere una quantità di martensite superiore al 90% in superficie e al 70% al cuore la velocità di raffreddamento nell'intervallo di temperatura sopraccitato dovrebbe essere pari ad almeno 1°C/s. Tuttavia, la scarsezza di dati sperimentali sull'argomento suggerisce di adottare come dato di progetto una velocità di raffreddamento di almeno 2.5-3 °C/s.

Per quanto riguarda la temperatura da raggiungere dopo lo stadio di raffreddamento rapido essa può essere calcolata con una formula reperibile in letteratura. Questa formula tiene conto del fatto che ogni aggiunta di lega tende ad abbassare la temperatura Ms, cioè la temperatura alla quale ha inizio la trasformazione martensitica. Considerando le più comuni aggiunte di lega (Ni, Mo, Mn etc.) la temperatura di inizio trasformazione si aggira attorno ai 220 °C perciò, considerando che la tempe-ratura di fine trasformazione è inferiore di circa 200°C sembra opportuno protrarre il raffreddamento fino a che i pezzi non raggiungono una temperatura di circa 50 °C prima di un eventuale stress relieving.

Il trattamento di stress relieving si rende spesso necessario per ridurre le tensioni interne dovute alla formazione di martensite che potrebbero dare luogo a problemi di fragilità del materiale.

Normalmente un forno continuo per la sinterizzazione di componenti di acciaio è costituito da tre zone: una zona di delubrificazione per l'eliminazione del lubrificante usato in fase di pressatura, una zona di sinterizzazione e una zona di raffreddamento lento in cui i pezzi vengono raffreddati al di sotto della temperatura di ossidazione prima di essere estratti dal forno.

Nel caso di atmosfera protettiva costituita da Endogas si rende necessaria la presenza di un'ulteriore zona denominata zona di ripristino del carbonio.

Questo perché, ad alta temperatura, l'atmosfera di Endogas tende a decarburare la superficie di pez-zi. L'entità della decarburazione, a parità di condizioni di processo, dipende dalla porosità e dalla "permeabilità" della superficie dei pezzi da sinterizzare. Nel caso degli acciai da sinterotempra, il controllo preciso del contenuto di carbonio è imperativo in quanto questi materiali non possono es-sere sottoposti ad ulteriori trattamenti termici di "aggiustamento" della microstruttura dopo sintero-tempra.

Per questo motivo i pezzi, dopo sinterizzazione, attraversano una zona in cui le condizioni termodi-namiche sono tali da consentire la ricarburazione superficiale da parte della stessa atmosfera di En-dogas.

Un forno per sinterotempra necessita ovviamente di un'ulteriore camera per il raffreddamento rapido dei pezzi dopo

#### sinterizzazione.

Per quanto riguarda le dimensioni delle varie zone del forno "ideale" per sinterotempra conviene fare riferimento alla lunghezza della zona di sinterizzazione che viene fissata in modo tale da assicurare, per una data velocità del nastro, una permanenza di almeno 20 minuti alla temperatura massima. Una volta fissata la lunghezza della camera di sinterizzazione si possono stabilire le dimensioni della camera di delubrificazione che dovrebbe avere una lunghezza pari ad almeno il 70% della camera di sinterizzazione. Tuttavia, per garantire la completa eliminazione dei lubrificanti, è opportuno mantenere una lunghezza della zona di delubrificazione pari al 100% della camera di sinterizzazione.

La lunghezza della camera di ripristino del carbonio può essere calcolata considerando le cinetiche di diffusione e le proprietà fisiche delle varie sostanze gassose. In pratica, l'esperienza suggerisce che la lunghezza della zona di ripristino del carbonio dovrebbe essere pari ad almeno 2/3 della lunghezza della camera di sinterizzazione.

Per il calcolo della lunghezza della zona di raffreddamento rapido le variabili da considerare sono: velocità del nastro, velocità di raffreddamento desiderata e salto di temperatura dei pezzi. Normalmente la camera di raffreddamento rapido sfrutta il principio della convezione forzata usando la stessa atmosfera protettiva come mezzo di raffreddamento. In genere, la camera di raffreddamento rapido viene progettata in modo da garantire una velocità di raffreddamento di 3°C/s nell'intervallo di temperatura dagli 850 ai 300°C.

La figura 5 rappresenta un forno per sinterotempra pilota suscettibile di utilizzo per attività di ricerca o per produzione di particolari di ridotte dimensioni. La sua configurazione e le dimensioni delle varie zone rispecchiano le considerazioni riguardanti il forno "ideale" per sinterotempra fatte in precedenza.