INVESTIGATION ON THE INFLUENCE OF SINTERING PROCESS PARAMETERS ON IMPACT RESISTANCE OF PM 316L STAINLESS STEELS

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Abstract

Sintered stainless steels are now common and well known engineering materials in the mechanical industry. However, their properties are limited by the presence of a certain amount of porosity, which affects their mechanical properties. A consistent reduction of porosity can be achieved by means of higher compacting pressures, as well as sintering time and temperature, or by specific infiltration processes. An innovative and alternative route is represented by the reactive sintering process. This paper presents the results of investigations on the impact properties of different PM stainless steels compositions, studied in order to obtain reactive sintering conditions; different processing parameters were used. The powders were sintered at two different pressures and three different atmospheres and temperatures: in pure hydrogen, in a vacuum furnace and in a pusher furnace with a hydrogen and nitrogen atmosphere. With respect to the AISI 316L reference samples, good increases of density and of impact properties were obtained.

Keywords: stainless steel, reactive sintering, corrosion resistance, toughness.

INTRODUCTION

PM stainless steel parts are typically used in applications where their enhanced properties justify their higher cost, compared to low-alloy steel or iron: in fact, stainless steels are characterized by high corrosion resistance, oxidation resistance, good appearance, good mechanical properties (ductility and impact strength) and wear resistance. Among the different stainless steel grades, one of the most widely used is AISI 316L, because it is an excellent combination of corrosion resistance and good machinability. Notwithstanding this, the properties of sintered stainless steels are inferior to wrought steels, because stainless steel powders are generally very difficult to compress and sinter without obtaining interconnecting porosity, since little further densification occurs during solid state sintering. These limitations start from the actual technological processes: nowadays stainless steel sintering is performed in mesh-belt furnace (temperature < 1150°C) or in a pusher-type (temperature < 1275°C).

The low density, and so the high level of interconnected porosity, is responsible for a lower corrosion resistance respect to the not sintered stainless steels: the interconnected porosity causes electrolytic stagnation, but it hampers the oxygen flux, which is necessary to re-establish passivated layer of chromium oxide. Density is also of prime importance in respect to the mechanical properties of sintered parts, because tensile

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and flexural strength increase in approximate linear proportion, elongation and resilience exponentially, with sintered density.

In order to overcome these problems, enhancing corrosion resistance and mechanical properties different methodologies can be employed, mainly by modifying the traditional compositions [1-4] or by the use of infiltration techniques [5, 6].

Even if great attention is placed on innovative alloys, many times the dimensional control problems or insufficient cost/benefit ratios hinder the industrial application of sintered stainless steels. The industrial production of stainless steel components is always directed to the traditional compositions and there is a considerable interest in research aimed at the improvement of the properties of conventional and unmodified AISI grades.

A wide research programme was undertaken to review critical areas related to the effect of compacting pressure, of sintering time and temperature, as well as the application of infiltration techniques on the properties of stainless steel parts [7-9].

A previous work [10] was dedicated to the study of the dynamic properties of AISI 316 L (EN 10088: type 1.4404) stainless steel in the sintered state or after infiltration with different alloys. The interesting results obtained encouraged further work and this paper is mainly dedicated to the dynamic properties of PM stainless steel produced using reactive sintering techniques [11-14] in order to increase the density and to improve their properties.

EXPERIMENTAL PROCEDURE

A fully alloyed AISI 316 L austenitic stainless steel powder was produced by water atomisation, its chemical composition (wt %) being: Cr 16.78, Ni 13.48, Mo 2.2, Si 0.77, Mn 0.11, C 0.024, P 0.015, S 0.01, Fe bal., O 1900 ppm, N 520 ppm. To perform a self-propagating high temperature-synthesis, SHS, some batches were obtained by adding Ni, Fe, Ti, Cu and Al powders so as to obtain seven different compositions and were tested (Tab.1). Powders, added with 0.75 wt% of Acrawax as lubricant, were mixed for 30 minutes in a conventional mixer. After they were pressed uniaxially with pressures of 500 and 700 MPa in order to produce specimens for an un-notched impact test. Before the sintering cycle, the samples were dewaxed in a pit furnace for 60 minutes at 600°C in N_2 atmosphere.

Tab1. Composition (wt %) of the powders added to pure AISI 316L.

A	B	C	D
19.75% Ni	18 % Ni	18 % Ni	18 % Ni
0.745 % Fe	2% Fe	0.8% Fe	0.1 % Al
0.022% Al	0.2% Ti	0.2% Ti	0.1 % Ti
bal. AISI 316L	bal. AISI 316L	bal. AISI 316L	bal. AISI 316L
E	F	G	
18 % Ni	18 % Ni	18 % Ni	
0.1% Cu	0.8% Fe	0.6% Mn	
0.1% Ti	0.3% Cu	bal. AISI 316L	
bal. AISI 316 L	0.1% Al		
	bal. AISI 316L		

The sintering procedure was performed using different conditions and atmospheres as indicated in Tab.2.

Tab.2. Sintering conditions and atmospheres.

Type	T [°C]	time [h]	Atmosphere
H	1150	1	Pure hydrogen; laboratory tubular furnace
D	1275	0.5	50% hydrogen – 50% nitrogen; pusher furnace
V	1250	1	vacuum furnace with nitrogen backfilling

Measurements of the sintered density were carried out by gravimetric methods; HRB was also measured. Morphology and microstructure were observed by both optical and scanning electron microscopy, the chemical composition was analysed using a EDS microprobe. The porosity of the samples was measured by an image analysis technique.

The impact properties on the un-notched specimens (according the UNI EN 25754-94 standards) were tested by an instrumented impact machine with the available energy of 300 J. The corresponding impact velocity is 5.5 m/s. Tests were carried out at room temperature (25°C).

EXPERIMENTAL RESULTS

The density of the samples was function of the composition and of the process parameters, mainly compacting pressure and reached values close to 7.45 Mg/m^3, as well as the hardness being in the range 34-62 HRB.

In Table 3 the values of the total impact energy and in Table 4 a-b the medium dimensions of porosity (μ^2) of the SHS samples are indicated. In Fig.1 it is possible to observe a comparison between porosity and impact energy for samples sintered in a type D atmosphere. It's interesting to note the absence of a correlation between these two parameters; this fact has still to be understood. In Table 3, it is possible to note the positive influence of the highest compacting pressure on the impact energies, other specific influences are coming from the chemical composition and from the microstructure characteristics of the samples; a correlation between compacting pressure and porosity is again not so evident.

Tab3. Total impact energy [J] of the reactive sintered samples, at 500 and 700 MPa.

	Serie 500 H	Serie 500 D	Serie 500 V	Serie 700 H	Serie 700 D	Serie 700 V
	Impact strength (J)	Impact strength (J)	Impact strength (J)	Impact strength (J)	Impact strength (J)	Impact strength (J)
A	28.5	64.5	43	64	113	93.0
B	54.0	83.5	79	59	110	106.0
C	35.0	79.0	79	59	100	94.0
D	35.5	81.0	26	59	112	54.5
E	39.5	91.5	57	62	125	91.5
F	52.0	94.5	49	72	118	78.5
G	38.0	85.0	45	54	116	119.0

In Figure 2, an example of load-time and energy-time curves for impact specimens of the AISI 316L samples sintered in hydrogen at 1150°C is shown; for all other samples the curves showed the same trend, but with higher load/energy values. In the plotted curves, it is possible to distinguish the values of the total impact energy (Et), of the energy for crack nucleation Ei (the absorbed energy up to the maximum load) and of the propagation energy

(Ep). It's interesting to observe that, for all the tested materials, the greatest total absorbed energy is given by the energy for crack nucleation, with a low contribution of energy for crack propagation. In fact, the ratio between the propagation energy (Ep) and the total impact energy (Et) for the different samples is ranging between 11 and 30%, the highest values being reached only in some exceptional cases. In Figure 3, the curves show the values of the three types of energies, Et, Ei and Ep for the samples pressed at 500 and 700 MPa and sintered in a hydrogen/nitrogen atmosphere at 1275°C. These trends shows high variations of the total impact energy and of the energy for crack nucleation, while the propagation energy shows minimum changes. Moreover, it is evident that the compacting pressure strongly influences the nucleation energy, while the propagation energy is quite insensible to this as well as to other sintering parameters.
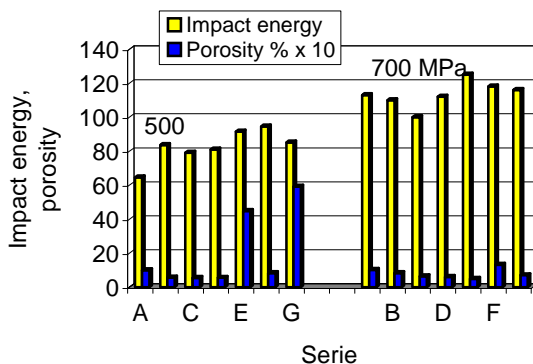

Fig.1. Comparison between porosity and impact energy for samples sintered in type D atmosphere.
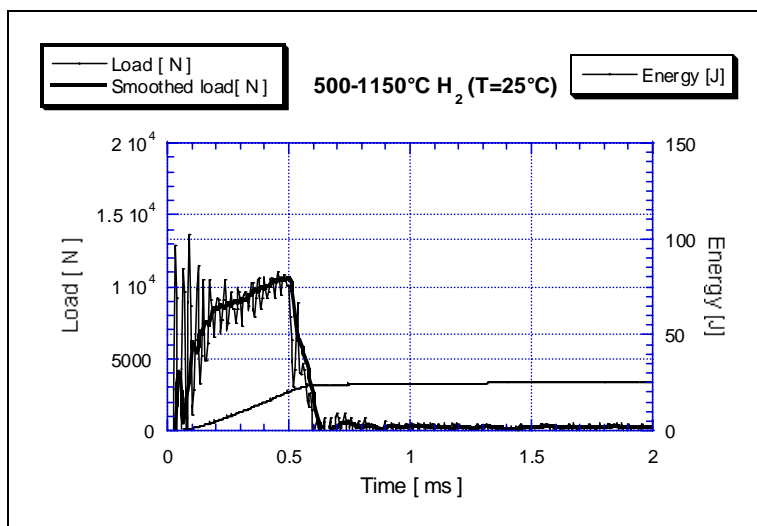

Fig.2. Load-time and energy-time curves for impact specimens of the AISI 316L.
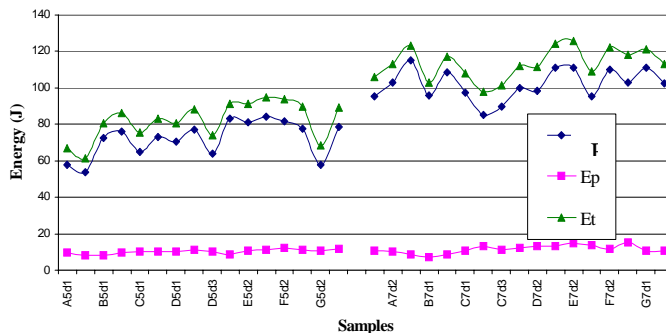
Fig.3. Trends of the energy values for samples sintered in hydrogen/nitrogen atmosphere.

In Figure 4 the microstructure features of samples showing the best properties, type F, are shown for the three different sintering conditions. On all the samples, the presence of intermetallic phases caused by the elements added to the AISI 316L powder can be observed. Moreover, when sintering in pure hydrogen, the presence of reduced quantities of small precipitates (mainly distributed at the grain boundaries) are also observable, as demonstrated by EDS analyses. These probably are due to the presence of residual of lubricant not completely removed during the debonding step. When the atmosphere contains nitrogen, type D or V (due to nitrogen backfilling), a consistent precipitation of complex precipitates, (probably nitrides, but the microprobe couldn't detect light elements like nitrogen), at the grain boundaries can be observed, especially in the hydrogen/nitrogen sintered samples (Fig.5).
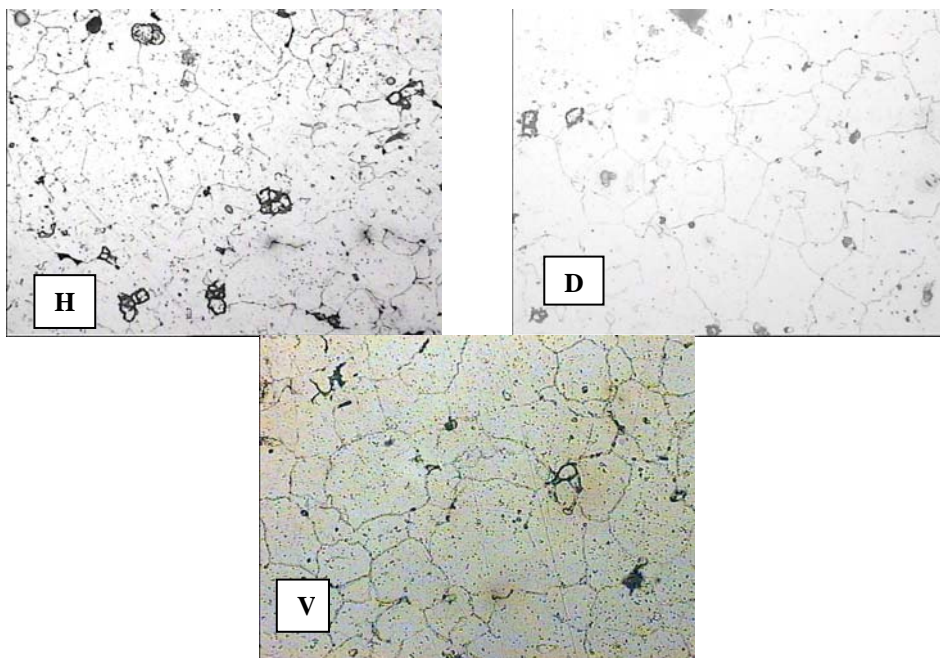

Fig.4. Microstructure characteristics of the samples type F, pressed at 700 MPa and sintered in the three different conditions: H, D and V.
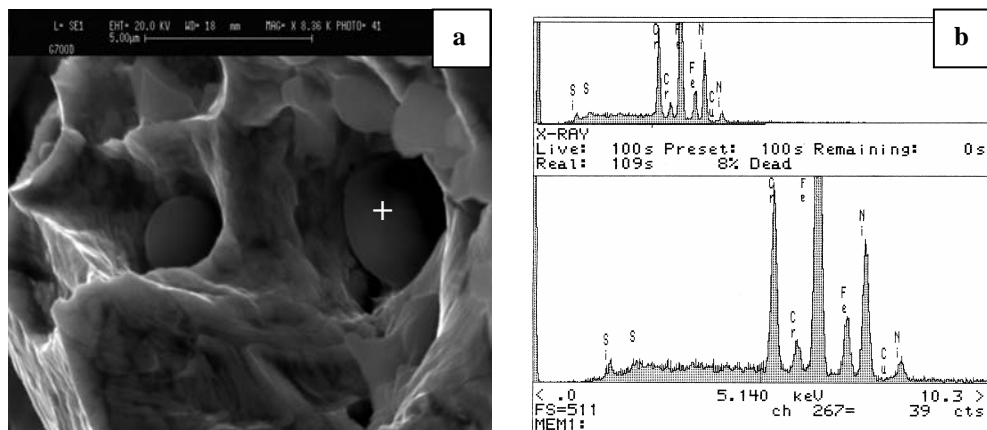
Fig.5. D sintered samples, SEM micrograph: precipitate (a) and
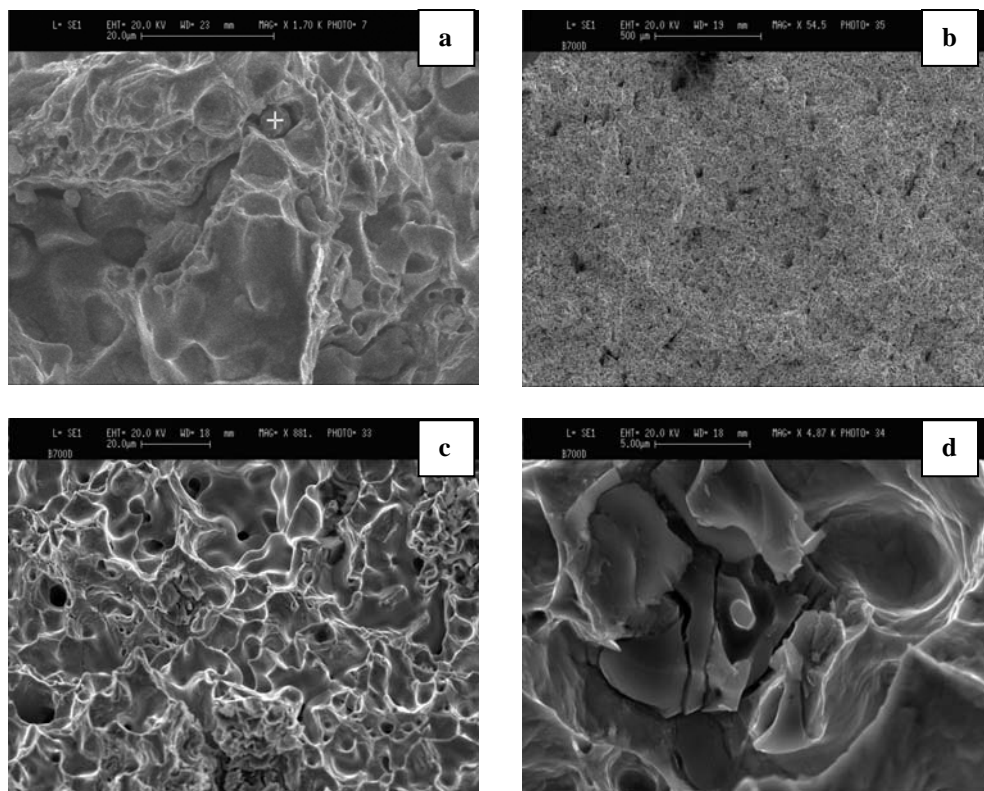EDS spectrum (b)."+" indicates the zones analysed by EDS microprobe.

Fig.6. Aspects of the fracture surface of samples type G (a) and B (b, c, d),
pressed at 700 MPa and D sintered.

In Figure 6 (a, b, c, d), some aspects of the fracture surfaces of samples type B and G pressed at 700 MPa and sintered in hydrogen/nitrogen atmosphere are shown. The fracture morphology is mainly of the ductile type (Fig.6c), the presence of porosity is quite evident (Fig.6b, c) and it is interesting to note a precipitated particle interested by the fracture process (Fig.6d). The fracture morphology strongly depends upon the sample composition and upon the sintering conditions, very ductile as well as brittle features being observed. Aside the benefits deriving from the increase of the compacting pressure, it was observed that the presence of hard precipitates in the matrix (Fig.6a) is helpful to deviate the cracks and to increase the absorbed energy. The presence of hard precipitates at the grain boundaries (partially due to the sintering atmosphere) is also beneficial, because it constitutes an obstacle to the slip at the grain boundaries with a consequent increase of the absorbed nucleation energy.

CONCLUSIONS

The work was dedicated to the study of the dynamic properties of PM AISI 316 L stainless steels produced with a reactive sintering process; the influence of the powder composition and of the sintering process parameters was studied.

The investigation allowed the following conclusions to be drawn:

1. The sintering pressure, as well as the sintering atmosphere and powder composition were all influencing parameters on the impact properties of the studied materials.
2. A too low of a temperature, even if for longer time (1 h) it is not sufficient to guarantee good diffusion of the added elements.
3. The sintering atmospheres and powder compositions which allow the formation of small, hard precipitates in the alloy (es: nitrides) are preferred, as they give better impact properties.
4. The highest impact energy values were obtained with:
   - the highest compacting pressure;
   - the F type composition;
   - the hydrogen/nitrogen sintering atmosphere at 1275°C for 30 minutes.

The conditions as chosen gave also the shortest processing time, and guaranteed the absence of oxides which could compromise the surface aesthetical characteristics of the parts.

REFERENCES

[1] Wood, J., et al. In: The 4th European Conference On Advanced Materials And Processes, 1995 Padua/Venice, Symposium G. Milano : Aim&Fems, 1995, P. 133
[2] Velasco, FJ., et al. In: Pm94' - Powder Metallurgy World Congress, Paris, 1994, Vol. 3. Les Editions De Physique Les Ulis, 1994, P. 2060
[3] Tandon, R., German, R.: The International Journal Of Powder Metallurgy, Vol. 34, 1998, P. 40
[4] Rosso, M., et al. In: Advances In Powder Metallurgy & Particulate Materials - 1996 World Congress, Washington, 1996. Princeton : 1996. P. 17
[5] Goetzel, CG. In: Metals Handbook, Vol. 7. 9th Ed. Metals Park : Asm Int., 1984, P. 551
[6] Rosso, M., et al. In: Proc. Of The 1998 Powder Metallurgy World Congress & Exhibition, Granada, 1998, Vol. 3. Shrewsbury : EPMA, 1998, P. 435
[7] Rosso, M., et al. In: Proc. Of Pm97. Advances In Structural Pm Component Production, Munich, 1997. Shrewsbury : EPMA, 1997, P. 280
[8] Rosso, M., La Verde, F.: Metall. Ital., Vol. 92, 2000, No. 10, P. 21

[9]   Rosso, M., et al.: Materials Engineering, Vol. 6, 1999, P. 11
[10] Rosso, M., Garagnani, GL. In: Proceedings Of The 3rd European Congress Stainless Steel '99 Science And Market, Chia Laguna Sardinia, 1999, P. 23
[11] Rosso, M., La Verde, F.: Metall. Ital., Vol. 92, 2000, No. 10, P. 21
[12] Rosso, M. In: Proc. Of ROPM 2000, 2nd Int. Conf. Powder Metallurgy Science And Technology, Cluj-Napoca, 2000, Vol. 1, P. 39
[13] Degnan, CC., Wood, JV., Actis Grande, M. In: Proc. Of ROPM 2000, 2nd Int. Conf. Powder Metallurgy Science And Technology, Cluj-Napoca, 2000, Vol. 1, P. 49
[14] Degnan, CC., Jackson, P., Weston, DP., Wood, Jv.: Materials Science And Technology, Vol. 17, 2001, P. 1624[1]

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