INFLUENCE OF THE INFILTRATION PROCESS ON PROPERTIES OF SINTERED STEELS

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Abstract: The properties of sintered steels can be improved by reducing the porosity, by means of higher compacting pressures, as well as sintering time and temperature. Infiltration process with a metal alloy can represent an alternative to high pressure compacting and to high temperature sintering, in fact the infiltration process allows large benefits with respect to density, hardness and, consequently, mechanical properties, as well as corrosion resistance. This paper presents the results of investigations on the properties and on the fracture behaviour of PM stainless steels, sintered under different processing parameters or sintered and infiltrated with bronze, Cu-10%Sn. Moreover, a second part of the paper deals with the comparison between addition of Cu-10%Sn to iron based powders, the aim to check the effects of the liquid phase sintering and the possible similarity with contact infiltration.

Introduction

The demand of alloyed sintered steels is continuously increasing and PM materials with higher mechanical and corrosion resistant properties are required. Sintered steels are generally less corrosion resistant than their wrought counterparts. There is a constant search for the optimisation of powders properties and of industrial processes in order to achieve higher mechanical properties and corrosion resistance.

The possible elimination of the porosity in the sintered materials can be obtained by infiltration techniques. Infiltration is the process of filling pores and reducing the porosity of a green or sintered compact with a liquid metal or alloy having a lower melting point and penetrating the pore system by means of surface or capillary forces. This process is similar to liquid phase sintering and the same principles are involved. During infiltration, the liquid metal or alloy wets or spreads over the surface of the porous solid it contacts. The reduction of total surface free energy of the system is a prerequisite during infiltration and determines whether a particular solid-liquid phase system is suitable for this widely used technique [1], that can be competitive against the multiple pressing and sintering operations [2, 3].

Porous iron or steels are commonly infiltrated with a variety of metals and alloys, mainly copper base. In fact, many commercial powders for infiltration contain metals like iron and manganese, to facilitate better wetting between iron skeleton and copper. In particular, iron is helpful to saturate the solubility of this metal in copper, avoiding erosion phenomena, while manganese favours oxide reduction and forms a porous skeleton which serves as a reservoir for molten alloy. This skeleton constitutes a non-adherent residue, easy to eliminate at the end of the process. In that possible elimination of porosity and good dimensional control are the main advantages of infiltration, any erosion phenomena must be
avoided. Infiltrated parts have higher hardness and strength, has well as improved corrosion resistance [4-11].

Following the experience acquired to study the sintering behaviour of different alloyed steels and to review the critical areas related to the effect of process parameters on the properties of stainless steel parts, as well as the application of infiltration techniques to stainless steels, in this paper the dynamic properties of as sintered or of sintered and infiltrated stainless steel samples are investigated and compared. A second part of the work has been dedicated to the study of the addition of bronze powder directly to the iron based powders, to evaluate the effects of the caused liquid phase sintering on the properties of sintered samples and the possible analogies with the contact infiltration.

Experimental procedure

Series of tensile test samples and un-notched impact tests specimens, 10x10x55 mm$^3$, were produced by compacting at 500 [MPa] the powders of a fully alloyed AISI 316 L austenitic stainless steel, mixed with lubricant, 0.75 wt%, type Acrawax. 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.

The samples, after a de-binding stage at 600 °C with N$_2$ atmosphere, were sintered or sintered and infiltrated at 1150 °C. The infiltration process with the single pass or step, that is the contact type with green samples and copper alloy compacts in contact and placed into the sintering furnace. The infiltrant alloy was a bronze: copper – 10 wt % tin. To demonstrate the effect of the compacting pressure and of the sintering temperature a set of samples was compacted at 700 [MPa] and, after the debinding step, was sintered at 1250 °C, all the samples were sintered for 30 minutes in dry hydrogen atmosphere. On the samples Rockwell B hardness, tensile properties and absorbed impact energy were measured. Microstructures were characterised by means of light microscopy, while the fracture morphologies were analysed by SEM.

The powders used for the second part of the work were iron based and prealloyed with 1.5 wt% Mo (code ST0) and with 1.5 wt% Mo and 2 wt% Ni (code ST2) and were mixed with bronze powder, copper – 10 wt % tin, in different amounts: 1, 2 and 3 wt%. The powders were compacted at 650 [MPa] to obtain un-notched bars for bending tests, then the compacts were sintered in vacuum furnace at 1120 and 1150 °C for 30 minutes. After sintering the bending strength has been measured, then the morphology of the fracture has been observed and optical microscopy has been performed on the etched transverse sections of the samples.

Results and discussion

Concerning the work done on the stainless steels, the density and the mechanical properties of as sintered and of infiltrated samples are reported in table 1 as a function of the compacting pressure, as well as of the sintering temperature. The highest values (more than 7 Mg/m$^3$) were reached when compacting at 700 MPa in the case of the as sintered samples, while the infiltration process allows density close to the theoretical value.

The apparent hardness, Rockwell B, of the as sintered samples depends on the density values, the lowest hardness properties are shown by the as sintered samples compacted at 500 MPa, while the infiltration process allows very interesting hardness values, higher than those obtained on the specimens compacted at 700 MPa and sintered at 1250 °C.
The yield strength and the ultimate tensile strength are favourably influenced by the infiltration process. The elongation properties are also positively influenced by the infiltration process, as well as by the highest sintering temperature.

The impact properties were evaluated by means of a Charpy impact machine on 6 bar specimens each series of samples, measuring the energy absorbed by the samples during the impact. In table 2 the average values obtained taking into account for the 5 best values are listed. The infiltration process allows a strong increase of the toughness properties, which became comparable with those of the samples sintered at 1250 °C.

Table 1: Density (ρ), Apparent Rockwell Hardness B (HRB), Yield Strength (Y.S.), Ultimate Tensile Strength (U.T.S.), Percentage of Elongation and Un-notched Impact Energy (U.I.E.) of the as sintered and infiltrated stainless steel samples.

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<tr>
<td>500-1150°</td>
<td>6.59</td>
<td>34</td>
<td>200</td>
<td>270</td>
<td>8</td>
<td>24</td>
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<tr>
<td>500-1150° +</td>
<td>7.46</td>
<td>64</td>
<td>290</td>
<td>540</td>
<td>30</td>
<td>100</td>
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<td>Infiltration</td>
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<tr>
<td>700-1250°</td>
<td>7.06</td>
<td>48</td>
<td>181</td>
<td>391</td>
<td>23.08</td>
<td>118</td>
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The un-notched specimens (according the UNI EN 25754-94 standards) were also tested by an instrumented impact machine with the available energy of 300 J.

In Fig. 1 are shown the load-time and energy-time curves for two specimens (sintered and sintered + infiltrated). The curves, typical of a material with ductile behaviour, are partially smoothed and the obtained results clearly demonstrate the higher maximum load and the higher plastic deformation, after yielding, up to the maximum load for infiltrated materials compared to the as sintered specimens.

Figure 1: Load-time and energy-time curves for impact specimens at 25°C and 220°C.

Interesting considerations comes by analysing the different contributions to the absorbed energy, that is the values of total impact energies (Et), the energy for crack nucleation Ei (the absorbed energy up to the maximum load) and the propagation energy (Ep). For all materials it is evident that the greatest total absorbed energy is given by the energy for crack nucleation, with a low contribution of energy for crack propagation. This result is
strictly related to the presence of residual porosity which favours the propagation of fracture. The ratio between nucleation and propagation components is lower in the case of as sintered specimens, in other words, filling the porosity with the infiltrant alloy clearly favours the increase of the nucleation energy.

The SEM analysis performed on the fracture surface of impact specimens highlights the morphology of some porosity and of fracture surface. Brittle and ductile fracture coexist in different amounts and secondary cracks between grains can be observed. The samples compacted at 500 MPa and sintered 30 minutes at 1150 °C show, in the as sintered state, higher porosity degree and reduced sintering grade, while the infiltrated specimens show a microstructure typical of a liquid phase sintered system. In fact, the grains of steel are surrounded by the infiltrant alloy and this justifies the highest ductility, being evident the large deformation degree of this phase due to the fracture process. Some intergranular cleavage facets are evident because of the strengthening effect experienced by steel matrix following the contact with the infiltrant alloy. However the adhesion between the steel matrix and the infiltrant alloy appears very good and continuous.

Concerning the second part of the work, the hardness properties of the samples based on ST0 composition with the different addition of bronze powders are summarized in the histogram of figure 2 for the two different sintering temperatures. The progressive addition of bronze powder to the iron based compositions favours the increase of hardness, owing to the reinforcement effects caused by liquid phase sintering. The ST2 based samples show the same effects, even if with lower values, probably the presence of Ni in the composition is not so favourable.

Fig. 2: The influence of sintering point and addition of bronze, on Brinell hardness, for ST0 (1), ST0 + 1% CuSn10 (2), ST0 + 2% CuSn10 (3), ST0 + 3% CuSn10 (4) compacts.

The impact resistance of the base sample and of those added with 3 wt% of bronze is summarized in the histogram of figure 3. The addition of 3 % wt of bronze powder to the iron based composition has a negative influence on the impact strength, in particular on the Ni alloyed samples (ST2 based composition). In general the results are more satisfying at the lowest sintering temperature, probably different factors contribute to negative effects, especially at the highest sintering temperature. In fact, owing to the presence of relatively low
melting point powders (bronze) on the base compositions, it could be possible that the liquid phase forms too early and probably in too large quantity to obstacle the sintering process, as effectively can be observed through the microstructure observations and the fracture analyses.

![Impact Resistance Graph](image)

**Figure 3.** The influence of sintering temperature on Impact resistance for samples without and with 3 % CuSn10 addition.

![Fracture Morphology](image)

**Fig. 4:** Fracture morphology of ST2+3 %wt bronze (A and B) and ST0 +3 %wt bronze (C and D) samples sintered at 1150 °C.

The fracture surfaces morphology of the sintered samples shows the presence of pores together with some inclusions coming from the used powders. In figure 4 are illustrated the fracture surfaces of samples added with 3wt% of bronze and sintered at 1150 °C. Ductile and brittle aspects are evident, in particular intergranular cleavage zones appears due to possible reinforcement effects on the matrix. Some large pores are present and globular grains
constituting the matrix are surrounded by the bronze phase, highlighting a strong liquid phase sintering process. Moreover, it is possible to observe that some grains are isolated, without the presence of any sintering neck, to state the scarce sintering degree; the effect is always more evident on the samples based on the ST2 powder, evidently the adopted process is not convenient especially for this type of alloy. Further tests are necessary in order to better understand the sintering mechanisms for this kind of alloys and to better fit the compositions and the process parameters.

Conclusions

The aim of this research was to study the influence of some process parameters and of contact infiltration on the mechanical and dynamic properties of PM AISI 316L grade stainless steel. In particular compacting pressure and sintering temperature play an important role in decreasing the porosity degree of specimens and increasing their mechanical properties. However, contact infiltration process can represent a suitable alternative to high pressure compacting and to high temperature sintering. In fact with this process it is possible to compact at lower pressure, 500 MPa, and sintering at a temperature range of about 1150 °C that usually is adopted in continuous furnaces, producing sintered parts with density close to the theoretical values and with very good mechanical and impact properties.

The evaluation of the energy absorbed during the impact tests by means of an instrumented Charpy machine shown that the highest amount of energy is consumed to nucleate the fracture, while the propagation of the fracture require only a small contribution of energy. The infiltration process more than compacting pressure and sintering temperature has a very positive and strong effect on the energy absorption capacity, and on the nucleation component in particular.

The second part of the work, dedicated to the study of the possible addition of bronze powder directly to the iron based powders to cause liquid phase sintering and to look for possible analogies with infiltration, was not fully successful and further tests are necessary in order to better understand the sintering mechanisms for this kind of alloys and to better fit the compositions and the process parameters.

References