Materials Research, Vol. 6, No. 4, 475-480, 2003.

Compresibility and Sinterability of HCx PM Steel Diluted with Stainless Steels

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Received: March 10, 2003; Revised: July 27, 2003

HCx powder metallurgy steel contains in its composition high contents of Cr and C, and significant quantities of alloy elements typical of tool steels (Mo, V, W), to provide the corrosion resistance of stainless steel with wear resistance of tool steels. HCx appears to be a suitable material for applications in aggressive environments, as valve seat inserts in automotive engines. However, this steel presents a low compressibility leading to high production costs. In this work, some results carried out to improve the compressibility of HCx are presented. The way to attempt this improvement is the dilution of base material with two stainless steels, the ferritic 430LHC and the austenitic 316L. The powder mixes prepared were uniaxially pressed to study the compressibility. The sinterability was study by determining of density, hardness, transverse rupture strength (TRS) and microstructural evolution after vacuum sintering at different temperatures. As a result, better compressibility is observed in the mixes although not all of them present the properties required.

Keywords: *powder metallurgy, dilution, stainless steels*

1. Introduction

The mayor quantity of parts produced by powder metallurgy (PM) techniques are consumed by the automotive industry, which is one of more active sector in the development of new products manufactured by PM[1,2]. Into the automotive sector, the most of PM parts are for engines. Some of them are submitted to high wear and corrosion demands, working usually at high temperatures. This is the case of valve seat inserts, frequently made of high-speed steels due to their ability to maintain hardness at high temperatures and their high wear resistance. However, the more demanding requirements for engines imply higher properties for materials and, for some applications, high-speed steels are not suitable.

Recently, a new type of PM high alloy steel has been developed, which commercial name is HCx, characterised for high Cr and C contents in its composition, besides of quantities of tool steel alloying elements, such as Mo, V and W. The microstructure after sintering consists of a dispersion of hard carbides in a ferritic matrix. This steel has been designed to combine high wear and corrosion resistance, provided that enough Cr remains in the matrix to be corrosion resistant and carbides support wear[3]. Therefore, this material seems to be suitable for applications that require both kinds of properties[4]. However its low compressibility and the high costs can limit the utilisation.

Some authors[5,6,7] have followed investigations related with dilution or blending of low compressibility powders with others presenting higher compressibility, looking for the improvement of this property remaining good mechanical and wear properties. The dilution has also the advantage of diminish the final cost of the material.

The present work shows some results from a study carried out through the dilution of HCx with different percentages of two types of stainless steel, one ferritic (AISI 430LHC), because of its compatibility with the HCx matrix, and other austenitic (AISI 316L). The interest of diluting with 316L lies on the posterior study about the influence of austenite on wear behaviour, as this effect is no clearly defined[8]. On the other side, the microstructure of the added steel can make an influence on the diffusion of alloying elements from the HCx particles to the stainless steel,

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Trabalho apresentado no XV CBECIMAT, Natal - RN, Novembro de 2002.

which will influence, as a consequence, in the interfaces and the sinterability of diluted materials.

2. Materials and Processes

The base material used in this study has been powder of HCx, from Powdrex. As diluting materials, two stainless steel powders, AISI 316L and AISI 430LHC, from Coldstream, have been used. The composition and characteristics of these powders (from suppliers) are shown in Tables 1 and 2.

Table 3 shows the composition of the mixes prepared, indicating the percentages of dilution. The powders were mixed in a ball mill at a rotary speed of 100 rpm, for 60 min.

After mixing, they were characterised by measuring flow speed, apparent density, compressibility and green strength. For the determination of flow speed and apparent density a Hall flowmeter was used, carrying out the tests under the MPIF03 and ISO 3923-1:1979 standards. The compressibility was determined by pressing powders of each composition at different pressures between 550 and 700 MPa. The compacts were used to determine green strength.

To study the sinterability of diluted materials, rectangular samples of each composition were prepared by uniaxial pressing at 700 MPa, pressure obtained from the compressibility curve. The dimensions of these samples are in accordance with those indicated in the standards for bending tests of PM products. Compacts were sintered in vacuum for 30 min, at different temperatures: 1200, 1220, 1240 and 1260 °C. Some properties were studied after sintering: density, hardness, dimensional change and transverse rupture strength (TRS). Density was determined by a method based on the Archimedes principle, and the results are presented as relative density, that is, measured density to the theoretical. Hardness was measured in Rockwell A scale, and TRS was evaluated through three points bending tests. All the values presented are the media of at least four samples. The study is completed with the microstructural analysis by scanning electron microscopy (SEM).

3. Results and Discussion

Figure 1 shows the compressibility curves of base material and diluted materials with both stainless steels. In all the cases green density increases when the compacting pressure increases, which is the normal tendency, but the most important is that the higher the dilution (the percentage of stainless steel), the higher the green density for the same compacting pressure, which is the wanted result.

The green strength values (Fig. 2) for these samples are less influenced for the dilution than density values, that is an interesting result as this property is important to be maintained for the industrial process.

Once the first objective has been reached, it is important to know the feasibility to sinter the diluted materials, and the best sintering conditions. Figure 3 shows the evolution of relative density with sintering temperature. The base material presents a high density (97% of the theoretical)

Table 3. Composition of the mixes prepared (% in mass).

Mix	HCx	316L	430LHC
1	100	-	-
2	85	-	15
3	70	-	30
4	55	-	45
5	85	15	-
6	70	30	-
7	55	45	-

Table 1. Composition of initial materials (% in mass).

	C	Cr	W	Mo	V	Si	Ni	Cu	Mn
HCx	1.6	22.6	2.7	2.8	1.9	1.4	-	-	-
316L	0.021	16.1	-	2.24	-	0.87	13.55	0.02	-
430LHC	0.018	16.85	-	-	-	1.15	-	-	0.18

Table 2. Characteristics of initial materials.

	HCx	316L	430LHC
Apparent density (g/cm^3)	2.5	2.95	2.77
Flow rate (s/50 g)	33.9	28.3	27.85
Particle size	0.57% > 150 µm 25% < 45 µm	0.22% > 150 µm 31.1% < 45 µm	99.8% < 150 µm

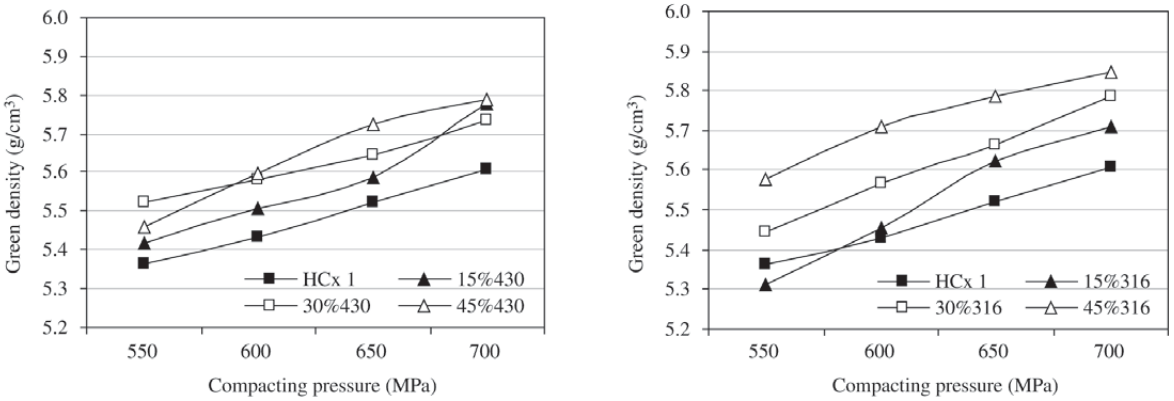


Figure 1. Compressibility curves of diluted materials.

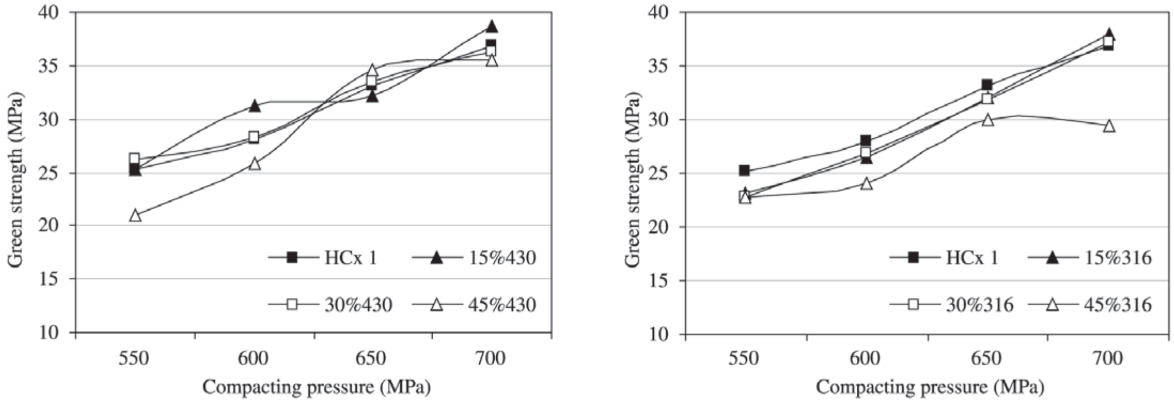


Figure 2. Green strength of diluted materials *vs.* compacting pressure.

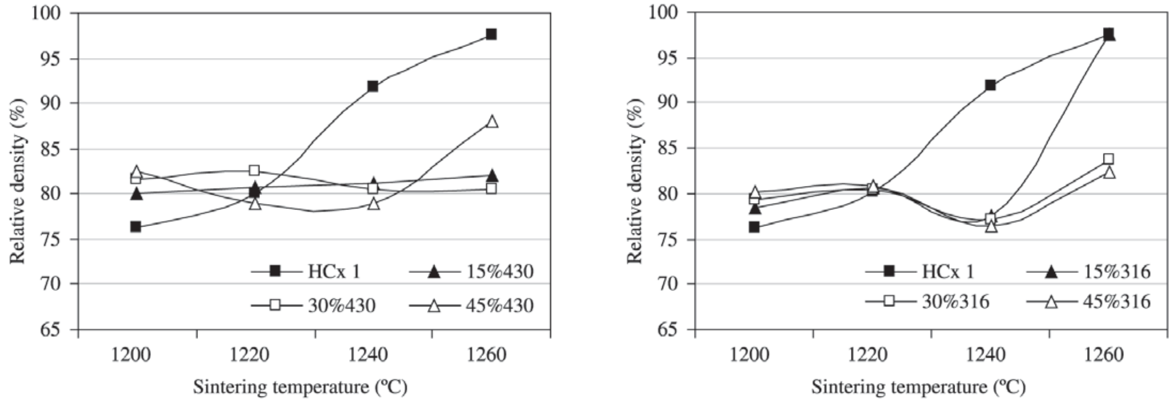


Figure 3. Relative density of diluted materials *vs.* sintering temperature.

when sintered at 1260 °C, and more than 90% of the theoretical is obtained for 1240 °C, which is an appropriate value for many applications. However, among the diluted materials only that containing 15% 316L presents the same values of density than base material. The rest of materials diluted with both stainless steels do not overcome 82% at the temperatures studied, being this an almost constant value. As all the materials have similar theoretical values, the de-

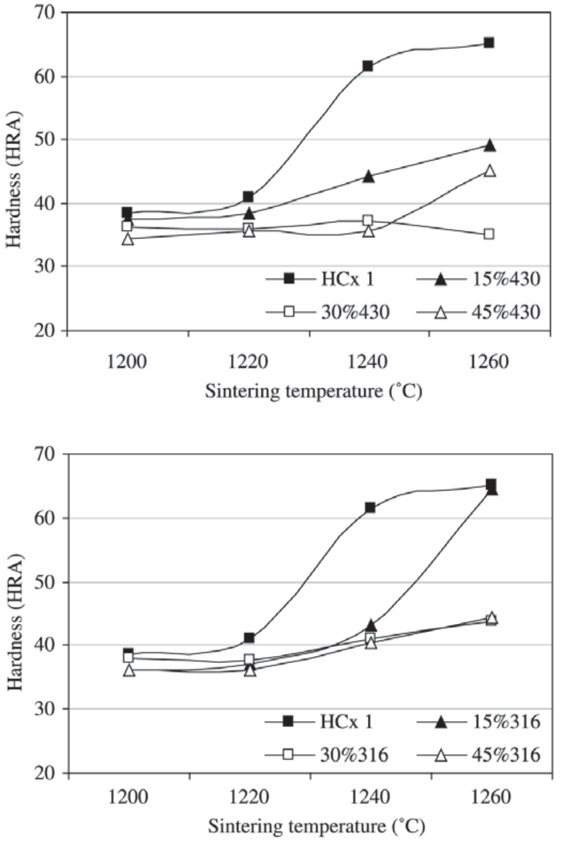


Figure 4. Hardness (HRA) of diluted materials *vs.* sintering temperature.

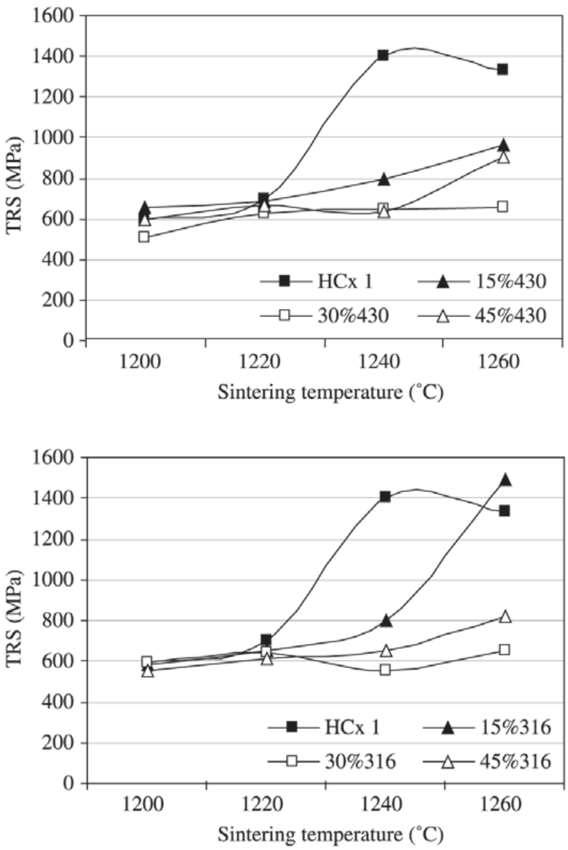


Figure 5. Transverse rupture strength (TRS) of diluted materials *vs.* sintering temperature.

crease of density is not due to this factor.

In PM materials, density values are intimately related with their properties. Thus, it can be observed on Fig. 4 that the highest values of hardness (65 HRA) are for base material sintered at 1260 °C and for the composition containing 15% 316L sintered at the same temperature, that is coincident with density values. It is worth to notice that although density values are almost independent on the sintering temperature for the most of the compositions studied, values of hardness present a tendency to increase with the sintering temperature. This feature can be observed for all the materials containing 316L, among which the addition of 15% leads to the same values of hardness than base material sintered at 1260 °C. Higher percentages of addition permit values of hardness at 1260 °C up to 10 points over those obtained at 1220 °C, although density does not increase. Regarding the materials diluted with 430LHC, the most significant change is for the composition with the lowest percentage of dilution, increasing the hardness 10 points from 1220 to 1260 °C, while density remains constant. These evolution point out to microstructural changes, probably due to the activation of diffusion processes, more than a decreasing of the porosity, if values of density are taken into account.

Results of TRS (Fig. 5) present the same tendencies than hardness. In this case, base material reaches the highest value (1400 MPa) for 1240 °C, meaning that a possible oversintering for materials sintered at 1260 °C has occurred. As in the previous properties, the only diluted material that reaches high values is the mixing with 15% 316L sintered at 1260 °C, which present values of 1500 MPa, higher than base material. To understand the results of TRS it is necessary to take into account that the stainless steels used as addition materials present lower TRS values than base material, so we can expect that diluted materials show intermediate values of TRS. The other factor of influence is density, which also makes values of TRS decrease. Both features seem to be applicable to materials diluted with 430, which low values can be explained for the mayor quantity

Table 4. Composition (% in mass) of phases in Fig. 6a.

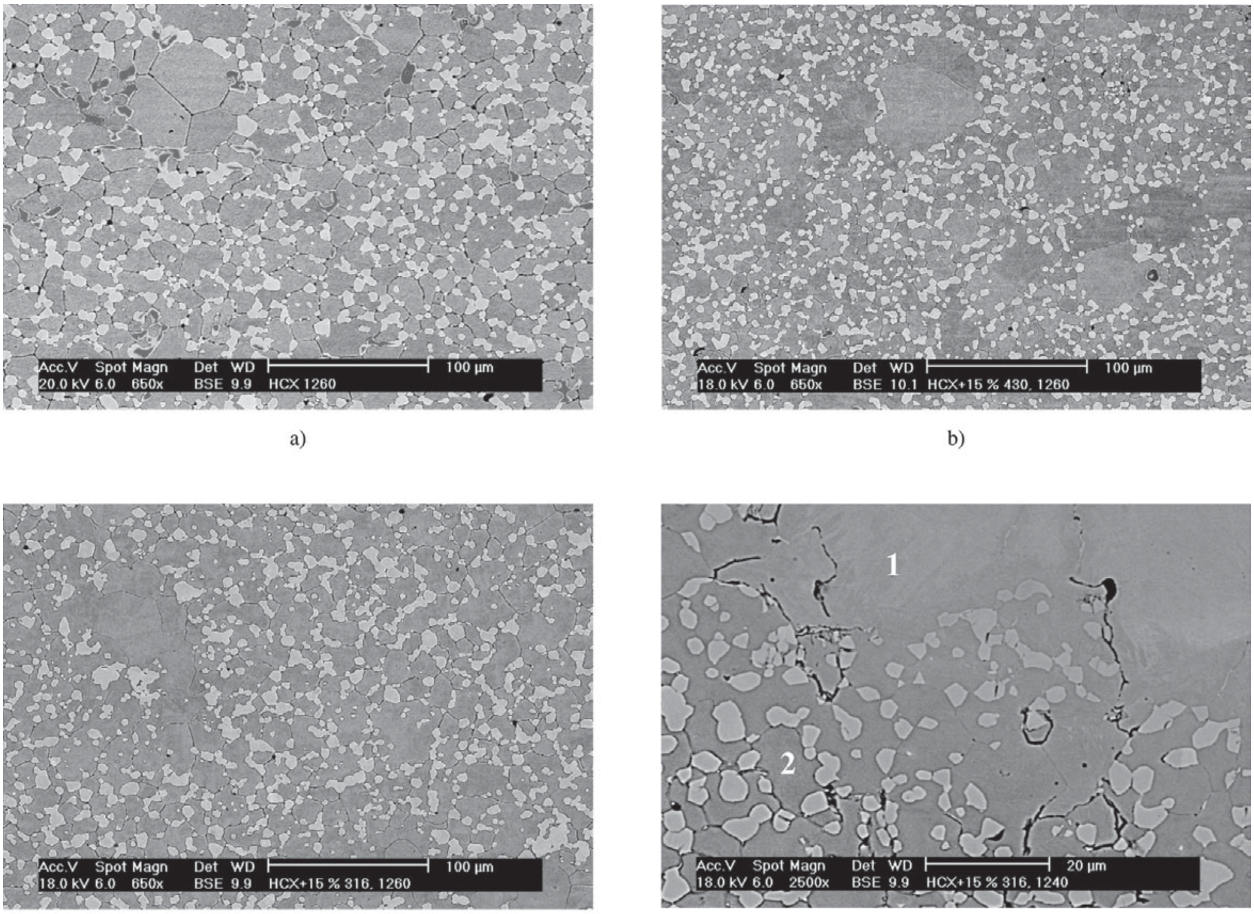
	Mo	V	W	Cr	Fe	Si
Matrix	-	1.4	-	14.6	82.1	2.3
Dark phase	0.8	9.8	0.8	63.1	25.7	-
Bright phase	5.3	4.8	6.6	46.9	36.4	-

Table 5. Composition (% in mass) of phases in Fig. 6d.

	Mo	V	Ni	Cr	Fe	Si
Zone 1 matrix in 316L	2.1	0.7	8.4	15	71.8	1.9
Zone 2 matrix en HCx	2.8	1.18	2.53	13.53	77.34	2.63

of ferrite and low density. However, in the case of dilution with 316L the final material can be considered to have a duplex stainless steel matrix, which mechanical properties are higher to the ferritic or austenitic stainless steels[9]. This point, besides the high density, can justify the high TRS value reached by the 15% 316L material sintered at 1260 °C. However, for higher percentages of dilution, the lower densities impede the material to reach higher values of TRS, remaining around 800 MPa.

The microstructure of materials helps to understand the properties and confirms some of the explanations above. Figure 6 shows SEM microstructures, taken in the back scattering (BSE) mode, of several materials sintered at 1260 °C. Figure 6a corresponds to base material; the bright contrast phase are complex carbides with Cr, Mo, W and V in their composition, where quantities of Fe are dissolved.



a) b)

c) d)

Figure 6. Microstructures (BSE) of several materials sintered in vacuum at 1260 ºC: a) HCx; b) HCx+15 % 430LHC; c) and d) HCx+15 % 316L.

The dark contrast phase are complex carbides with higher content of V and Cr than bright ones and almost no content of W and Mo. This dark phase is only found at the core of bright carbides placed at the grain boundaries, in the materials sintered at highest temperatures. It can be due to a microsegregation phenomena during the solidification of a liquid phase. The composition of the matrix, measured by EDX reveals that enough content of Cr (14%) remains dissolved, ensuring its stainless character. The composition of mentioned phases are in Table 4. In the same Fig. 6a, it is possible to notice the homogeneous distribution of carbides, but also, it can be seen the big size of carbides and ferrite grains, especially near dark contrast phases, where a liquid phase could be formed during sintering. These features indicate that the material have been slightly over-sintered, as mentioned before.

In materials diluted with 430, for the same sintering temperature, both the grain size and the carbide size are smaller than base material. In Fig. 6b, showing the 15% 430 material, areas free of carbides can be distinguish where former 430 steel particles. These areas are perfectly bonded to the matrix, being the boundaries between HCx particles similar to the boundaries between HCx and 430 particles. At higher magnifications no interfaces can be seen that indicates diffusion phenomena.

However, in materials diluted with 316L those interfaces can be found. In Fig. 6c, taken at low magnifications, is not easy to observe the interfaces, but other interesting features can be noticed, as grain size and carbide size, that appear similar to base material sintered at the same temperature. At higher magnifications it is possible to notice diffusion phenomena between HCx and 316L areas. This is the case of Fig. 6d where a gradual change of contrast indicates a gradual change of composition. In Table 5 the composition (from EDX analysis) of 316L near boundary (marked as 1 in Fig. 6d) and HCx matrix (marked as 2 in Fig. 6d) are presented, and they reveals the diffusion of Ni from 316L to HCx. Deeply studies of this diffusion phenomena are currently carrying out.

4. Conclusions

The compressibility of HCx powder can be improved by dilution, or blending, with conventional stainless steel powders like 316L or 430LHC.

Dilution of HCx with 316L steel permits to obtain materials after vacuum sintering with values of density, hardness and TRS similar or higher than base material. This represents an advantage in relation with plain HCx due to the higher compressibility of the diluted material and the lower cost.

Dilution of HCx with 430LHC, in the percentages studied, does not permit to obtain materials sintered in vacuum with high densities, and present lower properties than base material.

Acknowledgements

Authors want to acknowledge to Spanish Ministry of Science and Technology for the support through the research project COO1998AX149.

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