Characterization of a 17-4 PH stainless steel obtained through metal powder Hot Isostatic Pressing process for automotive application

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Abstract. Steels of the family 17-4 Precipitation Hardening stainless steels are often considered as potential candidates for applications requiring high levels of strength (typically above 600 MPa in tensile strength) and a moderate ductility. The aim of this work is the characterization of a 17-4 precipitation hardening stainless steel obtained through metal powder Hot Isostatic Pressing process. The main purposes of using Hot Isostatic Pressing process are the better chemical composition and microstructure homogeneity, the finer grain size (allowing to higher mechanical properties), and the possibility to obtain net shape products, avoiding subsequent finishing operations. The Hot Isostatic Pressing process temperature does not seem to significantly influence the tensile properties, while, the aging temperature is a key factor: lower aging temperature brings to higher strength and lower ductility. However, a higher influence of the Hot Isostatic Pressing process temperature was detected on the toughness. Improved toughness both at room and at low temperature was obtained by optimizing the aging treatments. An improvement of 32 % in impact energy was obtained by the optimization of the aging process thanks to a higher austenite content, which has shown a nanosized shape between the martensite platelets, in the steel structure.

1. Introduction

17-4 PH steel is a martensitic precipitation hardening (PH) stainless steel which offers a combination of high mechanical strength up to a temperature of 300°C, good corrosion resistance and good toughness, thanks to the possibility of a wide selection of heat treatments. It also exhibits excellent weldability and good workability. The great advantage of these steels is that they can be supplied in the "solution treated" condition. In this condition the steel is just machineable. Following machining, forming etc. the steel can be hardened by a single, fairly low temperature "ageing" heat treatment which causes no distortion of the component All these qualities make the 17-4 PH a valid solution in specific sectors: from aerospace, to chemical and petrochemical, to food, to the mechanical industry in general. [1]

The hot isostatic pressing process (HIP) consists in the simultaneous application of high pressure and high temperature to the component in special vessels. Because the pressure is applied through an inert gaseous medium, the process is denoted as isostatic pressing. This process is a valid alternative to more traditional processes, such as casting, and forging.

Among the main advantages over the latter are:
• better chemical composition homogeneity;
• better microstructural uniformity, which leads to isotropic properties;
• a finer grain microstructure, which improve mechanical properties.

Although process costs are higher, mainly due to the preliminary stages, part of these are recovered considering the minimum waste of associated material. In fact, with this technology, near net shape or even net shape products are obtained, thus avoiding to carry out all the finishing operations downstream of the traditional methods. Thanks to these advantages extended research was performed on the HIP process applied on many metals and alloys [2-5]. Moreover HIP is very effective for compacting different kinds of metal wastes in the form of granules and pellets [6-9]. Furthermore it was shown that high pressure and temperature during HIP enables to increase the level of mechanical properties of the forgings [10, 11].

Aim of this work is to characterize the mechanical properties and the microstructure of a 17-4 precipitation hardening stainless steel obtained through metal powder HIP and to investigate the effect of HIP process temperature and aging temperature on the properties of the steel.

2. Experimental part

In this study two kinds of 17-4 steel powders obtained by gas atomization were used. The powder A was obtained using argon as the gas atomizer while the powder B was atomized by nitrogen. The chemical composition of the two powders are summarized in Table 1.

| Powder | C (%) | Cr (%) | Ni (%) | Cu (%) | Nb + Ta (%) | N (%) | Si (%) |
|--------|-------|--------|--------|--------|-------------|-------|-------|
| A      | 0.05  | 16.1   | 3.6    | 3.5    | 0.31        | 0.01  | 0.48  |
| B      | 0.06  | 17.1   | 4.5    | 3.2    | 0.35        | 0.03  | 0.58  |
| Steel  | 0.05  | 16.7   | 4.6    | 3.4    | 0.33        | 0.01  | 0.55  |

Four cylindrical capsules of mild steel with 150 mm high and 180 mm in diameter were filled in a protected atmosphere, two with powder A and two with powder B. The filled capsules were degassed at temperatures of about 573 K and pressures of about 1 Pa. At the end of the degassing, the capsules were sealed using a crimping tool.

HIP process was performed at temperature of 1273 and 1313 K. After the corresponding pressing treatment, each billet was divided into 2 portions and each portion was submitted to aging at 2 different temperatures (813 and 873 K) for 4 hours.

The specimens necessary for the tests were extracted from each heat-treated portion: 3 specimens for the tensile test and 3 specimens for notched bar impact testing. Optical microscopy analysis was used to study the surface of the samples. The tensile tests were performed in a Schenck Hydropuls PSA 100 machine, at room temperature. The Charpy impact tests were performed with V-notched samples prepared as reported in accordance with standard ASTM E23. The grain size measurements were performed in accordance with standard ASTM E112.

3. Results and Discussion

The results of tensile tests for each material are summarized in Table 2 and compared with the same massive steel grade treated under the same conditions.
Table 2. Average results from tensile tests of hipped samples and massive steel.

| Powder | HIP Temperature* (K) | Aging Temperature (K) | Yield Strength (0.2%) (MPa) | Tensile Strength (MPa) | Elongation (%) |
|--------|-----------------------|-----------------------|-----------------------------|----------------------|----------------|
| A      | 1273                  | 813                   | 913 ± 4                     | 983 ± 3              | 11.9 ± 0.1     |
| A      | 1273                  | 873                   | 738 ± 1                     | 898 ± 2              | 12.3 ± 0.2     |
| A      | 1313                  | 813                   | 836 ± 3                     | 903 ± 4              | 12.4 ± 0.1     |
| A      | 1313                  | 873                   | 709 ± 3                     | 862 ± 4              | 13.0 ± 0.1     |
| B      | 1273                  | 813                   | 962 ± 3                     | 1028 ± 5             | 11.8 ± 0.2     |
| B      | 1273                  | 873                   | 849 ± 4                     | 927 ± 7              | 12.9 ± 0.1     |
| B      | 1313                  | 813                   | 955 ± 4                     | 1032 ± 3             | 12.2 ± 0.1     |
| B      | 1313                  | 873                   | 802 ± 1                     | 931 ± 3              | 12.5 ± 0.1     |
| Massive steel | 1273                  | 813                   | 1000 ± 15                   | 1070 ± 17            | 12.3 ± 0.4     |
| Massive steel | 1313                  | 873                   | 795 ± 17                    | 965 ± 11             | 14.6 ± 0.3     |

* for massive steel the temperature reported in the first column is the solution temperature.

The dispersion of the tensile tests results is very low, especially the results concerning the yield and tensile strengths. This behaviour is frequent in materials obtained in powder metallurgy thanks to their high compositions homogeneity and to an almost absence of segregations and to the high microstructural isotropy intrinsic to the HIP process [11].

The mechanical properties obtained by HIP processing of the two powders are close to the mechanical properties of the massive steel. It can be noted that samples obtained from powder B shows higher resistance than the corresponding samples obtained from powder A. The reason can be due to the higher amount of alloying elements (especially nitrogen) in powder B, which acts as a reinforcement of the matrix [12]. Instead, the difference in elongation percentage is negligible.

The temperature of HIP processing does not affect significantly the mechanical properties. In fact, an increasing in HIP temperature produces a very slight drop in the range between 4-1.5% in yield strength and a small increase in the range between 4 and 0.7% in elongation percentage. These ranges can be considered negligible.

Aging temperature has a significant effect on the mechanical properties. In fact, an increase of 60 K in aging temperature produces an increase in yield strength of about 20% and a reduction in elongation percentage of about 9%.

The results of impact tests are summarized in Table 3 and, as for the results of tensile tests, they are compared with the same massive steel grade treated under the same condition.

Table 3. Average results from Chapry tests of samples after HIP and massive steel.

| Powder   | HIP Temperature* (K) | Aging Temperature (K) | Fracture Energy (Joule) |
|----------|-----------------------|-----------------------|-------------------------|
| A        | 1273                  | 813                   | 40 ± 4                  |
| A        | 1273                  | 873                   | 33 ± 3                  |
| A        | 1313                  | 813                   | 51 ± 3                  |
| A        | 1313                  | 873                   | 44 ± 2                  |
| B        | 1273                  | 813                   | 39 ± 3                  |
| B        | 1273                  | 873                   | 31 ± 2                  |
| B        | 1313                  | 813                   | 52 ± 3                  |
| B        | 1313                  | 873                   | 45 ± 2                  |
| Massive steel | 1313                  | 813                   | 56 ± 5                  |
| Massive steel | 1313                  | 873                   | 68 ± 5                  |

* for massive steel the temperature reported in the first column is the solution temperature.

In this case the impact resistance of the samples after HIP is lower than the resistance of the massive steel. Although by HIP it is possible to eliminate all the porosities, the interfacial bonds between the particles which are created during the process may not be sufficient to guarantee performance similar to
massive material due to the impurities that can be present on the particles surface [13]. It can be noted that the difference between the samples produced by different powders is negligible. This is because the difference in alloying elements and impurities is not sufficient to generate different paths for the cracks growth.

The temperature of HIP processing affects the impact resistance of the materials. In fact, the treatment at higher temperature leads to the increase of the fracture energy, especially at room temperature. However, the aging treatment has a larger effect on the impact energy. In fact an increase of 60 K in aging temperature produces a reduction of fracture energy in the range between 20% and 30%.

The microstructure of the samples obtained from the powder A are reported in Fig. 1 and the microstructure of the samples obtained from powder B are reported in Fig. 2.

Figure 1. Microstructure of samples obtained from powder A, x1000:
(a) HIP temperature 1273 K, aging temperature 813 K;
(b) HIP temperature 1273 K, aging temperature 873 K;
(c) HIP temperature 1313 K, aging temperature 813 K;
(d) HIP temperature 1313 K, aging temperature 873 K.

Figure 2. Microstructure of samples obtained from powder B, x1000:
(a) HIP temperature 1273 K, aging temperature 813 K;
(b) HIP temperature 1273 K, aging temperature 873 K;
(c) HIP temperature 1313 K, aging temperature 813 K;
(d) HIP temperature 1313 K, aging temperature 873 K (a) first picture; (b) second picture.
It can be noted that there is a total absence of porosity. As expected, the isostatic pressing treatment leads to a relative density of about 1. The microstructure is, in all the samples fully martensitic, characterized by small plate martensite, similar to the massive steel microstructure. There are no traces of delta-ferrite. The structure is equiaxial and homogeneous. Samples obtained from powder B show a higher amount of precipitates at the martensite plates boundaries, probably due to a higher percentage of carbon. This justifies the increase of strength of the samples after HIP processing obtained from the powder B.

Average grain size analysis shows that the level of temperature in HIP processing do not influence on the microstructure, while the aging temperature is more important: in fact, the average grain size is increased from 70-80 µm to a range 130-150 µm with the increase of the aging temperature to 40 K. This correlates with the reduction in fracture energy observed in the samples aged at higher temperature.

4. Conclusions
The 17-4 PH stainless steel obtained through metal powder Hot Isostatic Pressing process, especially for the material obtained from powder B, have microstructure with higher amount of precipitates at the martensite plates boundaries.

Material treated at higher aging temperature is more deformable, while one treated at lower aging temperature has higher level of strength but reduction in ductility. Conversely, the temperature of HIP processing does not significantly influence the tensile properties.

Due to high chemical homogenization, low segregation, and high microstructural isotropy obtained after HIP process the results of tensile tests show minimal dispersions in mechanical properties, especially of tensile strength. The impact properties of 17-4 PH stainless steel obtained from powders are lower as compared with the massive material.

The influence of the HIP processing temperature is more relevant than the results of tensile tests. However, aging treatment at different temperatures has higher effect on impact properties. Fracture energy is higher to 20-30% for the material treated at lower temperature than for the same material treated at higher aging temperature. It can be explained by the increase of grain size at higher aging temperature.

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