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AISI 310 Stainless Steel Formed by Gelcasting: An Alternative Manufacturing Method

Oliveira Louise Fernanda Rodrigues[1,a*], Neves Maurício David Martins das[1,b], Ortega Fernando dos Santos[2,c]

[1]Centro de Ciência e Tecnologia de Materiais do Instituto de Pesquisas Energéticas e Nucleares, IPEN-CNEN/SP, Av. Prof. Lineu Prestes, 2242 – Cidade Universitária CEP 05508-000 – São Paulo – SP – Brasil

[2]Departamento de Engenharia de Materiais do Centro Universitário da FEI, Avenida Humberto de Alencar Castelo Branco, 3972 – Bairro Assunção – CEP 09850-901 São Bernardo do Campo – SP – Brasil

[a]louisefernandarodrigues@gmail.com, [b]mdmn@ipen.br, [c]ferortega@fei.edu.br
*corresponding author

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Abstract: This work evaluates the microstructure and the yield strength under compression at room temperature and at 800°C of specimens prepared with AISI 310 stainless steel powder (D50 = 10 μm), manufactured by gelcasting. Parts were vacuum sintered in a single batch at 1280°C. At room temperature, specimens presented average yield strength of 270 MPa, and at 800°C, 105 MPa. Microstructure analysis involved the measurement of grain size along the vertical axis of cylindrical specimens, with special attention to the effect of particles settling, and was conducted using scanning electron and optical microscopy, and X-ray diffraction. Settling effect was assessed considering the position where the specimen was taken and was negligible: both density and yield strength did not vary significantly along the vertical axis.

Introduction

The gelcasting process is a forming technique originally developed for ceramic systems, which is based on an *in situ* polymerization of organic monomer dissolved in the aqueous phase of a suspension. After the polymerization, a gel rich in crosslinks is formed, holding the powder particles together (1, 2). Gelcasting process enables the production of parts with complex geometry, homogeneous microstructure and high green strength using a small quantity of organic binders (1) with potential advantages over some traditional powder metallurgy processes and metal injection molding (3). Most recently, gelcasting process was used to obtain metal foams or porous parts, as stated by Lin and Kennedy (4) and Kapat, Srivas and Dhara (5), and dense parts, as announced by Ye et al. (6) and Ortega and Oliveira (7). A problem of this process is that metallic particles are generally denser and larger than ceramic particles, leading to fast sedimentation in liquid media. Nonetheless, the settling of particles can be delayed by using a suspending agent, such as a water-soluble long chain polymer. If there is nothing to avoid particles settling, a strong influence on the microstructure and densification during sintering could happen, since there is an induction period of a few minutes before the sample gelling takes place. This work studies the microstructure of parts formed by gelcasting, considering the possibility of settling effects and densification differences. And also, the yield strength values at room temperature and at 800°C, in comparison with data published of the same stainless steel (8).

Materials and Methods

AISI 310 (HK-30) stainless steel powder 20F grade was supplied by ATMIX Corporation, whose chemical composition is shown on table 1. Particle size measurement of powder was performed with laser diffraction technique (Microtrac BlueWave).

Table 1 – HK-30 stainless steel chemical composition

Elem.	Ni	Cr	C	Si	Mn	Cu	Nb	P	S	Mo	Co	V	Sn
wt.%	20.47	25.22	0.476	0.98	0.83	0.06	1.35	0.021	0.07	0.04	0.07	0.05	0.1

A suspension with 59vol.% of powder was used (7, 9). A premix consisting of 30wt.% of monomers was prepared by dissolving methylacrylamide (MAM, Sigma-Aldrich) and polyethyleneglycol-dimetacrylate (PEG(DMA), Sigma-Aldrich) in distilled water at a 2:1 molar ratio. Was used as dispersant in a 10wt.% solution of the 0.5wt.% of sodium polyacrylate (Dispex N-40, Lubrizol). Polyvinyl alcohol (PVAl, Mallinckrodt) was used as suspending agent at 1wt.% of the premix. After premix preparation, small amounts of powder were added to it under vigorous stirring to avoid settling and bubble formation. A quantity of 4.5 μL of tetrametylethylenediamine (TMED, Sigma-Aldrich) per gram of suspension was added as catalyst and 9.0 μL of ammonium persulfate (APS, Merck) in a 30wt.% solution per gram of suspension was added as initiator to start the polymerization.

The suspension was poured into an aluminum cylindrical mold of 90 mm high and a diameter of 14 mm, where it gelled into rigid bodies in a few minutes, at room temperature. The gelled bodies were demolded and immersed in an aqueous solution containing 40wt.% of polyethyleneglycol (PEG, Oxiteno) for a few hours in order to dry the samples by osmosis, a drying technique that reduce stresses during this operation (10). After washing the samples with tap water were dried (110°C - 4h). Parts were vacuum sintered in a single batch, in order to avoid the influence of any different conditions on the results. Since the sintering step was carried out in an industrial furnace, a testing cycle was held in order to verify if the parts would resist the proposed heating rate. The parts were vacuum sintered at 1280°C for 60 min with a heating rate of 8.5°C/min, followed by slow cooling inside the furnace. Vacuum atmosphere at 10^{-5} Torr was preferred because it provides a good densification (11) and the final temperature was based on a previous work (3). A thermogravimetric analysis (TG-DSC Setsys Evolution 1750, SETARAM) was done to verify the weight loss on a gelcast sample and on the powder (6). It was expected that debinding would occur during the sintering cycle, without a need of a specific step for it. After sintering, each cylindrical part was cross-sectioned into four samples, with diameter of 10 mm and length of 15 mm from the top and bottom positions, producing a total of 30 specimens. Density measurements was done to check if there was any difference between the samples taken from the top and the bottom of the part. Density of powder and of a sintered sample were measured by pycnometry (AccuPyc II 1340, Micromeritics) (12) and compared to the results obtained by geometrical method. Optical micrographs (BX60M, Olympus) and SEM (CamScan 3200 LV, Oxford Instruments) images were taken in order to verify the microstructures regarding to pores, densification and grain size as per ASTM E112-13 standard (13). For this check, 10 micrographs were taken from 10 random positions. After previous characterization of specimens, compression tests at room temperature and at 800°C were performed, as stated by ASTM E9-09 and ASTM E209-00 standards, respectively (14, 15). Specimens were put in the testing machine randomly, without specifying from where in the cylinder it was took. The aim was to verify if they would achieve values reached by previous studies and also determined by ASTM A351/A351M-14 standard (16), and also to evaluate the settling effect.

Results and Discussion

The powder density measured by pycnometry method was 7.5709 g/cm³ for AISI 310 (HK-30), matching with the literature value of 7.5 g/cm³ (17). Spherical particles were predominant in the powder sample, as shown on figure 1. This characteristic improves particles packing and densification during sintering process, reducing (but not eliminating) porosity in microstructure. Although spherical particles predominate in this powder, a few elongated particles were observed, probably due to the coalescence of metallic drops during atomization.

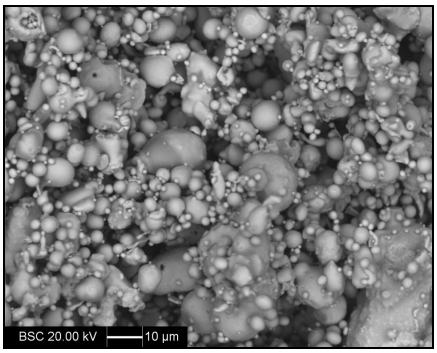


Fig. 1 – Powder SEM image with magnification of 1000x

The particle size distribution on figure 2a shows a HK-30 10F with D50 = 7 µm and HK-30 20F has D50 = 12 µm. Data from a finer powder was took only for comparison and also to show its settling velocity differences. These diameters, when applied to the Stokes' Law, presents a difference of almost 3 times between their settling velocity, according to equation 1 (18). This difference could result in an undesired variation of microstructure along the vertical axis. From this equation, v = settling velocity [m/s]; g = gravity [m/s²]; a = particle diameter [µm]; ρ_p = steel density [g/cm³]; ρ_L = liquid density [g/cm³]; e η_L = liquid viscosity coefficient.

$$v = \frac{a^2 \cdot (\rho_p - \rho_L) \cdot g}{18 \cdot \eta_L} \rightarrow \frac{v_{20F}}{v_{10F}} = \frac{\frac{12^2 \cdot (\rho_{HK-30} - \rho_L) \cdot g}{18 \cdot \eta_L}}{\frac{7^2 \cdot (\rho_{HK-30} - \rho_L) \cdot g}{18 \cdot \eta_L}} = \frac{12^2}{7^2} = \frac{144}{49} = 2.94 \quad (1)$$

The TGA analysis shows that the formed green sample presented a weight loss of 4.0%, which occurred at the temperature range up to 550°C. On the other hand, the weight loss of the powder was smaller than 0.5wt.% in the same temperature range. Therefore, it maybe concluded that in this temperature range occurs the complete volatilization of the polymeric gel (see figure 2b). The powder presented negligible weight loss, and flow variation, what confirms the previous conclusion that debinding was successfully performed during the vacuum sintering process, which was not changed for this purpose. The shrinkage occurrence is predictable and isotropic, respecting dimensional tolerances (19).

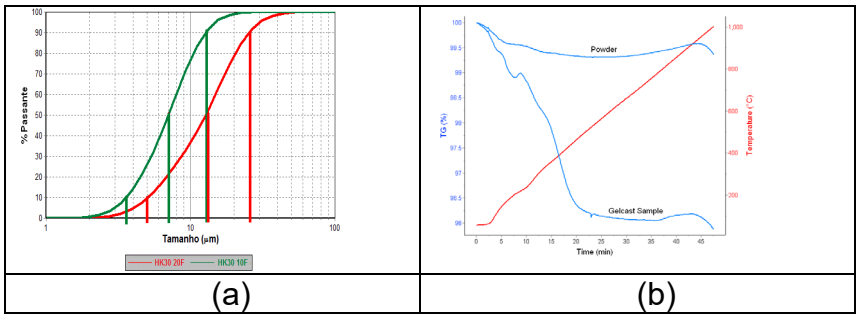


Fig. 2 – (a) Particle size distribution of HK-30 10F (green line) and HK-30 20F (red line) powders obtained on laboratory and (b) DSC/TGA tests of HK-30 gelcast sample and powder.

The geometric density and real density of specimens taken from the top and the bottom of the cylinder. Figure 3 shows that within the statistic fluctuation of the test, there was no difference between their values of 10 samples for each case.

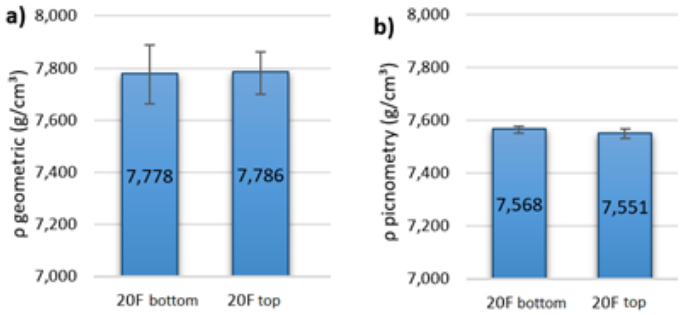


Fig. 3 – Specimens density measurements by a) geometric method and b) pycnometry method

According to both measurements, there was no evidence of settling, since the density was essentially the same at the top and at the bottom of the cylinders, regardless the powder grade or density measurement technique employed. Considering the value of 7.5 g/cm³ for a sample of HK-30 made by casting, and therefore, well-nigh free of pores, the values from figure 3b) corresponding to a little over 100% of theoretical density, indicating the possibility of an experimental error associated with the weight or the dimensions' measurements of the samples. Although it is not expected that a sample manufactured by powder metallurgy could reach a higher density than one produced by casting. It was expected that the values obtained through the geometrical method were slightly lower than the ones measured by pycnometry. This occurs because the volume measured by pycnometry is lower than that obtained with manual techniques (i.e. micrometer), since the last tends to overestimate the dimensions due to the presence of surface roughness along the sample. Even though both techniques show a few imprecisions, the evidence of an excellent densification after sintering is clear. Comparing the pycnometric density values obtained for samples produced with HK-30 20F with the values obtained for the powder, the relative density was 99.96% and 99.73% for the bottom and the top regions, respectively. The microstructure of the specimens produced by HK-30 20F had demonstrated an apparent elimination of porosity, as shown in figure 4a. It also suggests that the grain size is similar with the value found by Ekström and Jonsson (8), obtained by casting, a traditional way of manufacturing. Based on these micrographs. The figure 4b shows that for both samples, the grain size distribution variates mostly from 4,0 to 6,0 G, which means a variation from 0,00806 to 0,00202 mm² of average area, or from 0,0898 to 0,0449 mm of average diameter. Both samples presented similar grain sizes.

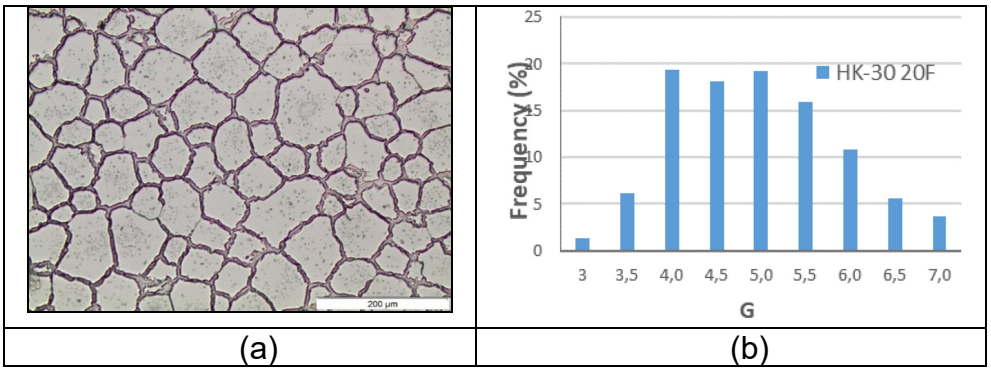


Fig. 4 – (a) Optical microscopy with magnification of 200x, (b) Grain size distribution of samples

SEM image on figure 5 shows the HK-30 20F microstructure, which little porosity.

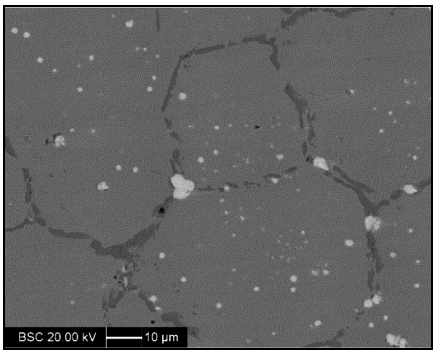
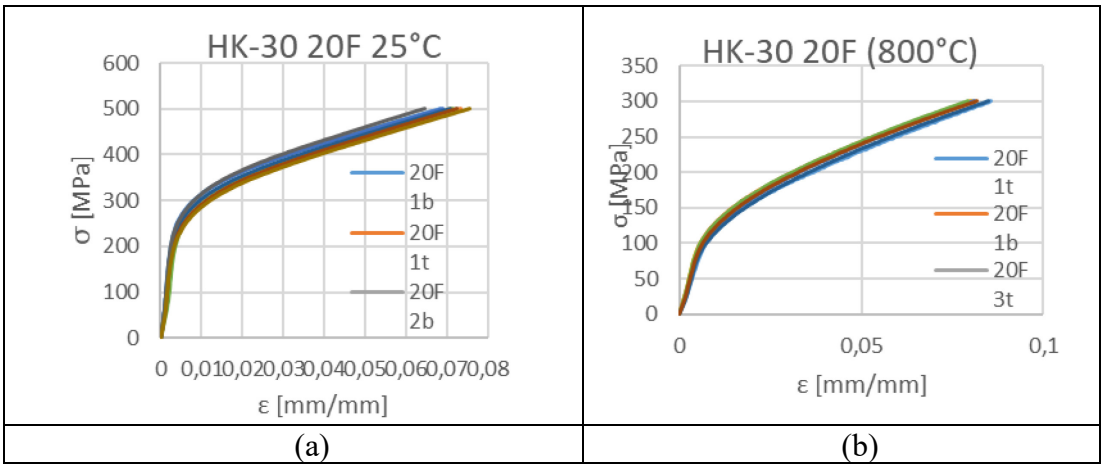


Fig. 5 – SEM microstructures with magnification of 1000x

The results of the room temperature compression tests are shown on figure 6a. The average yield strength measured was of 262 MPa on bottom position and of 246 MPa on top position. Despite this numerical difference between its localization on the cylinder, these results are higher than the standardized values for cast parts made by the same stainless steel, which is defined within the magnitude of 240 MPa (16). Compression tests performed at 800°C temperature have led to an expected decrease on the values, as also found in the literature (8). The same procedure of room temperature tests was held, in order to clearly evaluate the results. Although the load application of a tension test is opposite than in a compression test, there is no difference between its values (20). For this reason, the comparison between the results achieved by this study and the results reached by Ekström and Jonsson was found to be adequate, since there in no standardized value. The results of the 800°C temperature compression tests are shown on figure 6b. The average yield strength measured was of 111 MPa on bottom position and of 117 MPa on top position.



(a) (b)

Fig. 6 – Compression test curves (a) at room temperature and (b) at 800°C temperature

The average results found in literature is in the magnitude of 100 MPa (8). Following the trend of room temperature tests, it where found results about almost 10 percent over yield strength of a cast part of the same stainless steel. Regarding the settling effect, it was successfully shown that there is no any statistical difference between parts taken from the bottom and the top position of the raw cylinder part.

Conclusion

Samples with relatively large dimensions were successfully formed by gelcasting process. All the gelcast samples presented uniform shrinkage, with total removal of binders and good densification, in one single sintering cycle, under vacuum atmosphere and a heating rate of 8.5°C/min. Density results did not present any statistical variation along the settling direction, which confirmed that there was no evidence of settling effects during the forming. Thus, the amount of both dispersant and suspending agent were adequate for the process. Samples produced with HK-30 20F were fully densified. Mechanical properties (yield strength) achieved by this study reached values stated by standard at room temperature and also by previous literature at 800°C. Even though

it was not possible to make any other tests, these preliminary results were sufficient to prove that gelcasting process is feasible for obtaining dense and resistant metal parts. Although it was not possible to perform any other complementary tests, these preliminary results were important to guide the next steps of further work with this process and material.

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