Sintering study of 316L stainless steel reinforced with NbC


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Keywords
316L, sintering, NbC, phase, heating rate and composites.

Abstract
The heating rate is a parameter that can affect the microstructure of the sintered stainless steel reinforced with NbC. An understanding of the effects on the final density can be used to optimize the sintering process. In this work, sintering behavior of stainless steel reinforced with NbC at three different heating rates was investigated. The main aim was to identify the effect of the different heating rate over the microstructure of the sintered samples. Matrixes of pure AISI 316L stainless steel were compacted with addition of up to 3% wt NbC in a cylindrical steel die (Ø = 5.0 mm) at 700 MPa and sintered in an electrical resistance furnace under argon atmosphere. The compacted samples were sintered at temperatures up to 1300°C and then characterized by density measures, dilatometer curves, X-Ray Diffraction, Scanning Electron Microscopy and optical microscopy. The results show the sintered microstructure as a function of heating rates.

Introduction
Metal matrix composites (MMC) appear to be promising option for wear applications. These materials combine a soft metallic matrix with hard ceramic particles that resist to the wear. The ceramic particles that can be used to reinforce MMCs include carbides and oxides. Recently, the use of iron-based alloys or steels as the matrix materials for MMCs has attracted considerable attention from researchers [1,2,3]. Fe-based alloys are by far the most widely used metallic materials because of their low cost and good mechanical properties. Fe-based alloys, particularly austenitic stainless steels, exhibit higher strength, stiffness, and ductility compared to aluminum. Furthermore, a wide range of mechanical properties combined with excellent corrosion resistance make stainless steels very versatile in their applicability. However, austenitic stainless steels have poor sinter ability. Moreover, they have poor wear resistance due to their low hardness. The incorporation of carbide particles into stainless steel matrices can lead to a dramatic improvement in their hardness and wear properties. Recently [4], we have been able to produce very homogeneous compacts with a relative density between 97% and 100%, without exaggerated grain growth, using shorter processing times and lower temperatures than those necessary when conventional sintering techniques are
employed. Moreover, the highest hardness was observed for highly densities compacts. These results were attributed to the incorporation of NbC particles into stainless steel matrices. As far as we know, there are no published studies of the sintering of stainless steel with the addition of NbC at the temperature of 1290°C. The present study investigates the effect of NbC addition on the sintering of stainless steel 316L. We used a dilatometer to produce 316L compacts with and without additives. The compacts were heated up to sintering temperature in three different heating rates: 10°C/min, 20°C/min and 40°C/min. The effect of carbide addition and heating rates on the sintered samples were investigated by optical microscopy, dilatometer curves and density measures.

**Experimental Procedure**

Composites were manufactured by conventional P/M using two different powders: water atomized AISI 316L as metallic matrix supplied by Höganas and NbC as reinforcement synthesized in the UFRN and supplied by Aesar Alpha Johnson Matthey Company. The D$_{50}$ of 316L and NbC supplied by other manufacturer were 95 µm and 5,6 µm respectively. Pure samples and reinforced with 3% of carbides were cold compacted in a cylindrical steel die (Ø = 5,0 mm) at 700 MPa without lubrication, in order to avoid lubricant effects during dilatometer measurements. Sintering was carried out in a (RB 115, BP Engenharia.) dilatometer in commercial argon atmosphere. The compacts were heated to the sintering temperature (1290°C ± 10°C) with different heating rates: 10°C/min, 20°C/min and 40°C/min and held at that temperature for 30 minutes. The samples will be analyzed by density measurements, dilatometer curves and optical microscopy.

**Results and Discussions**

X-ray diffraction patterns of the carbides before mixing with stainless steel 316L are shown in figure 1.

![X-ray diffraction patterns of niobium carbide used in the present work.](image)
As can be observed only characteristic peaks of NbC were identified. Moreover, we also notice a difference in the broadness of the peaks. The carbide produced by UFRN [5] has less intense and wider peaks than that supplied by other manufacturers. This may be due to the refining of the particles. The difference size of particles presented in the figures 2-3 (to see arrows) confirms that possibility. The carbides synthesized by UFRN are finer than those supplied by the others manufacturers. Moreover, comparing figure 2 and figure 3 note that light area in the magnification of 500x when viewed at 5000x in figure 2b is an agglomerate of fine carbides while in figure 3b the own particle

Fig. 2. Scanning electron micrographs of the synthesized NbC by UFRN: (a) Magnification 500x e (b) Magnification 5000x.

Fig. 3. Scanning electron micrographs of the synthesized NbC by other manufacturing: (a) Magnification 500x e (b) Magnification 5000x.

Figures 4 - 6 show dilatometer curves of the sintered samples in three different heating rate. As can be seen the start of shrinkage is anticipated by an increasing heating rate. This suggests the occurrence of some time dependence phenomenon that anticipates shrinkage start. Schatt and Friedrich [6] studied the dislocation activated sintering process for copper, showing that the dislocation density changes considerably in the contact region (boundary grain) as consequence of the produced plastic deformation during the heating. The conclusions of the authors is that when the dislocations density decreases, shrinkage starts, in particular after the recovery of self-activated regions in which the shrinkage rate is correlated to the dislocations density. In the samples of present study to explain the effect of heating rate in the shrinkage initiation is possible to hypothesize the occurrence of dynamic recovery and/or recrystallization which decreases the dislocation density of the self-activated zone, anticipating shrinkage initiation when heating rate is increased.
Figure 7 shows dilatometer curves. The samples were heated to the desired temperature at 40ºC/min. It is observed that in the sample sintered with addition of fine carbides the total shrinkage increases significantly. This is associated probably with pore size filled by carbides during mix with stainless steel. The carbides produced by UFRN are finer (see figure 2-3) and fill smaller pore than the coarse carbide. In the figure 8 this can be seen with details. This figure [7] shows the dispersion of the tantalum carbide (light area) in the metallic matrix of stainless steel (figure 8a). The carbide with melting temperature higher than the temperature used in this experiment (1290ºC) is free and filling blank proportionally to starting particle size or agglomerate (figure 8b). As niobium carbide (NbC) has a high melting temperature, is also waited that sintered microstructure present blank of proportional size to the starting particle size or agglomerate. Therefore, as smaller is the starting particle size or agglomerate (NbC), greater will be the total shrinkages of the samples.
Fig. 8. Scanning electron micrographs of the dispersion TaC in the metallic of stainless steel: (a) Magnification 500x e (b) Magnification 5000x.

Table 1 presents the values of the measured densities for samples heated at different rate up to 1290°C at 30 min. The sintered density increased more for heating rate above than 10°C/min. That happens because normally a higher heating rate (>10°C/min) induces large thermal stresses that accentuate sintering beyond that found isothermal sintering [8]. Moreover, higher heating rate tends to increase the driving force for the densification mechanisms at higher temperature.

Comparing the values of densities presented in the table 1 (20°C/min and 40°C/min) to that respective shown in the figures 4-6 is noticed through one side the need of increase of time and/or sintering temperature for obtaining of a larger densification in the samples. But by other according to densities values presented for sample B17, how this modifications affect in the increase of relative density limited in 96% or in the anomalous grain growth.

Table 1. Results of density obtained for steel 316L samples with and without addition of niobium carbide (3% wt) sintered in a dilatometer at 1290°C during 30 min.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Heating Rate °C/min</th>
<th>Density Sintered (g/cm³)</th>
<th>Relative (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B16</td>
<td>10</td>
<td>7.27 ± 0.07</td>
<td>91.2</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>7.53 ± 0.08</td>
<td>94.5</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>7.61 ± 0.08</td>
<td>95.5</td>
</tr>
<tr>
<td>B17</td>
<td>10</td>
<td>7.41 ± 0.08</td>
<td>93.0</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>7.69 ± 0.08</td>
<td>96.5</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>7.58 ± 0.07</td>
<td>95.1</td>
</tr>
<tr>
<td>B18</td>
<td>10</td>
<td>7.38 ± 0.07</td>
<td>92.6</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>7.57 ± 0.09</td>
<td>95.0</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>7.60 ± 0.07</td>
<td>95.4</td>
</tr>
</tbody>
</table>

Obs: B16 = 316L; B17 = 316L + NbC (J. Matthey) and B18 = 316L + NbC (UFRN)

Conclusions
1. The increase of the heating rate reduces the density of dislocations in the boundary grain and anticipates shrinkage initiation;
2. Higher heating rate (>20°C/min) induces large thermal stresses that accentuate sintering beyond that found isothermal sintering and tends to increase the driving force for the densification mechanisms at the higher temperature;
3. The size of carbide, particle or agglomerate influence significantly in total shrinkage of the samples. Those reinforced with fine carbides they presented a larger total shrinkage.
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References