Borax as flux on sintering of iron Ancor Steel 1000® under glow discharge

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Abstract. This work studies the flux effect of borax (di sodium tetraborate decahydrate) on sintering of iron Ancor Steel 1000® in abnormal glow discharge. The incidence of the percentage by weight of borax and the sintering temperature in the process were observed. Samples of powder metallurgical iron were prepared with proportions of 0.50%, 2.0%, 4.0% and 6.0% by weight of borax using the procedures of powder metallurgy. The samples were sintered at 800 and 1100°C for 30min, by glow discharge at low pressure in a reducing atmosphere composed of 20% H₂+80% Ar. The samples in compact green-state were analyzed by TGA-DSC to determine the fusion process and mass loss during sintering. The analysis of microhardness and density, shows that at a sintering temperature of 800°C the sample density decreases and the sample microhardness increases with respect to sintered samples without borax. Sintered samples were analysed by DRX showing the absence of precipitates.

1. Introduction

Powder metallurgy is one of the processes used in industry for obtain chemical compositions in precise ratios in the manufacture of metallic materials [1] through the powder mixture which is subsequently compacted and sintered. The Sodium tetraborate pentahydrate (borax), with chemical formula: B₄O₇Na₂⋅10H₂O, is used in this study as metallurgical powder to analyse its effect as a lubricant and as a flux during sintering iron Ancor Steel 1000®. The fluxes have the property of lower the melting point of a mixture, and are widely used in welding. The Borax dissolves iron, tin, silver and nickel oxides [2], which can enhance the formation of metallurgical bonds in the sintering process. The oxide can be formed in the metal powder during the steps of: mixing, compaction and sintering.

2. Methodology

The powders Ancor Steel 1000® and Borax were weighed to have the desired amount of weight percentage; this process was performed using a microbalance. The Table 1 shows chemical composition of Ancor Steel 1000®. Metallurgical powders were mixed for 30 minutes and compacted in a steel matrix of diameter 10mm, 700MPa [3,6]. Sintering was performed in a plasma reactor by glow discharge with reductive gaseous atmosphere consisting of the mixture: 20% H₂+80% Ar for 30min with a pressure of 3Torr.

Sintering temperatures were 800°C and 1100°C that were achieved at a heating rate of 77°C/min. The porosity was analysed by micrographs at 100x magnification. DRX analysis was performed to detect the presence of precipitates. Density analysis was performed using a caliper and a microbalance.
Microhardness measurement was obtained using a Vickers microhardness tester type, which was calibrated with a load of 100g for a time of 15s according to ASTM E384 [7].

![Table 1. Chemical composition of Ancor Steel 1000®.](image1)

| Element       | Wt (%) | Element       | Wt (%) |
|---------------|--------|---------------|--------|
| Carbon        | 0.004  | Manganese     | 0.190  |
| Sulfur        | 0.016  | Copper        | 0.180  |
| Oxygen        | 0.120  | Nickel        | 0.080  |
| Phosphorus    | 0.008  | Molybdenum    | 0.050  |
| Silicon       | 0.003  | Chromium      | 0.008  |

3. Results

3.1. Compaction
The compacting step of Ancor Steel 1000 samples with different borax compositions was performed successfully; the samples were removed from the steel matrix without difficulty. This allows us to recommend the use of borax as a lubricant during the compaction step of powder metallurgical materials.

3.2. Thermogravimetric analysis
DSC-TGA analysis of Fe-6% mixture of borax are shown in Figure 1. Thermal processes show a dehydration process at 143.82° C due to the presence of borax and borax melting occurs at 745.16° C. Thermal dehydration process is presented between 140°C and 153°C, and causes a mass loss of 0.378mg.

The allotropic transformations of iron are also notorious, the allotrope \( \beta \) (Beta) with lattice parameter of 2.90Å and BCC crystal lattice is presented to 763.22°C and allotrope \( \gamma \) (Gamma), with FCC structure and 3.6Å lattice parameter, is presented around of 914.06°C.

![Figure 1. Thermogram of Fe-6% Borax.](image2)

3.3. Porosity
Samples of porosity increases with its borax content at 800°C sintering as shows in Figure 2. This increases because the regions occupied by borax during the forming step are then evacuated during the sintering step. Image analysis was performed at the micrographs in Figure 2 to quantify the porosity of the samples as a borax composition function, those results are summarized in Table 2.
3.4. Density
The green and sintered samples show that density decreases with increasing addition of borax. Table 3 shows that the lowest density values were obtained with a percentage of 6% by weight of borax in both groups of samples.

| Borax (% peso) | Porosity (%) |
|----------------|--------------|
| 0.5            | 7.88         |
| 2.0            | 8.65         |
| 4.0            | 9.33         |
| 6.0            | 13.10        |

Table 3. Iron-Borax samples density.

| % of Borax | Density (g/cm³) | Density (g/cm³) |
|------------|----------------|----------------|
|            | Green state    | Sintered to 800°C | Green state | Sintered to 1100°C |
| 0.5        | 6.87           | 6.88            | 6.85        | 6.87            |
| 2.0        | 6.51           | 6.51            | 6.65        | 6.63            |
| 4.0        | 6.21           | 6.19            | 6.35        | 6.16            |
| 6.0        | 5.88           | 5.87            | 6.10        | 6.01            |

The sintered density should increase with respect to samples in green, but due to loss of mass during the heating step caused by borax output, sample density value is affected. The bombardment of the samples at the cathode of the glow discharge, also contributes to this mass loss.

3.5. Microhardness
Microhardness values are recorded in Table 4. Sintered samples at 800°C shown an increase in microhardness; it shows that the concentration of 2%, by weight of borax, has a microhardness value increase twice when sintered at 800°C. On the other hand, samples of 2.0%, 4.0% and 6.0% by weight of borax, sintered at 1100°C for 30 minutes, have a lower microhardness value than those sintered at 800°C. This is due to the rapid exit of borax during the heating step to reach the 1100°C temperature sintering. Because of flux properties of the Borax, the sintering is enhanced in its presence and then the mechanical properties of the material.
Table 4. Microhardness sintered 30min.

| % of Borax | HV 0.1 Microhardness |
|------------|----------------------|
|            | Sintering to 800°C | Sintering to 1100°C |
| 0.5        | 77.17                | 83.80                |
| 2.0        | 115.26               | 72.68                |
| 4.0        | 110.87               | 85.10                |
| 6.0        | 113.47               | 91.70                |

3.6. Analysis of DRX

DRX analysis carried out on the samples of 2.0%, 4.0%, 6.0% by weight of borax at 1100°C and the samples with 2.0% of borax sintering at 800°C, do not exhibit the formation of precipitates. The analysis only shows the characteristic peaks of ferritic iron. Figure 3 shows that DRX analysis is common to all samples.

![Figure 3. Analysis of DRX.](image)

4. Conclusions

Ancon steel 1000® iron samples with 0.50%, 2.0%, 4.0% and 6.0% by weight of borax were manufactured using the standard procedure of the powder metallurgy. The sintering step was carried out in the cathode of abnormal glow discharge in a reducing atmosphere composed of 80% Ar and 20% H₂. The application of borax as a lubricant material during the forming step and application of the fluxing properties during sintering at 800°C was verified successfully.

The density obtained at 800°C sintering temperature is similar to that achieved at a sintering temperature of 1100°C. Samples sintered at 800°C, have higher microhardness than samples sintered at 1100°C. This is due to the oxide removing and flux properties of the borax at this temperature.

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