Production of Nanocrystalline Powder of Fe-Si by Mechanical Alloying

Fidel Romel M. Espinoza and Lírio Schaeffer
Metal Forming Laboratory, Universidade Federal do Rio Grande do Sul/UFRGS, Porto Alegre - Brazil
E-mail: fidel@vortex.ufrgs.br, and schaefer@vortex.ufrgs.br

Keywords: Soft magnetic; Nanocrystalline; Mechanical alloying

ABSTRACT
This work has as objective the study of the nanocrystalline Fe-Si sintered soft magnets obtained by Powder Metallurgy. We are use the technique of Mechanical AlloYing in the processing of the powder.
These materials are use for electric and magnetic applications, having as base the development of an alternative process to the conventional processes of production of this material.
The used composition in Mechanical AlloYing process was Fe-3%Si processed starting from elementary powders of iron and Fe-17%Si pré-alloy, during 0.5, 1, 2, 4, 6, 10, 14 and 20 hours of milling. The structure of the obtained Fe-Si alloys is the same as that of bcc α-Fe. The final powder obtained mainly consist of particle with a grain size of from a few hundred nanometers.

Introduction
Iron-silicon alloys are well know excellent soft magnetic materials [1, 2, 3]. The production of a composite by adding a hard phase to a ferritic matrix involves a dispersion stage, commonly accomplished by attrition or ball milling. During this stage, a uniform powder mixture is formed. In some cases, milling also serves as a means of breaking down the often brittle hard particles, thus assisting in the adjustment of particle size distribution [4, 5]. However, milling can also lead to hardening and to changes in the morphology and size of the particles, resulting in improved specific area [6, 7, 8].
We know that nanocrystalline materials have different physical properties from noncrystalline, policrystalline and single-crystalline materials; we also know that mechanical alloying can conventionally, economically and completely synthesize nanocrystalline binary alloys; therefore, in this work, we report the preparation of nanocrystalline Fe-Si alloys using the mechanical alloying method.

Experimental Procedure
The process of Fe-3%Si magnetic materials obtaining involves the following stages: mixing, milling, compactation and sintering.
Mixing
The obtaining of Fe-3%Si alloy will be made from elementary powder of Fe and alloy Fe-17%Si powder mixed with lubricant, to obtained a mixing as homogenous as possible.

Milling
Then the load is submitted to a milling process in a high energy mill for 0.5, 1, 2, 4, 6, 10, 14 and 20 hours, using a load ratio of 10. The tests are made under a protector argon atmosphere.

Pressing
The objective here is to conform the powder in the projected way, taking into account the possible dimensional variations that can occur during the sintering. For this stage, we will use the floating matrix compaction system. The pressure used was 200 MPa.

Sintering
The sintering temperature was almost 1220°C, followed by a slow cooling into the furnace. The atmosphere used in the sintering was dissociated ammonia.

Result and discussion
The X-ray diffraction pattern for Fe-3%Si powder before and after 2 and 20 h ball milling are shown in Figures 1 and 2, respectively. Analysis of Figures 1 and 2 reveals that, after 20 h ball milling, all the Fe-17%Si atoms substitute for Fe in the bcc Fe matrix and form Fe-3%Si alloy. The initially sharp diffraction lines are considerably broadened after 20 h ball milling due to refinement of the crystalline structure size and increase of atomic level strains. In this work, we used the Sherrer formula for the estimation of the grain size.

Fig.1 X-ray diffraction patterns of Fe-3%Si powder after 2 h ball milling.
Accordingly, the X-ray diffraction data is analyzed for Fe-3%Si alloy. The average grain size is about 35-40 nm for Fe-3%Si alloy after 20 h ball milling.

Figures 3 and 4 illustrate the change in the morphology of Fe-3%Si particles as a function of milling time (2 and 20 h). After milling for 2h, irregular plateletlike particles of 30 μm average size were formed. A few cracks and agglomerates were also noticed. Increasing the milling time to 4h considerably increased the number the agglomerates. Although the average particles size reduction was not significant, considerable cracking took place, corresponding to a refinement of the remaining larger particles. Increasing the milling time to 6, 10, 14 and 20 h resulted in a change in the morphology of the particles platelets to foils, accompanied by a substantial reduction in particle size. Figure 5 shows microstructure of Fe-3%Si powder after 20 h ball milling and Figure 6 shows the particle medium size of Fe-Si powder as a function of milling time.
From SEM analysis, we know that Fe-3%Si alloys after 20 h milling are mainly composed of irregular particles with a grain size of from a few nanometers to several μm; from X-ray diffraction analysis, we know that the average grain size of the obtained Fe-3%Si alloys is about 35-40 nm. Therefore, using the mechanical alloying process, we prepared nanocrystalline alloy, the microstructure of which is characteristic of that found for nanocrystalline metals produced by other methods.

Results to the hysteresis loops are given in table 1. The value of maximum saturation induction was of 130 emu/g, what indicates that the material processed by M. A. had a larger saturation induction. The Fe-3%Si soft magnetic sintered had a coercivity of about 120 to 140 A/m.

<table>
<thead>
<tr>
<th>Process</th>
<th>Material</th>
<th>Coercive Force (emu)</th>
<th>Saturation Induction (Am)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M. A.</td>
<td>Fe-3%Si</td>
<td>130</td>
<td>130</td>
</tr>
</tbody>
</table>

Fig. 4 SEM showing particle size and morphology after milling for 20 h.

Fig. 5 Microstructure of Fe-3%Si powder after 20 h ball milling.
Conclusions
- This is work, we have prepared nanocrystalline Fe-3%Si alloys by high-energy ball milling.
- The structure of the obtained nanocrystalline Fe-Si alloy is the same as that of bcc $\alpha$-Fe.
- The obtained Fe-3%Si alloy mainly consist of particle with a grain size of from a few nanometers to several $\mu$m, and are composed of grains with a nanocrystalline size about 35-40 nm an almost random orientation with respect to each other.
- As the milling time is increased, it is produced a little increment of the material resistance that was verified by the increment of hardness and the difficulty of compactation, that was more notorious from a milling time of 2 hours.
- With the increment of the milling time, there was a variation of the size and the form of particle that diminished by aumet time milling.
- According to metallographic test, it was appreciated a diminishing of grain size with an increment of milling time.
- Metallographically was also determined a higher homogeneity in micro-structure with an increament of milling time.
- The values of the specific saturation magnetization are slightly high than those of the corresponding polycrystalline Fe-Si alloys and the values of coercive force $H_c$ was too slightly more high.

Acknowledgement
The authors would like to acknowledge CAPES for financial support of this research project.

References