THE CHARACTERIZATION OF MnS PARTICLES PRECIPITATION IN THE Fe-3%Si STEEL DURING THERMOMECHANICAL PROCESSING.

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Abstract
Grain-oriented electrical steel Fe-3%Si was heated and mechanically conformed by hot process. The analysis of MnS particles precipitation has been made with samples processed at different conditions. The specimens were initially heated at 1573K for 1800 seconds for solution treatment, after this, they were cooled down to 1273 K, compression of 50% is applied during the first hold time (10 seconds), then the compression of 30% is applied (second holding time : 26, 110, 345 and 410 seconds), then water quenched. Grain size characterization was carried out using OM. The study of precipitation has been carried out on the surface and the center of each sample. For TEM characterization of MnS particles, an extraction replica and thin foils techniques have been used. An image analyzer measured the particle diameter and the data were microcomputer processed. Optical micrographs showed that grain size decreases with the second holding time process. Hot compression process with two holding time provides more dislocation and defects increasing the possibility of MnS particles and providing higher MnS particle density.

Keywords: hot deformation, electrical steel, compression process, MnS precipitation, TEM characterization.

1.INTRODUCTION.

MnS particles are used in Fe-3%Si steels as inhibitors of secondary recrystallization process with the aim to obtain the development of Goss orientation grains(1). The retardation effect depends on their total volume fraction and dispersion.

The dispersion parameters depend both on chemical composition and on the history of thermal deformation of the steel. Recently, some authors have investigated the behavior of particle dissolution and MnS precipitation after thermomechanical treatments(2,5), using hot compression or creep technique or torsional simulation process tests. Many processing factors are already optimized empirically, but the understanding of MnS precipitation will give us a further possibility of improvement(2).

Grain boundary migration is the basic mechanism of recrystallization and grain growth in the solid state that represents an important part of the processing materials.

To explain the structural changes of materials during its thermomechanical treatment, grain boundary migration and related phenomena have been extensively studied during the past decades(6). One of the materials, which in the grain boundary have been often investigated, is Fe-3%Si alloy. The large interest in the study of dynamic behavior of grain boundaries in this system was evoked by its favorable magnetic properties and, in consequence, by its use for transformer cores(6).

In previous investigation, a recently developed hot compression process technique was employed to detect the occurrence of MnS precipitation at hot working temperature(7). The influence of the holding time in the MnS precipitation at high temperature was investigated in the present study.

2.EXPERIMENTAL PROCEDURE.

The Fe-3%Si alloy has been provided by Brazilian steel plant industry. The compression test was made in the Mechanical Properties Laboratory-Metallurgical Engineering Faculty of Universidade de Minas Gerais (Belo Horizonte, M.G., Brazil). The chemical composition of alloy Fe-3%Si is given in Table 1.

Table 1 - Chemical Composition of Fe-3%Si (in wt%)

Element	wt(%)	Element	wt(%)
Carbon	0.030	Nickel	0.02
Manganese	0.060	Molybdenum	0.006
Silicon	3.12	Aluminum	0.002
Phosphorus	0.012	Titanium	0.002
Sulfur	0.023	Nitrogen	0.0028
Chromium	0.024	Iron	96.78

Specimens were studied in two regions, surface and the center of each sample and they were heated at 1573K for 1800s for solution treatment. Thermomechanical treatment was performed in halogen lamp furnace with low thermal inertia, water refrigeration and compression system. This equipment has four halogen lamps with 8kW-power operation. After solution treatment specimens were cooled down with rate cooling of 1.5K/s to 1273K hold for 10 seconds when the compression of 50% is applied. After this holding time the compression of 30% is applied during 26, 110, 345 and 410 seconds (second holding time) and then quenched in water.

The observation of MnS particles was made by transmission electron microscopy (JEOL-JEM-200C and by Philips CM-200), using an extraction replica and thin foils techniques. The freshly precipitated particles in the electrical steel were identified by EDX examination spectrum and it was acquired on a carbon extraction replica. All of micrographs acquired in this way have a magnification of 60,000 times, and then the sizes and densities of particles were measured using an auxiliary image analyzer and the data were compiled and elaborated in microcomputer electronic table.

3. EXPERIMENTAL RESULTS

3.1.Optical Observations.

Effect of deformation temperature on the microstructures was observed at first. Table 2 shows the grain size in the two studies regions. The grain size decreases when second holding time increases on the surface while the grain size increases when the second holding time increases was observed for the center of the sample. Careful comparison of the two regions reveals that the grain size increases from the surface to the center of the sample.

Table 2- Mean Grain Size.

Temperature (K)	Analyzed Region	End Holding Time (s)	Mean Grain Size (μm)
1273	Surface	(10+26)	2162
		(10+110)	1877
		(10+345)	1238
		(10+410)	1174
	Center of the Sample	(10+26)	2884
		(10+110)	1969
		(10+345)	1383
		(10+410)	1187

3.2.Transmission Electron Microscopy Observations.

Figure 1 shows the result of EDX analysis reveals the strong Mn and S peaks associated with most of particles formed in this steel. It should be pointed out that Si peaks visible are due to this elements presence in the matrix and Fe peaks are from matrix itself. The copper peaks are due to the sample holder.

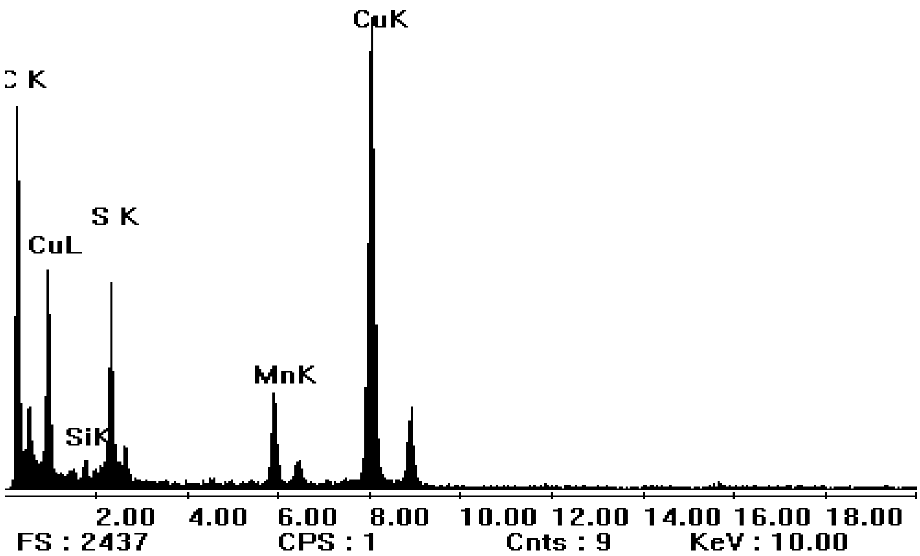

Fig.1. MnS and S peaks obtained by EDX analysis.

One of the microscopic photographs obtained after compression test at 1273K for 26, 110, 345 and 410 seconds on the surface and the center of the sample is shown at right of Figures 2 and 3, while the size distribution associated with these samples is displayed at the left of the each figure.

It can see from the diagram of figure 2 that precipitates size ranges from about 9 to 13nm on the surface region.

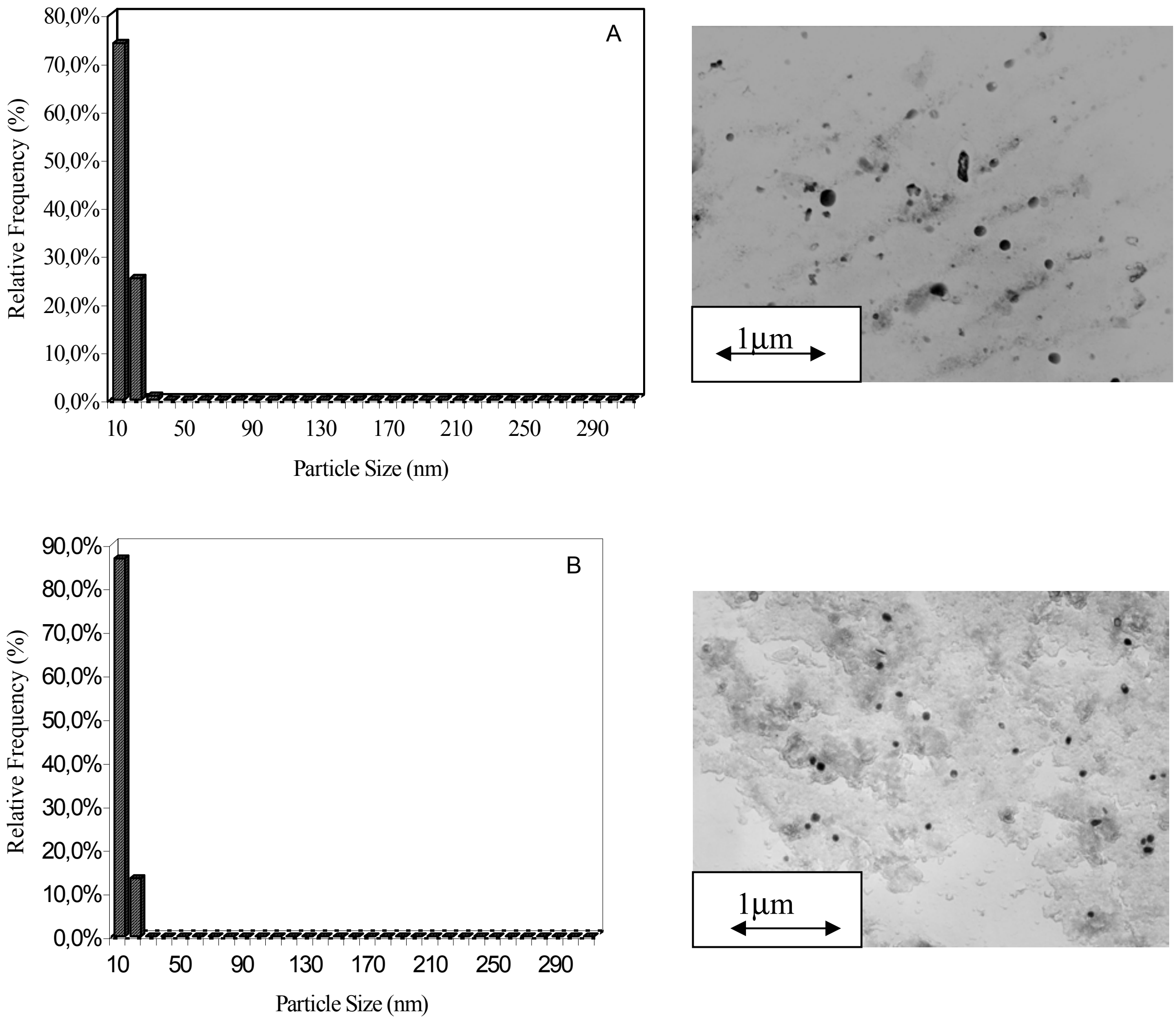

Fig.2. MnS precipitates and their particle size distribution in samples at 1273K, on the surface region. a) (10+26s), b)(10+448s)

An examination of microscopy photographs, figure 3 reveals that in the center of the sample the diameter particle ranges from 19 to 23nm.

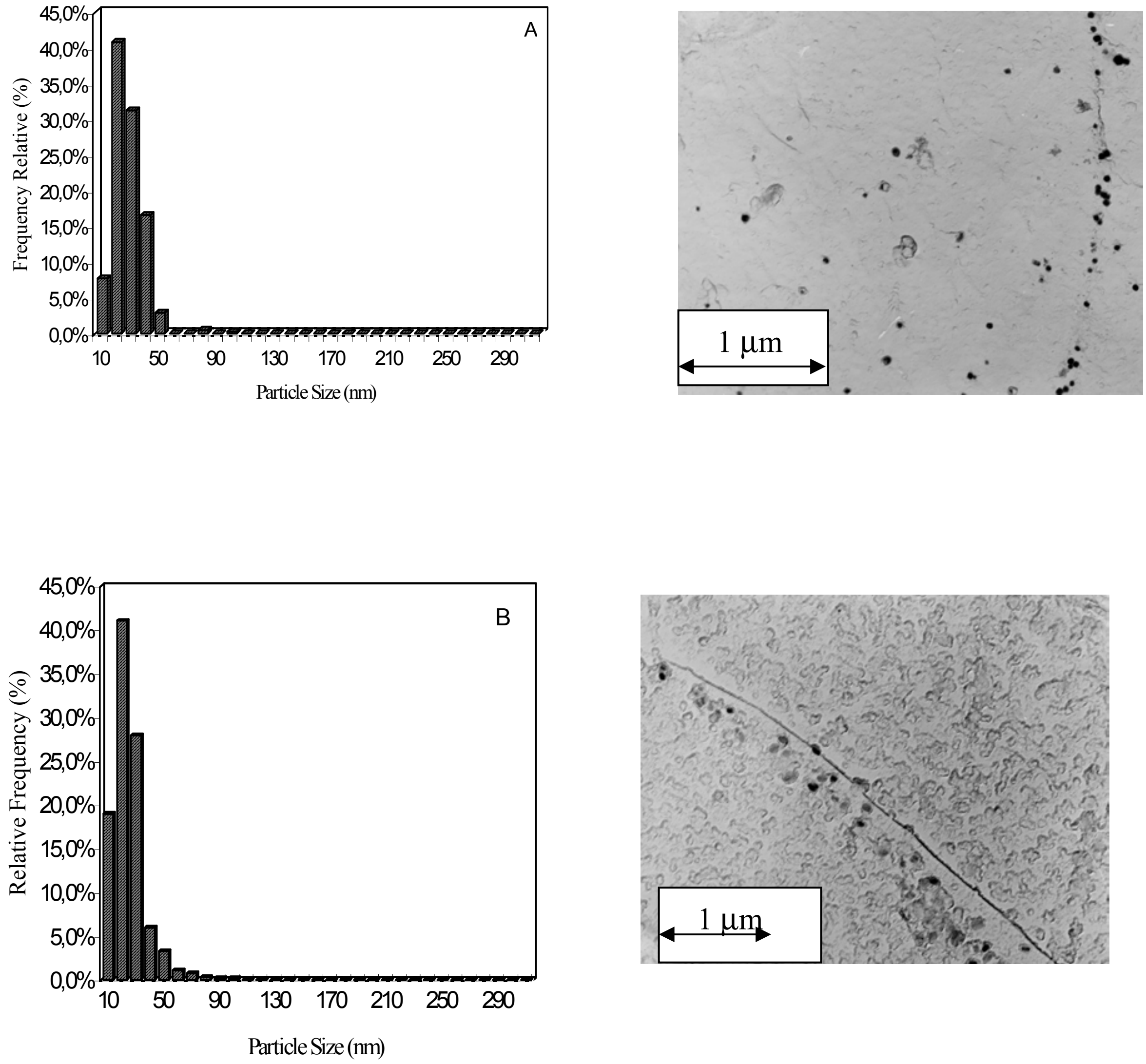

Fig.3. MnS precipitates and their particle size distribution in samples at 1273K, in the center of the sample. a) (10+26s), b) (10+448s)

An examination of Table 3 reveals that the mean particle size is a function of the second holding time. It suggests that increasing second holding time, the hot compression process generates dislocation network producing new subboundaries. Therefore, the particles assemble in the smaller particles that result in grain inhibition on the surface[(8).]

Table 3 Mean Particle Size (nm)

Temperature (K)	Analyzed Region	End Holding Time (s)	Mean Particle Size (nm)
1273	Surface	(10+26)	9.0
		(10+110)	9.0
		(10+345)	9.5
		(10+410)	13.0
	Center of the Sample	(10+26)	23.0
		(10+110)	22.0
		(10+345)	20.0
		(10+410)	19.0

At 1273K was noticed that the particle size decreases with increasing second holding time. It suggests that there are two mechanisms that can be explained the kinetics precipitation of MnS particles.

1.At high temperatures, the deformation provides cellular arrangement that follows the subboundaries formation. During the cellular arrangement increases the possibility of particles precipitates which are inhibitors of subboundaries and hence, this process produces smaller size grains.

2.For the hot compression with 50% and 30% deformation, it can produce "dead zone area"[(9)] and in the center of the sample, for the hot compression process, the mean particle size decreases with increasing the end holding time, since MnS is the precipitates having a tendency of fussion under stress, the decrease of particle size with increasing holding time is possibly explained as the result of increase in the number of newly precipitated-small particles [(2,3).] This result shows that newly precipitation process prevails over growth or Ostwald ripening of precipitates[(5)]; this provides less particles precipitation and the grain growth faster.

4.CONCLUSIONS.

1. For the temperature of 1273K, the grain size decreases with increasing second holding time on the surface and the center of the sample. By TEM observation, the particle size increases from surface to the center of the sample. It suggests, due to

"dead zone area" a fewer deformation, consequently less particles precipitation which increases the final grain size.

2. According to the size particle for the temperature and dwell time control of hot compression process, MnS precipitates showed a remarkable homogeneity all over the sample.

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