COMPARISON BETWEEN PRECIPITATION KINETICS OF MANGANESE SULFIDE DURING HOT COMPRESSION PROCESS WITH ONE AND TWO HOLDING TIME IN GRAIN-ORIENTED ELECTRICAL STEEL.

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Abstract. The aim of this work is the study of manganese sulfide particles precipitation (Fe-3%Si) after one and two holding time during hot compression process. Samples were heated at 1573K for 1800s. After the specimens were cooled down to 1373 and 1173K, hold for 32, 60 and 338s and the compression of 50% is applied during the holding time and then water quenched, in the process with two holding time, the first holding time is 60s (50% is applied), then the compression of 30% is applied (second holding time: 32, 60 and 338s), then water quenched. Grain size characterization was carried out using OM. The study of comparison precipitation has been carried out on the surface and the center of each sample. For TEM characterization of MnS particles an extraction replica technique has been used. Image analyzer measured the particle diameter. Optical micrographs showed that the grain size decreases with the second holding time process for the two temperatures on the two studied regions. Hot compression process with two holding time provides more dislocation and defects increasing the possibility of MnS particles precipitate and providing higher MnS particle density.

1.INTRODUCTION.

Soft magnetic materials have been used perform a wide variety of magnetic functions. Some applications demand high permeability as core materials in electrical machinery, such as motors, generators and transformers; others emphasize low loss at high frequencies, thus necessitating high resistive; etc. To meet the divergent requirements of applications, numerous commercial products in the form of complex alloys or mixed ferrites were developed by metallurgical methods. The development of soft magnetic for industrial applications started long before the turn of the century. Some authors choose 1885 as the year when the history of commercial soft magnetic materials formally began. The metallurgists have played an important and sometimes essential role in development of industrial magnetic product [1].
In this work was carried out a study about the precipitation of MnS particles after one and two holding time during hot compression process.

2. EXPERIMENTAL PROCEDURE.

The Fe-3%Si alloy has been provided by Brazilian steel plant industry. This material is normally mechanically conformed by Steckel hot rolling processing. In our work, to simulate the former process, the compression test was made in the Mechanical Properties Laboratory-Metallurgical Engineering Faculty of Universidade Federal de Minas Gerais (Belo Horizonte, M.G., Brazil). The chemical composition of alloy Fe-3%Si is given in Table 1.

In order to make a comparison between precipitation kinetics of manganese sulfide during hot compression process with one and two holding time, specimens were studied in two regions, surface and the center of each sample.

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		

Cylindrical specimens with the diameter of 10mm and the height of 15mm were machined from a hot-rolled sheet bar. Thermo-mechanical treatment was performed in halogen lamp furnace with thermal inertia, water refrigeration and compression system. This equipment has four halogen lamps with 8kW power operation. The specimens were heated at 1573K for 1800s for solution treatment. After the specimens were cooled down with rate cooling of 1.5K/s to 1375 and 1173K, hold for 32, 60 and 338 seconds and the compression of 50% is applied during the holding time and then water quenched. The same procedure was used in the process with two holding time, in this case the first holding time is 60 seconds when the compression of 50% is applied, then the compression of 30% is applied during the second holding time (32, 60 and 338 seconds), then water quenched. For the characterization of grain size, the samples were chemically etched by 5% Nital and the observation and measurement were made by optical microscopy. The observation of MnS particles was made by transmission electron microscopy. For TEM characterization of MnS particles, an extraction replica was prepared and observed by JEOL-JEM-200C and by Philips CM-200. The freshly MnS particles in electrical steel were identified by EDX examination spectrum and it was acquired on carbon extraction replica. The sizes and densities of particles were measured in TEM micrographs (magnification of 60,000 times) using an auxiliary image analyzer and the data were compiled and elaborated in microcomputer electronic table.

3.RESULTS AND DISCUSSION.

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 the visible Si and Fe peaks are from the matrix. One of the electron microscopic photographs obtained after compression test at 1173K for 32, 60, and 338 seconds on the surface of the sample is shown at the right of figure 1, while the size distribution associated with these samples is displayed at the left side of the each figure. It can seen from the diagram of figure 1, that precipitates size ranges from about 90 to100nm on the surface region while in the center of the sample the precipitates size ranges from about 27 to 71nm. It can see from the diagram of figure 2 that the particle size has broadened to cover range from 16 to 29nm at 1173K, on the surface of sample with two holding time. An examination of the electron micrographs, reveals that in the center of sample, the diameter ranges from 8 to 20nm at 1173K, with two holding times. An examination of table 2 reveals that mean particle size is minor in the process with two holding time, (on the surface and in the center of the sample). In the center of the sample, the mean particle size decreases with increasing the holding time (one and two holding time), since MnS isn't the precipitates having a tendency of fission stress, the decrease of particle size with increasing holding time is possibly explained as the result of increase in the number of newly precipitated particles [2,3].

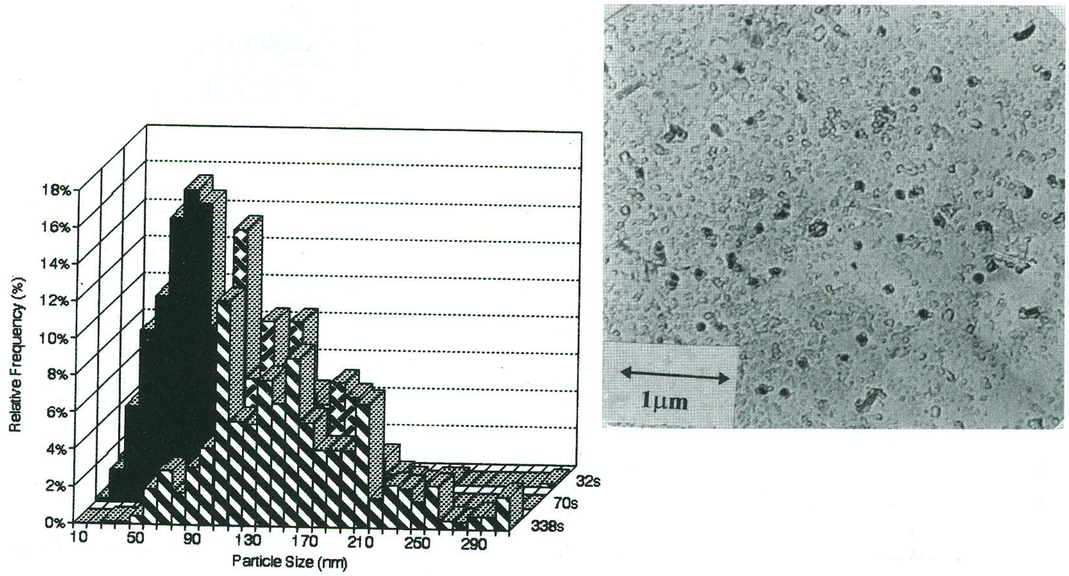

Fig.1.MnS precipitates and their particle size distribution in the sample at 1173K, one holding time, on the surface region.

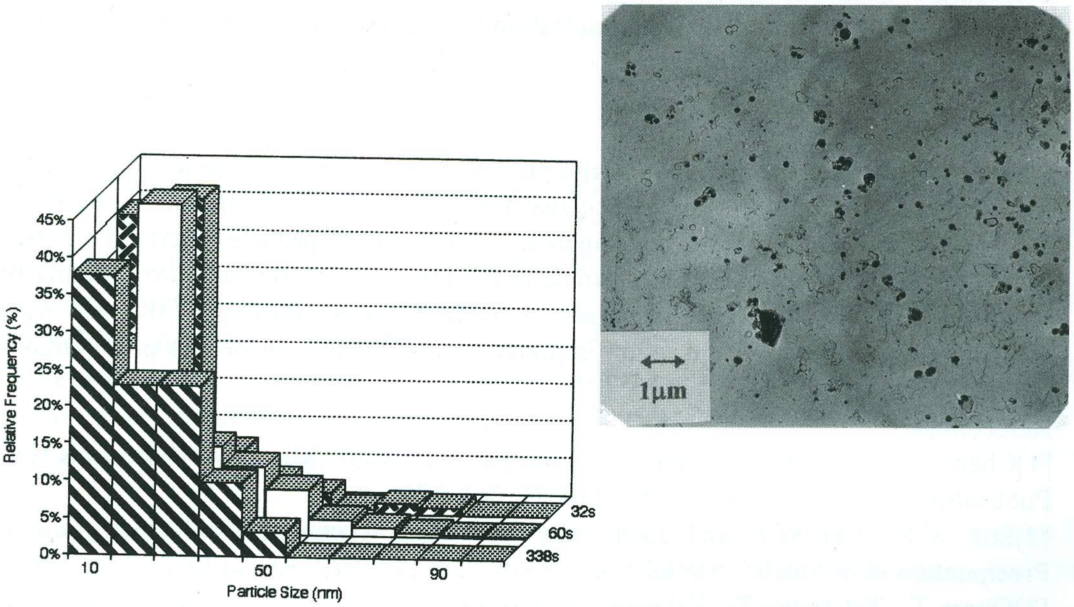

Fig.2.MnS precipitates and their particle size distribution in the sample at 1173K, two holding time, on the surface region.

Table 2.Mean Particle Size

Temperature (K)	Analyzed Region	One Holding Time (s)	Mean Particle Size (nm)	End Holding Time (s)	Mean Particle Size (nm)
1173	Surface	32	90	(60+23)	16
		60	70	(60+60)	20
		338	100	(60+338)	29
	Center of the Sample	32	71	(60+23)	8
		60	60	(60+60)	20.5
		338	27	(60+338)	19
1373	Surface	32	38	(60+23)	13
		60	14	(60+60)	11.5
		338	14	(60+338)	13
	Center of the Sample	32	36	(60+23)	18
		60	15	(60+60)	18
		338	10	(60+338)	6.5

This result shows that newly precipitation process prevails over growth or Ostwald ripening of precipitated [4]. However hot compression process with two holding time provides more dislocation and defects on the center of the sample increasing the possibility of MnS particle density [3]. On the other hand, nucleation is preferred at high temperature (1373K) and nucleation occurs preferentially at grain boundaries. Dislocations provide more sites for nucleation than grain boundaries; hence their density increases with temperatures [4].

4.CONCLUSIONS.

1-The mean particle size is minor in the process with two holding time, (on the surface and in the center of the sample) because there is a newly precipitation process that prevails over growth or Ostwald ripening. 2-The mean particle decreases from the surface to the center region of the sample in the process with one and two holding time, then, the number of particle increases. 3-At high temperature (1373K) the particle density increases because nucleation is preferred and nucleation occurs preferentially at grain boundaries.

References.

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