

# KINETICS OF THE MANGANESE SULFIDE IN SILICON - IRON STEEL.

COLEÇÃO PTC  
DEVOLVER AO BALCÃO DE EMPRÉSTIMO

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## Abstract.

Grain-oriented electrical Si steel was heat treated and mechanically conformed by Steckel hot roll processing. The grain growth and the MnS precipitates were investigated after heat treatment at temperatures 1573 and 1623K during 300,600, 900 and 1200 seconds. The grain size covers the range from 0.3 to 0.5mm. The diameter of MnS precipitates showed values ranging from 10 to 30nm. The analysis of MnS precipitates distribution after the hot roll process at lower temperatures (1448, 1458, 1493 and 1503K) showed that the diameter of the precipitates, covered the range from 30 to 90nm. Characterization was made by transmission electron microscopy utilizing a precipitate replica and thin foil techniques.

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## **1-Introduction**

The properties of grain-oriented electrical steel have been continually improved since commercially produced since 1945. The products are widely used as core materials in electrical machinery, such as motors, generators and transformers [1].

One of the most important metallurgical basis for producing grain oriented Si steel is controlling the dissolution and precipitation behavior of MnS precipitate. As it is well known, MnS particles have been used as grain growth inhibitor [2-3].

Recently, some authors have investigated the behavior of particle dissolution and MnS precipitation after thermo-mechanical treatment [3-6], using hot compression or creep technique or torsional simulation process tests. Many processing factors are already optimized empirically, but understanding precisely the precipitation and dissolution phenomena will give us a further possibility of improvement [4].

In this work the precipitation and dissolution of MnS particles in Fe-3%Si after solution treatment and hot rolling process.

## 2-Experimental Procedure

The Fe-3%Si alloy has been provided by a brazilian steel plant industry (Acesita). 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	0.029	Nickel	0.014
Manganese	0.059	Molybdenum	0.006
Silicon	3.19	Aluminum	0.028
Phosphorus	0.017	Titanium	0.021
Sulfur	0.022	Tin	0.021
Chromium	0.024	Nitrogen	0.028

In order to study the dissolution behavior of MnS particles, specimens with dimensions of 0.015 x 0.015 x 0.0020m were heated in an argon gas atmosphere at 1573 and 1623K for 300, 600, 900 and

1200 seconds and quenched in water. The samples were chemical etched by 8% Nital. The characterization of grain size was made by optical microscopy and transmission electron microscopy for the observation of MnS particles was used. The precipitation particles were studied at four different temperatures 1448, 1458, 1493 and 1503K. The temperature was measured only on the initial region of the strip and estimated for the others regions. Three different regions of the strip (initial, middle and final regions in longitudinal direction) were used for analysis on the surface and center each strip. The characterization of MnS particles by TEM was carried out by extraction replica and thin foil techniques utilizing a JEOL-JEM-200C. The diameter was measured by an accessory image analyser (Mini-Mop) and the data were compiled and elaborated in microcomputer electronic table. The data gave us a serie of curves about the MnS particles diameter, distribution and mean particle size.

### 3-Results and Conclusions.

Figures 1 and 2 show the increase of grain size with increasing of the time of heat treatment. This effect seems to be due to the precipitation dissolution in the matrix of the steel resulting in a release of the grain boundary. This phenomena was also observed by Obara et. al [4] in specimens after solution treatment at 1623K for 600s without deformation.

Transmission electron microscopy showed that the number of particles decreases within the current time of the heat treatment and



it is consistent with optical microscopy analysis. The particles size covers a range values from 20 to 30nm.

The size distribution of MnS particles is presented in Figs.3 and 4. These curves show that for the four temperatures and (measure on the surface and center of in the initial and middle strip region), the particle size has broadened from 30 to 70nm. This shows evidence for homogeneity in the precipitate size according to the depth.

Careful comparison of Figs.3 and 4 reveals that the number of MnS particle increased from the surface to the center at temperatures 1448 and 1503K and decreased from the surface to the center at temperatures 1458 and 1493K. There are a higher number of precipitate particles in the middle of the sample, but lower mean size diameter comparing with that which occurs at the surface. This suggests that a rapidly cooling on the strip surface during the contact with the rolls produces dislocations (nucleation sites) that can induce the beginning of the precipitation. The particle nucleation site dislocations can be dissolved during the lamination step by reestablishing the initial temperature condition. Consequently, very low precipitation occurs on the strip surface.

During the process, some of these nuclei can be grown and produce stable particles that are bigger than that produced at the end of the lamination process. Fig. 4 shows in the initial part of the strip (for the center of thickness) homogeneity particle distribution is evident for all temperatures.

Fig. 5 and 6 show electron micrographs (TEM) used to follow the progress of precipitation in steel for one temperature, on the surface

and in the center of the strip. It is evident that fewer number of particles are on the surface than in the center of thickness sample.

The mean particle size values reported in Fig.7 show variation within the current temperature control of hot rolling process. There is a higher variation of standard deviation on the surface of strip compared with the center of thickness strip. It has little variation in the mean particle size with increasing temperature, as reported recently by W.P.Sun [7].

#### 4-Conclusions.

The grain size increases with increasing time of the heat treatment. It seems that this is due to the dissolution of MnS particles in the matrix of the steel and causes the release of the grain boundary.

The analysis of MnS precipitates distribution after heat treatment shows that the particle size covers range from 20 to 30nm. The decrease of the number of particles with increasing time of the heat treatment corroborates the optical microscopy observations.

The distribution of MnS precipitates after hot rolling process showed a remarkable homogeneity according to the size particle in the initial region on the surface and in the center of thickness. For the other regions there is no homogeneity according to the size and distribution of the particles. This aspect is due to the variation in the temperature and in the hot mechanical treatment for each temperature.

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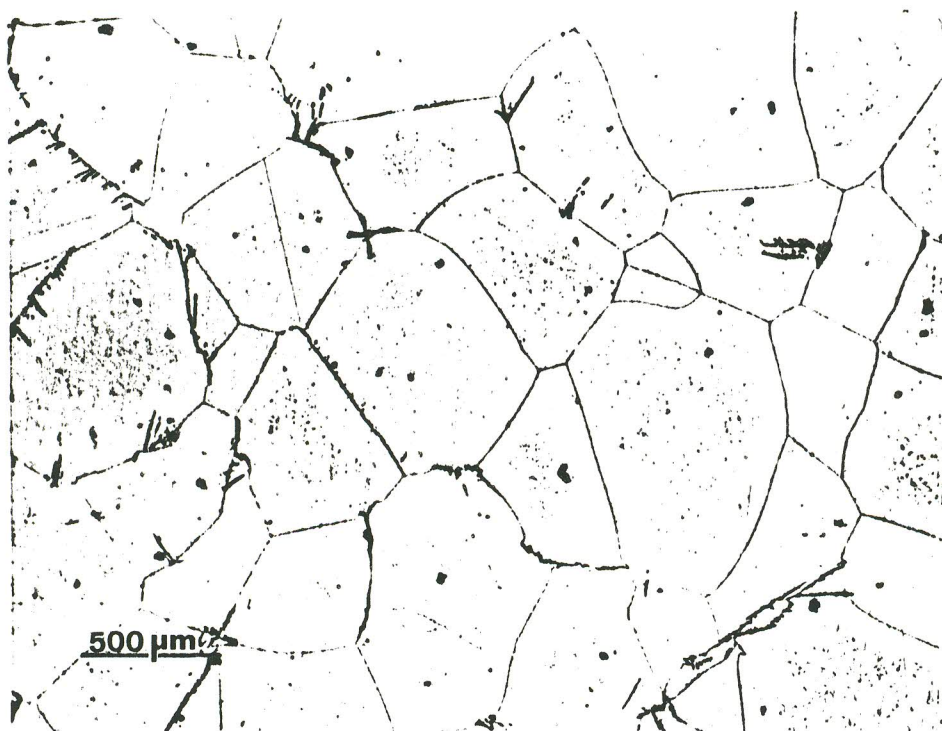
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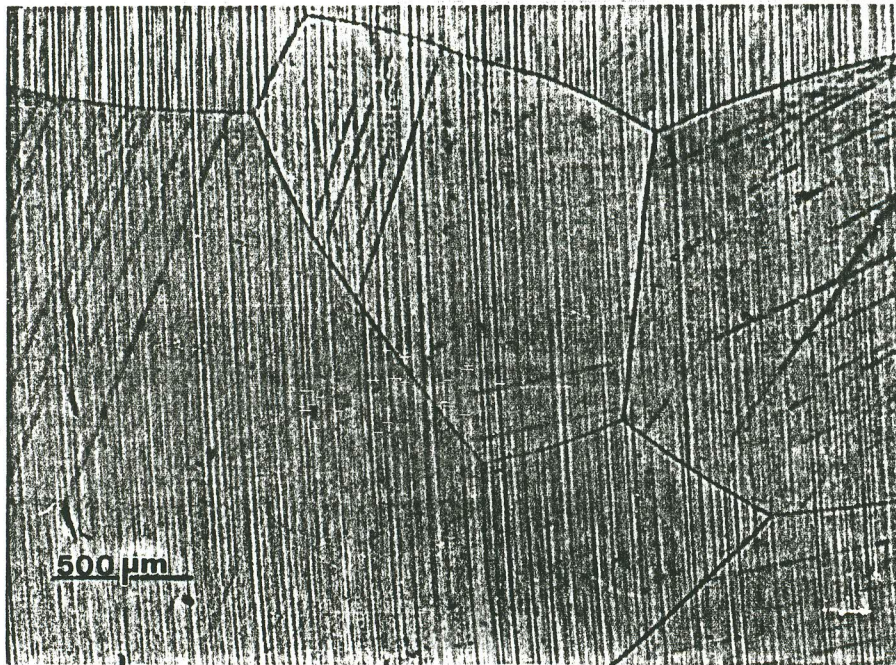
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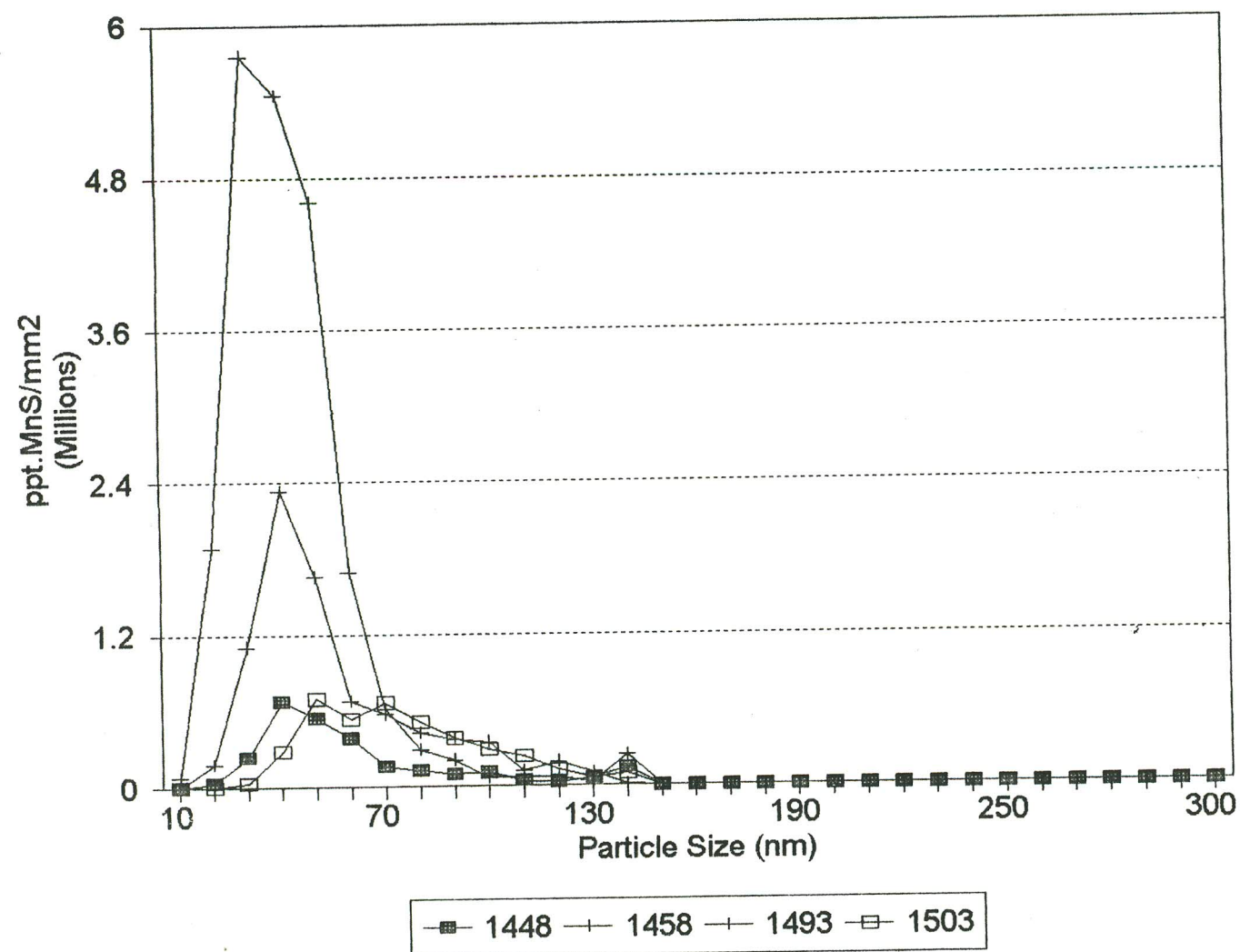
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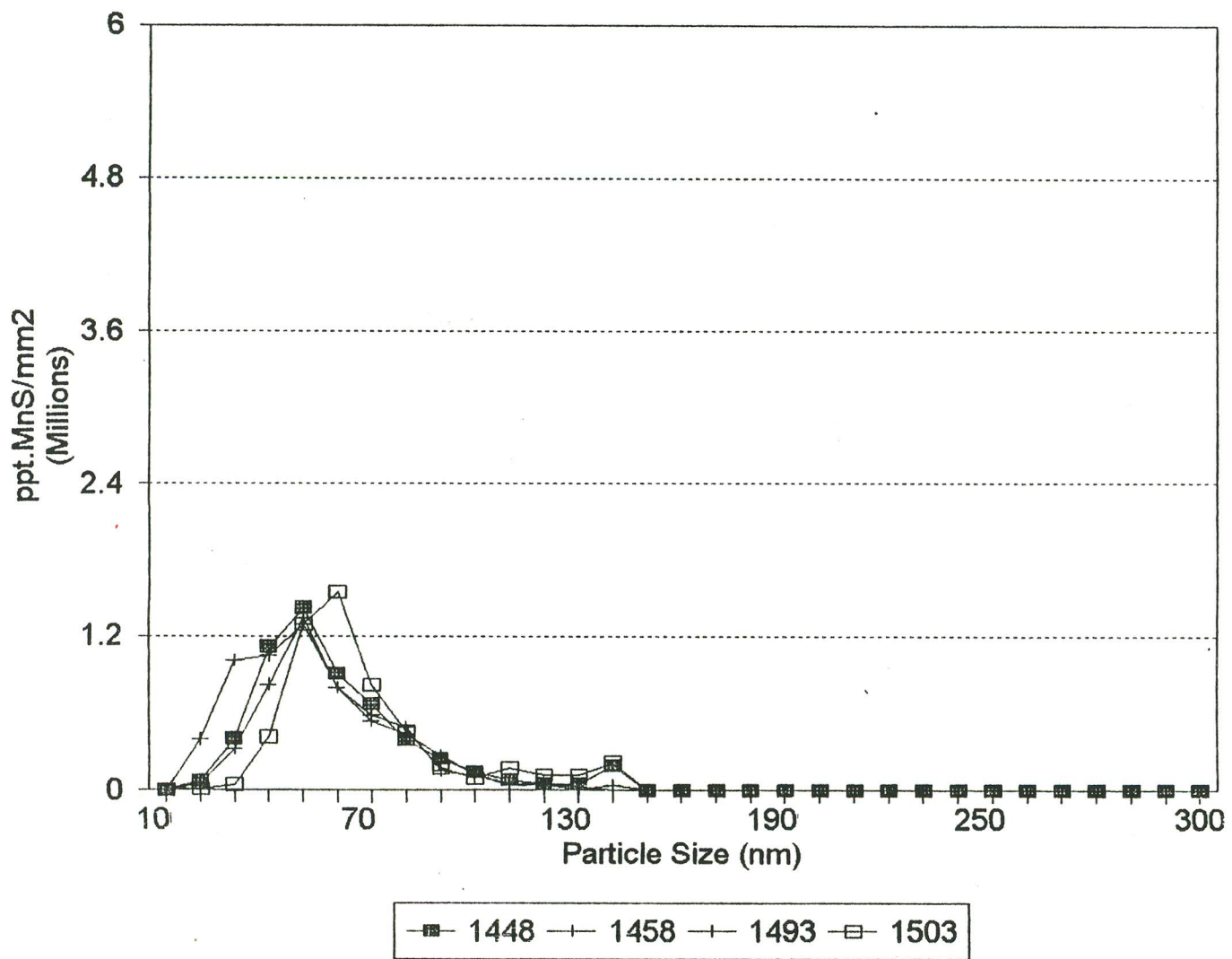
**Fig.1. Optical microstructure of the specimen after solution treatment at 1573K for 300 seconds.**



**Fig.2. Optical microstructure of the specimen after solution treatment at 1573K for 900 seconds.**

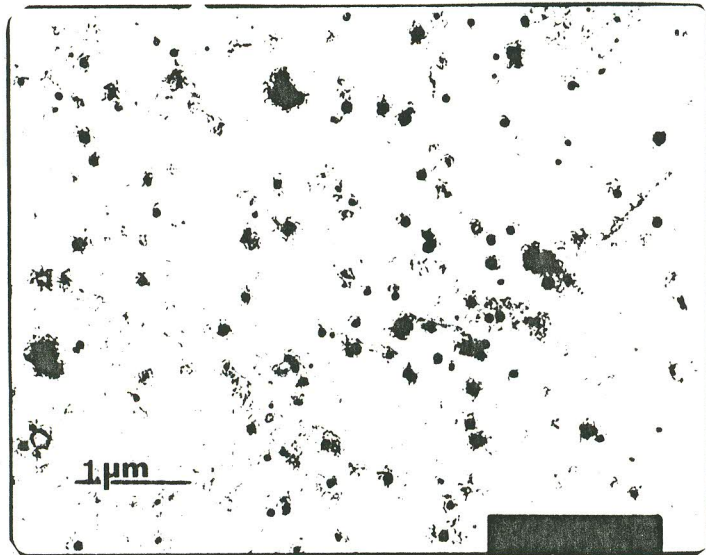


**Fig.3.**Size distribution in Si steel referring to hot rolling process at temperatures 1448, 1458, 1493 and 1503K on the sample surface.

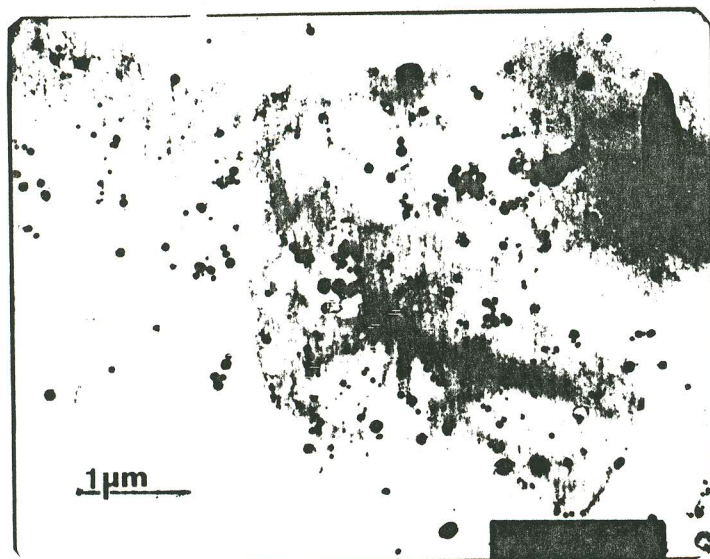


**Fig.4. Size distribution in Si steel referring to hot rolling process at temperatures 1448, 1458, 1493 and 1503K in the center of the thickness of the sample.**

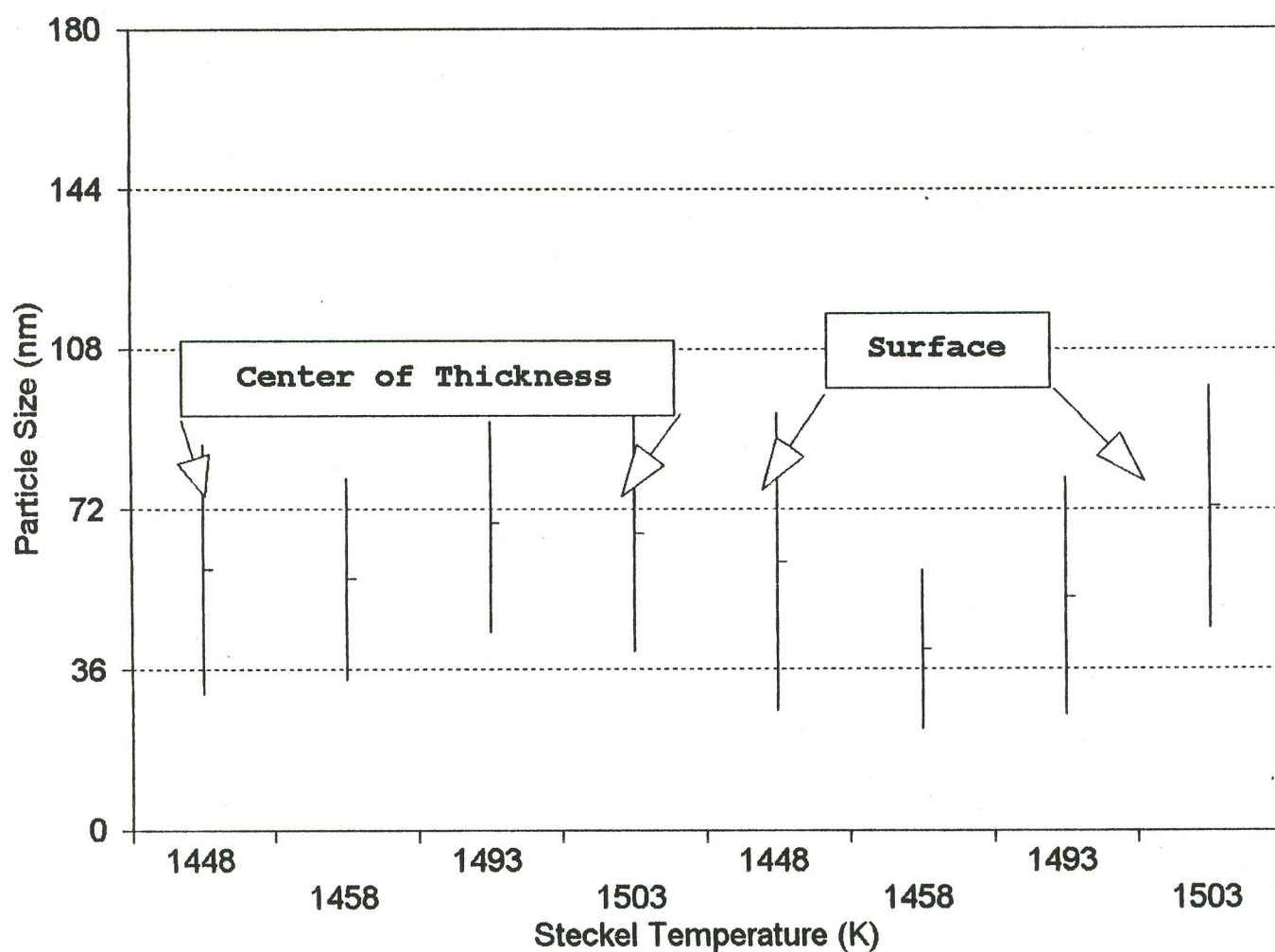




**Fig.5.Mns precipitates observed by TEM on the surface of the sample (Bright field).**



**Fig.6.Mns precipitates observed by TEM in the center of thickness ofthe sample (Bright field).**



**Fig.7.**The mean particle size of MnS particles in the center of thickness and on the surface, in the initial region of the strip, for the temperatures 1448,1458, 1493 and 1503K.