RELATIONSHIP BETWEEN MAGNETIC AGEING, METALLOGRAPHICAL EVIDENCE OF AGEING AND COMPOSITION FOR COLD ROLLED Si-Fe ELECTRICAL SHEETS

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ABSTRACT

The cold rolled Si-Fe sheets used for the production of the current cores for electricity meters suffer from ageing which causes an irreversible change in the magnetic properties. The ageing process is due to the precipitation of mixed iron silicon carbides in the microstructure. There exists already a thermal treatment and magnetic properties measurements method to detect the susceptibility to ageing. This requires a minimum of 1000 hours. In order to find the correlation between magnetic ageing, composition and metallographical evidence of ageing, different Si-Fe sheet compositions were investigated using the optical microscope to observe the times to appearance of visible carbide precipitates after ageing at 150°-C and 200°-C. It was found out that ageing does not occur below a certain carbon composition and that the time required for evidence of ageing to be visible increased with decreasing carbon content. It was concluded that the combined thermal treatment and metallographic examination does not bring the saving in time expected and that carbon analysis of the electrical sheet would be a better control.

1 INTRODUCTION

Silicon iron sheets with carbon content less than 0.08% are mainly used as dynamo and transformer core sheets, as rolled sheets in the building of relays and as yoke parts in electromagnets. Cold rolled Si-Iron electrical sheets suffer from magnetic ageing [1, 2], which is due to the precipitation of Carbides in the microstructure. Magnetic ageing is defined as the change in magnetic properties (deterioration of the magnetic properties of magnetic materials for electronic and electrical applications) of a material resulting from a metallurgical change [5]. The ageing depends on the composition of the electrical sheet specially the Carbon and Silicon content. In an attempt to find a short time test for this ageing susceptibility using mainly metallographic examination of the microstructure after short time ageing, it was discovered that the time to the apperance of visible evidence of ageing varied greatly with the composition of the material [1]. It was not possible to identify with clarity the point of departure from short time observation of ageing to observations after relatively long time. In most of the available literature [1, 3] the carbon content required to cause ageing were higher than the actual contents used today.

For negligible ageing, for example in applications in the current core of electricity meters the carbon content should be lower than 0.002% for steels with 2% Silicon. Formerly the limit used to be 0.006% [2], then dropped to less than 0.003% C. It has also been suggested that the determination of Carbon content should be used as a means of assessing ageing susceptibility.

In an earlier investigation [1], it had been hoped that it would have been possible to identify a short time combined metallographic and thermal test to act as a pre-sorting test while the normal test was going on or to replace this test. The normal testing conditions for electrical steel sheets to be used in the current core of electricity meters are: Ageing at 150°C followed by measurement of magnetic values (in this case H_o values). The change in magnetic values compared to the as received values should not exceed a given value (for H_o the change should not exceed more than ±6.3 mA/cm after 1000 hours of ageing).

In order to be able to give reliable information about the feasibility of the short combined metallographic and thermal test, the investigation presented in this paper was proposed.

2. INVESTIGATION PROCEDURE

Test specimens were exposed to two different ageing temperatures for different times followed by measurement of magnetic properties and cutting of specimens for microstructural examination. The magnetic values chosen in this investigation were the curve form factor H_o and the coercive field strength H_o. In the microstructural investigation evidence of carbide precipitation was searched for. The mixed carbide precipitates were expected to appear as rod, plate or round like depending on the time and temperature of ageing.

3. SPECIMENS AND TEST CONDITIONS

Two different batches of cold rolled electrical sheets, i.e. batch 1 (ca. 1% Si) and batch 2 (ca. 2% Si) were used. Batch 1 consisted of 19 different specimens with different carbon contents and batch 2 consisted of 25 specimens with different carbon contents. The specimens used were strips normally used as Epstein frame probes. Ten strips were obtained for each carbon content. The specimens were aged at 150° and 200°C. Evidence of magnetic ageing was searched for at 150°C with magnetic measurements (curve form factor H_o obtained from magnetic yoke measurements). This was supplemented by coercive field strength H_o measurements. Microstructural evidence of ageing was searched for after ageing at 200°C. In order to compare the ageing behavior from magnetic measurements and metallographic examination H_o values were also measured after ageing at 200°C.

Seven strips with H_o values closest to each other were chosen from the 10 strips available for each composition. Three strips were aged at 150°C, three strips at 200°C, and the seventh was divided into two halves from which metallographic specimens were taken after ageing at 150°C and 200°C for different durations.

4. RESULTS

4.1 Magnetic Measurement Results

The result of the magnetic measurements are plotted in figures 1 to 8. It has been established that not all the specimens with the same composition age to the same extent [4]. This phenomenon was also observed in these tests.

In figures 1 and 2 the H_o values (mA/cm) - Ageing Time (hours) relationship can be seen for the 1% Si steel sheets after ageing at 150° and 200°C. It can be seen that for the 1% silicon steels only the specimens with 0.0015% C do not show an increase in the H value at 150°C and that the majority of the specimens with higher amounts of carbon show a tendency to overage.

The H_o values - ageing time relationship for the 2% silicon steel sheets can be seen in figures 3 and 4 after ageing at 150 and 200^o C respectively. It can be seen in fig. 3 that those specimens with a carbon content less than 0.002% do not show a significant increase in the H_o values. The same results can be seen in fig. 4 where those specimens with carbon contents of less than 0.002% show an improvement (a drop) in the H value after ageing for 1000 hours at 200^{o}C. Only the specimen with 0.003% C and 1.81% Si seems to approach a maximum in this test (fig.4). At both temperatures 150^{o}C (fig.3) and 200^o C (fig.4) the specimen with 0.00000015% C: 1.83% Si falls outside the general patternm showing a larger increase in the H_o values than would be expected when compared to the other composition close to it, e.g. 0.0016% C and 0.002% C.

The values of the degree of ageing ΔH_o % in relation to the ageing and carbon contents for the two batches 1 and 2% silicon sheets respectively are plotted in figures 5 and 6. All sheets with a carbon content greater than 0.0015% C and roughly 1% silicon (fig.5) suffered from ageing [ΔH_o > 10%].

The ΔH_o % values increased generally with increasing carbon content after 1000 hours of ageing at 150^{o}C with the exception of the specimen with 0.0016% C: 1.46% Si (fig.5) which showed a ΔH_o % value between that of the 0.0025% and that of the 0.002% C specimen. This cannot be due to the Si content as the specimen with 0.002% C, had the same Si content as the 0.0016% C one (both 1.46% Si). This could therefore be due to a mistake in determining the carbon content, which can very easily occur when one is dealing with such small amounts of carbon as are found in these specimens. A mistake in measuring the H_o value is out of question as measurements were taken after different time intervals and all fall on the same curve.

The incubation time (i.e. the ageing time to a noticeable increase in the ΔH_o % value) tends to increase with decreasing carbon content.

The ΔH_o % values for the 2% Si sheets (fig.6) start to increase with a carbon content of 0.002 % and by a carbon content of 0.0025% C has increased by more than 150% compared to the as received condition after ageing for 1000 hours.

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[1] The degree of ageing in percentage is defined as follows:

$$\Delta H_o \ \% = \frac{H_{ot} - H_{o2} \times 100}{H_{oa}}$$

where H_{ot} = H_o value (mA/cm) after ageing for t hours

H_{oa} = H_o value in the as received condition.

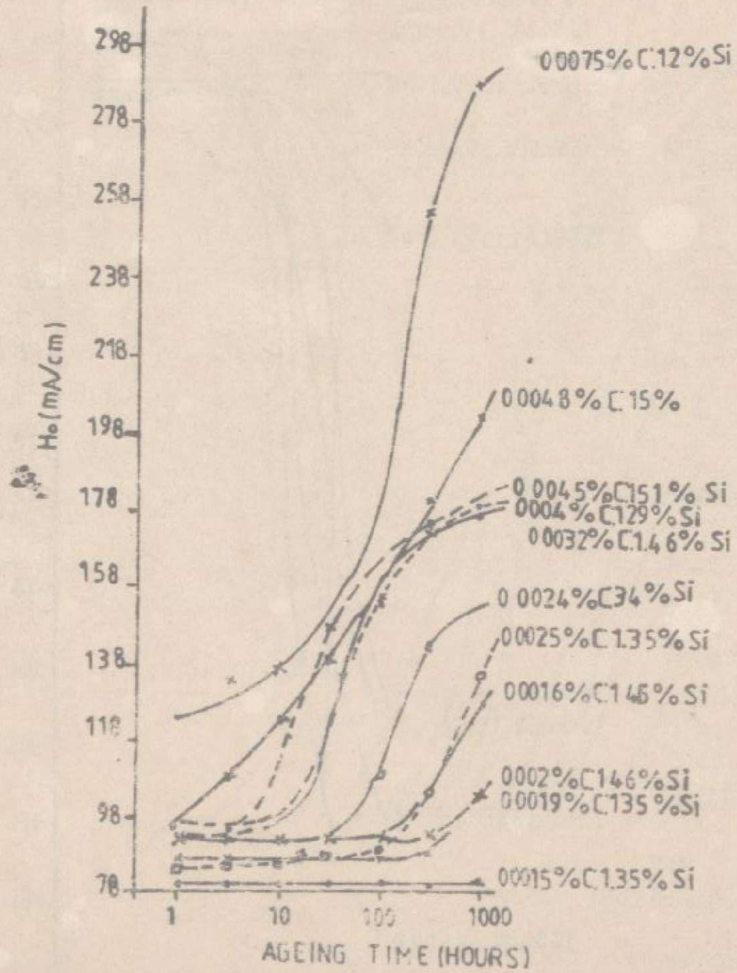

Fig. 1. H_o values vs Time at 150° C for the 1% Si-sheets

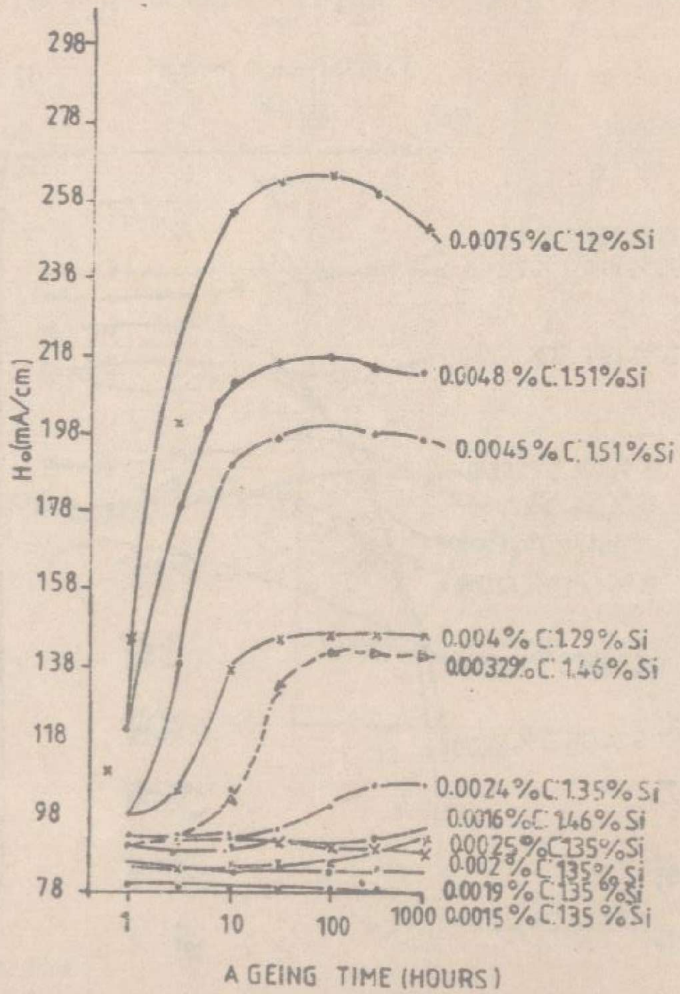

Fig. 2. H_o values vs Time at 200° C for the 1% Si-sheets

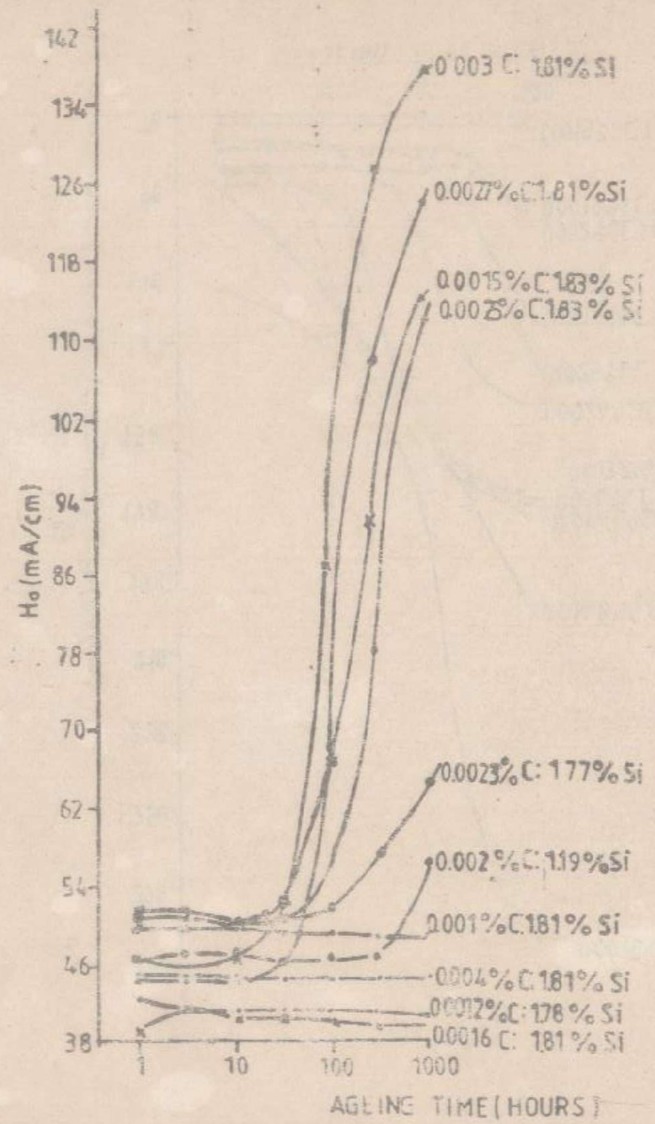

Fig. 3. H_0 value vs Time at 150°C for the 2% Si-sheets

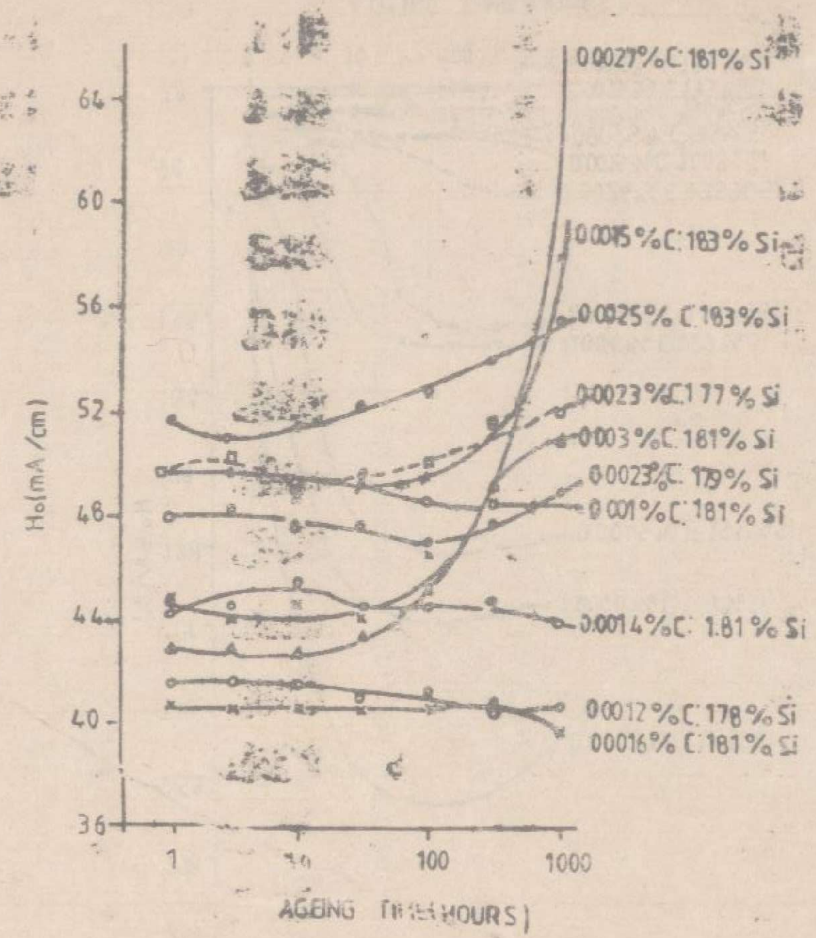

Fig. 4. H_0 values vs Time at 200°C for the 2% Si - sheets

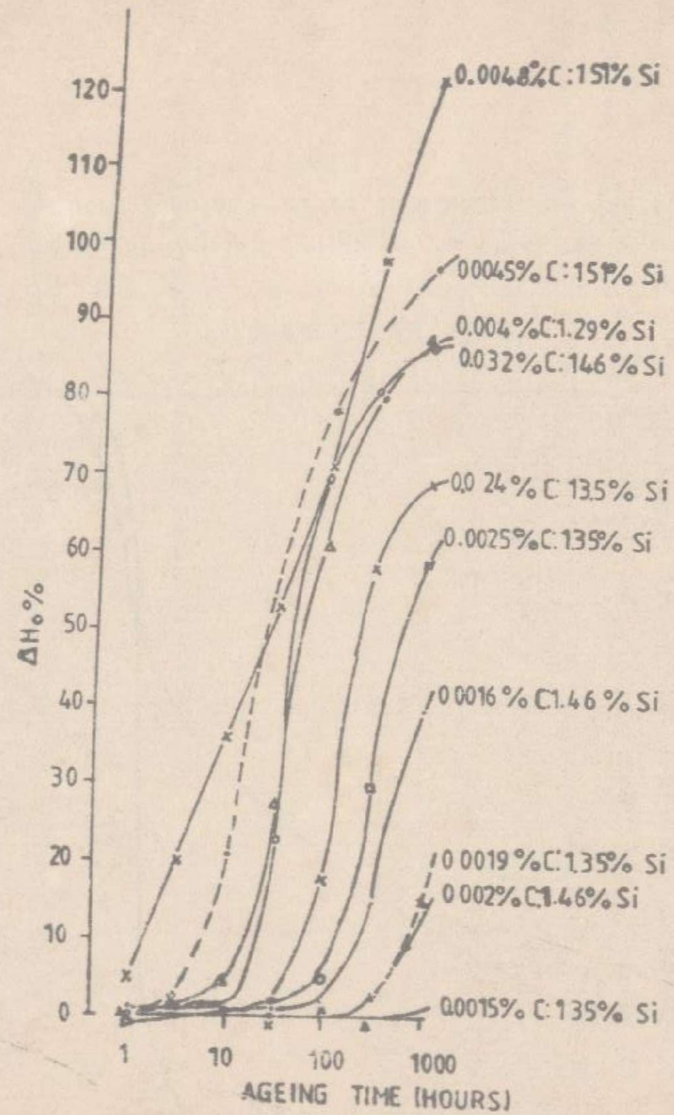

Fig. 5. H_0 variation in comparison to the as received condition after ageing at 150°C [1% Si]

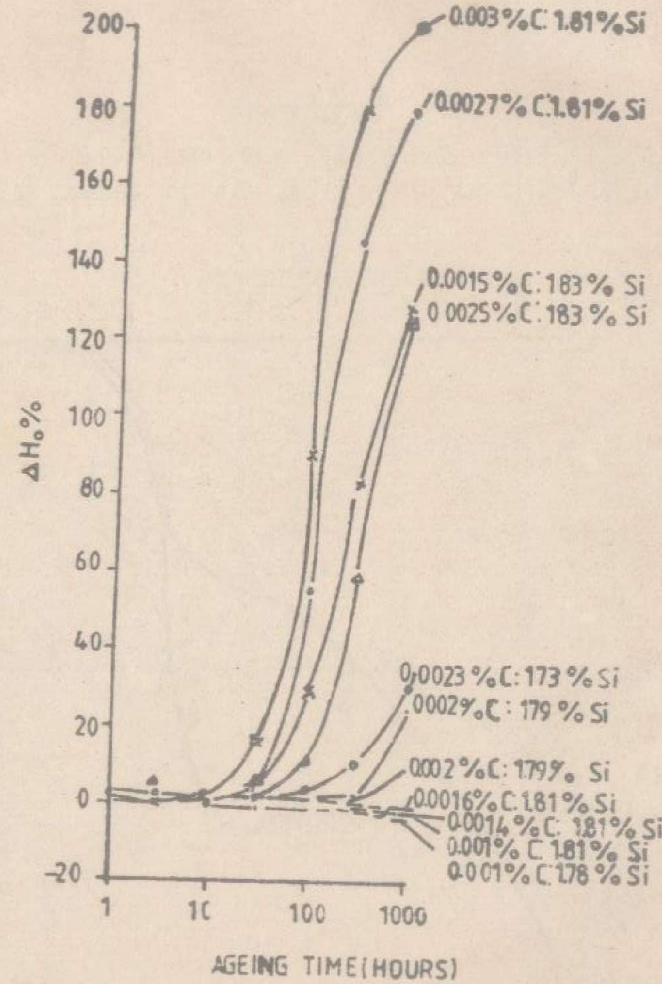

Fig. 6. H_0 variation in comparison to the as received condition after ageing at 150°C [2 %Si]

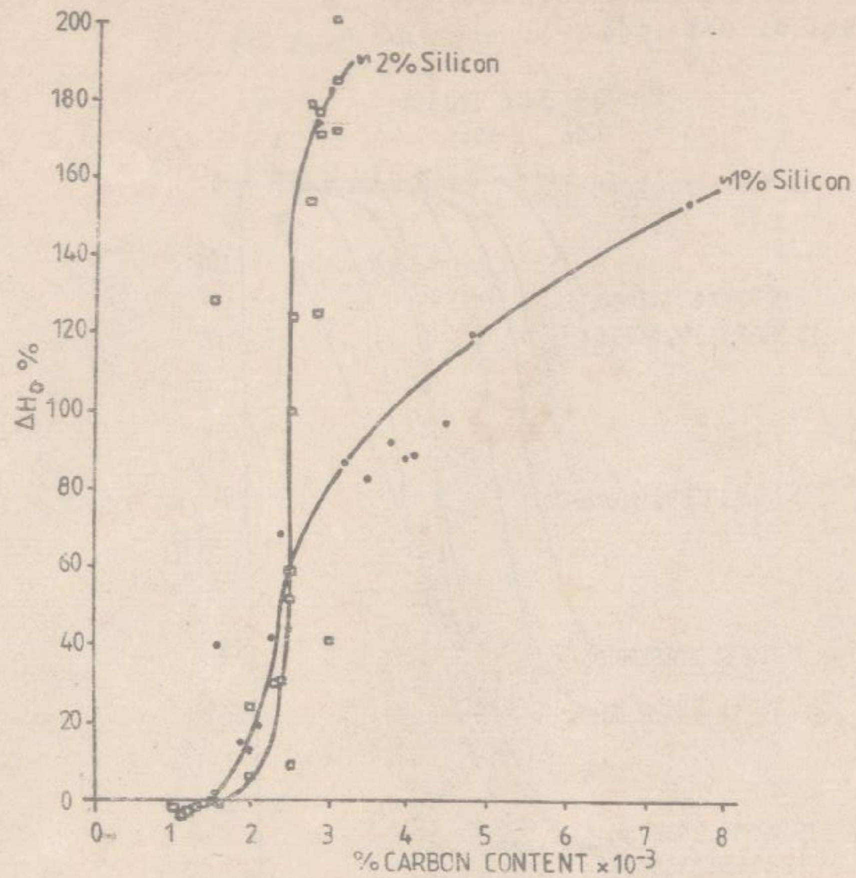

Fig. 7. Degree of ageing (ΔH_o) in relation to the carbon content after 1000 hours at 150°C [1% & 2%Si]

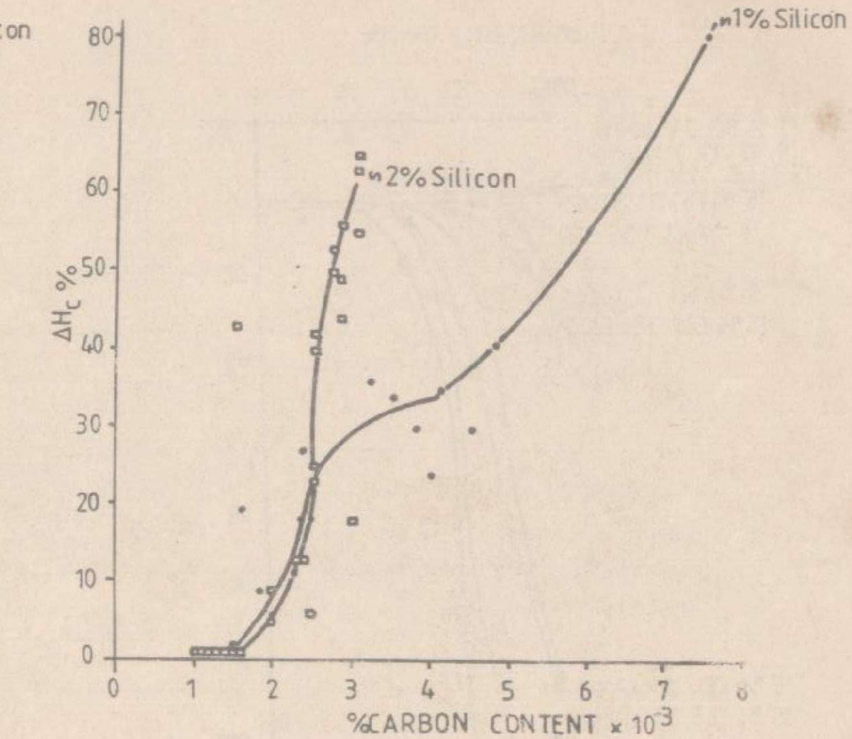

Fig. 8. Degree of ageing (ΔH_o) in relation to the carbon content after 1000 hours at 150°C [1% & 2%Si]

The percentage change of the H_o values (fig. 6) generally increases as would be expected with increasing carbon content with the major exception of the specimen with a carbon content of 0.0015% C: 1.83% Si which falls out of the pattern having a ΔH_o % increase between that of the specimens with 0.0025% C and 0.0027% C.

Those specimens with a carbon content less than 0.002%C showed very little susceptibility to ageing after testing for 1000 hours with the exceptions already mentioned. Incubation times also increase with decreasing carbon content.

Figure 7 is a plot of the degree of ageing in relation to the carbon content for the two batches (1 and 2% Si). The degree of ageing is defined as ΔH_o % after 1000 hours of ageing at 150° C. Generally the 1% silicon sheets show a lower degree of ageing than the 2% silicon sheets for roughly the same carbon content. Measurement of H_o values confirm the higher susceptibility of the 2% silicon sheets to ageing. This is however limited to carbon contents higher than 0.002%. Below this level the 2% silicon sheets have the same susceptibility to ageing as the 1% silicon sheets in some cases being slightly less. It is possible to draw the following conclusion from figures 7 and 8: significant ageing does occur for the 1% silicon sheets with carbon contents equal or less than 0.0015% C and for the 2% silicon sheets with less than 0.002% C.

4.2 Metallographical Examination Results

It was observed that for the 1% Si sheets and an ageing temperature of 200°C the time required to visible precipitates was between 30 and 70 hours as the carbon content drops from 0.0075% C to 0.0032% CC. This increased to between 300 and 500 hours as the carbon content further dropped from 0.0025% C to 0.0016% C. As the carbon content dropped further to 0.0015% C were not available. Exceptions to the above general behavior were ob served with the specimens with carbon contents of 0.0020% C: 1.46% Si and 0.0019% C: 1.35% Si. The specimen with the composition 0.0020%C : 0.0020%C : 1.46% Si produced no visible precipitates even after 1000 hours of ageing at 473 K. The specimen with a carbon content of 0.0019% C exhibited as strange behavior in that carbide precipitates were observed only after 125 hours of ageing. Higher times of ageing resulted in no visible carbide precipitates. This behavior persisted even after the preparation of a second metallographic specimen.

For the 1% Si, sheets aged at 150°C it was observed that the times to observation of visible carbide precipitates agree with those mentioned above except for one specimen with 0.0010% C and 1.35% Si in which carbide precipitates were observed only after 1000 hours as in contrast to the behavior at 200°C.

For the 2% Si sheets aged at 200°C it was observed that the time required to visible precipitates was between 135 to 300 hours as the carbon content dropped from 0.003 to 0.0027%. This time interval increased to between 300 hours and 500 hours as the carbon content further dropped to 0.0023%. Between 0.0025% C and 0.0015% C uncertainties stgart to creep in and the behavior of the electrical steels in this range is difficult to predict. There was no observation of visible carbide precipitation below 0.0015C.

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The uncertainties mentioned above are:

Out of 5 specimens with a carbon content of 0.0025%, two developed visible precipitates between 125% and 300 hours, two others between 300 and 500 hours and one specimen showed no visible carbide precipitates even after 1000 hours of ageing. The silicon content varied from 1.82% down to 1.79%. The specimen without visible carbides was

fairly clear with few inclusions as compared to the ageable 0.0025% C specimen which had large inclusions in the microstructure.

Two specimens with carbon contents lower than 0.0025% showed visible precipitates between 125 and 300 hours.

Of the two specimens with a carbon content of 0.002%, one showed no visible precipitates even after 1000 hours of ageing

TABEL 1

COMPARISON OF OBSEATIONS FROM MAGNETIC AND METALLOGRAPHIC TESTS

COMMENT	ca. 1 % SILICON SHEET	2 % SOLICON SHEET
1. MAGNETIC MEASURENT		
1.1 Generally no ageing below	0,0015 % C	0,002 % C
1.2 Exceptions and Discrepanci:	0.0016 % C	0,0015 C
1.3 Ageing Time Temperature	1000 hours/150 °C	100 hour / 150 °C
1.4 Sensitivity to Ageing:	less	more sensitive
2. METALLOGRAPHI EXAM		
2.1 Generally no ageing below (No carbide were seen.	0,0015 % C	0,002 % C
2.2 Exceptions and Discrepancies:	0,0019 % 0,0016 % C	0,0025 % C 0,0015 % C
2.3 Time to obsavation of visible precipitate		
for 200 °C	minimum:30 hours(0,0075 %C)	30 hours (0,003 % C)
	maximum:30-500 h (0,0016 %C)	≥ 500 hours(0,002 %C)
for 150 °C	minimun:30 hours(0,0045 % c)	30 hours (0,003 % C)
	maximum:1000 hours(0,0019%C)	≥ 500 hours(0,002 %C)
2.4 Grain Structure	fine	
2.5 Chemical Attack:	sensitive (less etching time)	

N.B. The sensitivity varies also with decreasing carbon content for both types of silicon sheets ageing although the other showed some precipitates between 500 and 1000 hours of ageing

The next specimen with a low carbon content (0.0016% C) showed no evidence of visible carbide precipitates, but another one with a carbon content of 0.0015% exhibited a highly ageing behavior with rod-like precipitates being visible after 70 to 125 hours of ageing, while another specimen with the same carbon content showed no evidence of visible carbide precipitates.

The 2% Si sheets aged at 150°C behaved similar to the 1% Si sheets which had been aged at 150°C, although a slightly different type of precipitate was observed (round-like developing toward short rod like).

Typical microstructure are depicted in figures 9 and 10. A detailed summary of the comparison between metallographic examination and magnetic measurement is given in Table 1.

5. DISCUSSION OF RESULTS

The results of the magnetic measurements seem to agree generally with the observations made during metallographic examination in that those specimens which were observed to undergo ageing by the metallographic examination were also ageing according to the magnetic measurements. The magnetic measurements were carried out after ageing for 1000 hours at 150° C. This is taken as the shortest time possible before reliable results can be obtained.
For the time possible before reliable results can be obtained.

For the confirmation of ageing through metallographic examination a minimum of about 500 hours of ageing at 200° C was required. This time depends on the composition of the silicon sheets, being as short as 30 hours for carbon contents around 0.0075% and 1% and 1% Si and increasing as the carbon content decreased to more than 500 hours at a carbon content around 0.002% and 2% silicon.
This general trend was adhered to except for the few discrepancies already mentioned. The exceptions to the above trends were observed by both metallographic and magnetic measurement tests as summarized in table 1.

One specimen (0.0015% C: Si) did not follow the general pattern described above both from magnetic measurements and metallographic examination. This can be explained as possibly due to errors in the carbon content determination, a fact which can easily occur when dealing with such small quantities of carbon. It can be seen from figure 5 that this specimen should have a carbon content closer to 0.0025% C. This was also confirmed by the density and number of rod like precipitates observed only after 300 hours in this specimen. Another specimen (0.0010% C: 1.35% Si) would appear to have a carbon content greater than indicated or slightly higher than 0.002% C which does not suffer from ageing. The fact carbides were observed only after 125 hours to 200 C and not later confirms the assumption that this specimen is on the transition zone from the higher ageing steels to the non ageing low carbon content ones.

Fig.9 0.0075%C:1.29%Si
Fig.9a As received 200X

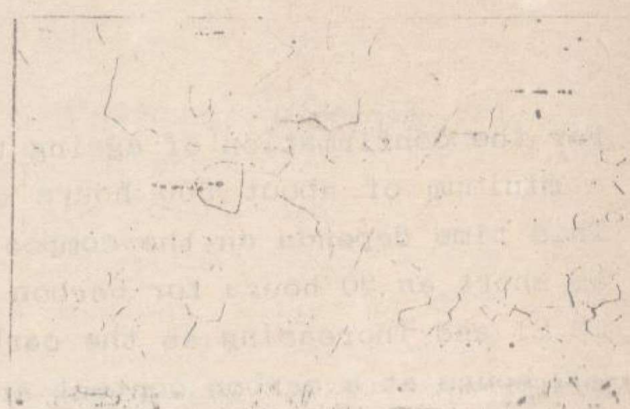

Fig. 9b After 30 hours at 200 C
200X

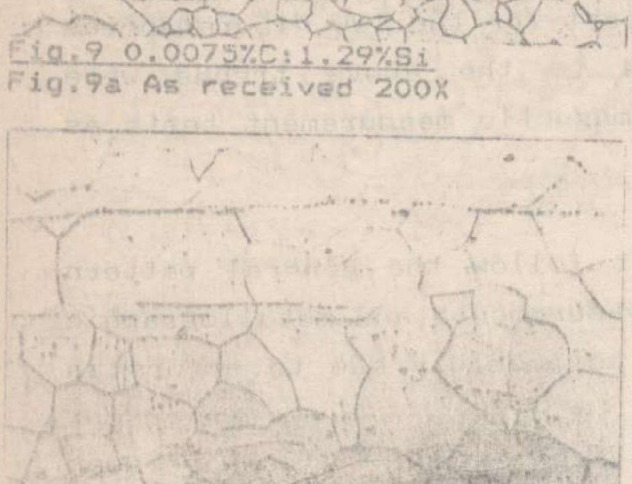

Fig. 9c As received 500X

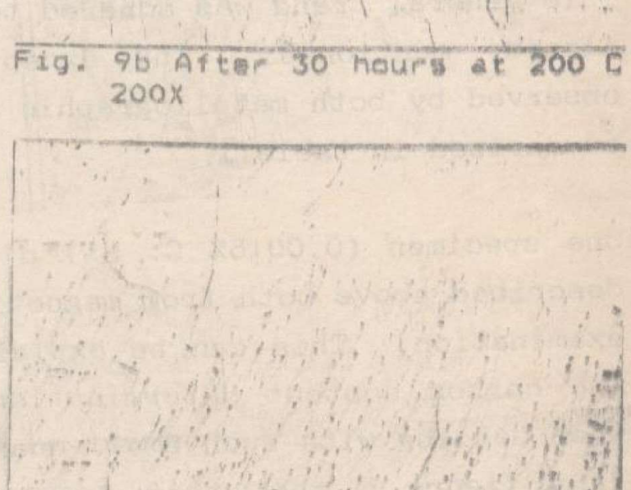

Fig. 9d After 30 hours at 200 C
500X

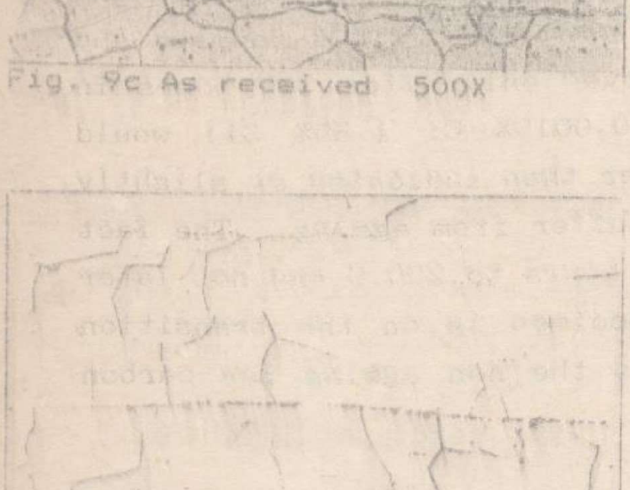

Fig. 9e As received 500X
(Interference Contrast)

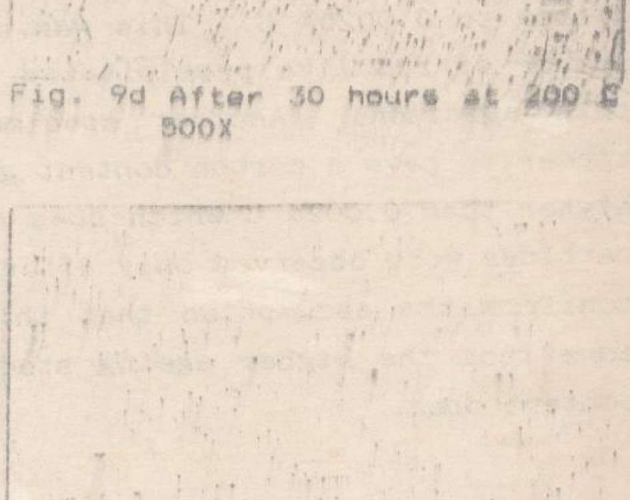

Fig. 9f After 300 hours at 200 C
500X

Fig.10 0.003%C:1.81%Si
Fig.10a As received 500X

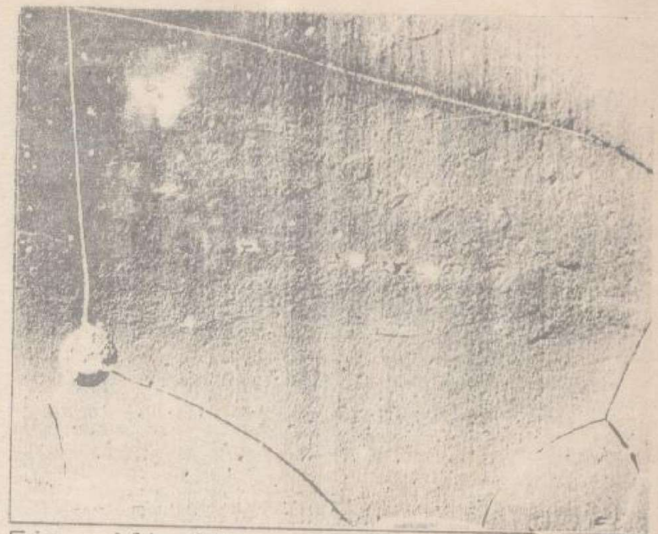

Fig. 10b After 70 hours at 200 C 500X Rod like Carbides

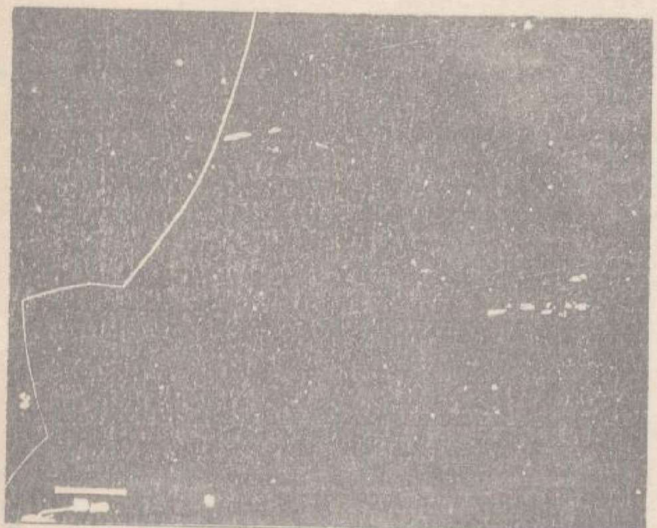

Fig. 10c After 300 hours at 200deg C 500X Rodlike Carbides

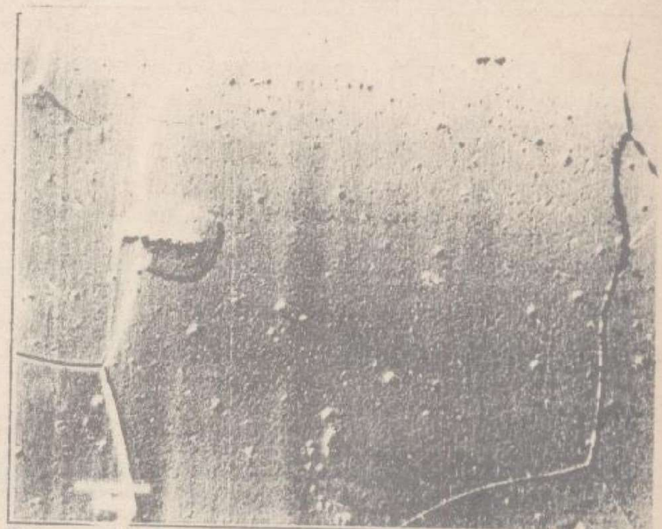

Fig. 10d After 70 hours at 150 C 500X Many round like Carbides

Fig. 10e After 300 hours at 150deg C. Coarsened roundlike Carbide 500X

Fig. 10f After 1000 hours at 150deg C. Mixed elongated rod and round like Carbides. 500X

Th[illegible] sa[illegible] [illegible]ption of a mistake in the carbon content [illegible] can be used to explain th[illegible] behaviour of another [illegible]en (0.0015%C, 1.81%Si) in fig. 3. The density and size of [illegible]ne rod-like carbvides which were observed confirmed this assumption.

6. SUMMARY AND CONCLUSIONS

Two different batches of cold rolled electrical steels, i.e. batch 1 (ca 1% Si) and batch 2 (ca 2% Si) were investigated for the relation between metallographical/magnetic ageing and carbon content at ageing temperatures of 150 and 200 C.

From the magnetic measurement results, it was found that:

- For both batches the ΔH% increased generally with decreasing carbon content.
- The incubation time at 150^{o} C tendered to increase with decreasing carbon content for both batches.
- All sheets with a carbon content greater than 0.0015% C and 1% Si suffered from ageing.
- Those specimens with a carbon content less than 0.002%C and 2% Si showed very little susceptibility to ageing even after 1000 hours at 150^{o}C
- Not all specimens with the same composition shared the same susceptibility to ageing.

- The 2% Si sheets were more sensitive to ageing than the 1% Si sheets when the carbon content for both sheets exceeded 0.0023%. Below this limit they showed approximately the same sensitivity.

From the metallographical examinations it was found that:

- Carbide precipitates could not be observed with the light microscope below 0.0015% C for the 1% silicon sheets and below 0.0020% C for the 2% silicon sheets at both 150 and 200°C.

- The number and density of the carbide precipitates observed decreased with decreasing carbon content.

- The times required for ageing to result in visible carbide precipitates generally increased with decreasing carbon content ranging from 30 hours to 500 hours, for the 1% silicon and from 30 hours to slightly above 500 hours for the 2% silicon sheets at an ageing temperature of 200°C.

- Carbide precipitates were observed with the light microscope at both 150° and 200°C, although the precipitates were of a slight different appearance.

- The grain structure of the 1% silicon sheets was observed to be finer and more sensitive to etching than that of the 2% silicon sheets.

It has been showed in this investigation that the use of the combined metallographic and short time thermal treatment is fairly viable as a sort of a pre-sorting test when the time of 500 hours is acceptable in comparison to the full standard test for 1000 hours. If as envisaged that evidence of ageing should be visible around 24 hours to 48 hours, then the combined metallographic and short time thermal treatment is not viable as in this time a number of low carbon electrical sheets still show no evidence of visible carbide precipitates.

Carbon analysis can be used as a control to check for susceptibility to ageing only when reliable and accurate analysis is possible. Difficulties or errors in determining the carbon content can result in condemning a good non-ageing sheet and vice versa.

In my opinion other parameters like the different sensitivities to chemical attack observed also in this investigation should be investigated.

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