Room Temperature Straining Behaviour of Iron–Silicon Alloys

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(Received on August 28, 2002; accepted in final form on October 31, 2002)

A series of iron–silicon alloys were produced to study the mechanical behaviour at room temperature of alloys ranging from 3.3 to 6.5 wt% Si. Cast samples were machined into compression specimens to be tested in a servo-hydraulic computer driven testing machine at a constant rate of strain, polytetrafluoroethylene tape was used to reduce the effect of friction while testing. Constitutive equations were fit from the stress–strain data. It was possible to establish a relationship between the parameters used in the constitutive formulation and the amount of silicon added. Metallographic observation of deformed specimens revealed the presence of twins in the material. It was found that the twin density increased with the amount of deformation and silicon. A relationship between the stress for saturation and the distance between the twins was established.

KEY WORDS: mechanical properties; twinning; microstructure.

1. Introduction

Fe–Si alloys are used as soft magnetic materials in electrical appliances and devices, conventional steels containing up to a 4.5 wt% Si are rated in terms of the power lost when subjected to magnetization in an alternated electric field. These steels are used as thin strips of shapes punched from cold-rolled sheets, called laminations, and are coated or oxidized to reduce electrical losses. Magnetic characteristics of iron–silicon alloys are improved as the content increases. An optimum is found at around 6.5 wt% Si in which almost zero magnetostriction, concurrent with high electrical resistance and magnetic permeability, are found to occur. Such combination of properties allow for achieving low electrical losses, due to Eddy current and hysteresis cycles, especially at high frequencies.1–3) Fe–Si alloys have the drawback of becoming brittle as the silicon content increases. This phenomenon is more acute at silicon contents above 4 wt%, and makes them difficult to cold-roll. Such a lack of ductility has been attributed to structural ordering into at least two phases known as B2 (FeSi) and DO3 (Fe3Si). A considerable amount of experimental and theoretical work has been undertaken to determine the composition and temperature ranges, over which as B2 and DO3 phases are stable, to establish the nature of the order/disorder transformations and the critical temperatures at which these transformations take place. It is known that iron alloyed with more than 8.5 wt% Si has the atomic arrangement characteristic of a DO3 phase.4–6) Irons alloyed with silicon in the range of 5 to 8.5 wt% fall in a two-phase mixture region of B2 and DO3.7–10)

The aim of this work is to present the results obtained while studying the plastic behaviour of a series of iron-silicon alloys deformed in compression at room temperature, the range of variation of silicon was kept between 3.3 and 6.3 wt%.

2. Experimental Procedure

Four experimental iron–silicon alloys were prepared by melting them in air in an induction furnace using ultra low carbon steel scrap and a ferroalloy containing 75 wt% Si. The composition of the experimental alloys are shown in Table 1. The liquid metal was poured into permanent moulds to obtain ingots of 30 mm thickness, 180 mm width and 250 mm height. Cylindrical specimens of 15 mm height by 10 mm diameter were machined from the ingots to be tested in axisymmetric compression conditions. The specimens were tested in a computer driven servo-hydraulic machine designed to compute and maintain a

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C (wt. %)</th>
<th>Si (wt. %)</th>
<th>Al (wt. %)</th>
<th>Mn (wt. %)</th>
<th>S (wt. %)</th>
<th>P (wt. %)</th>
<th>Grain size (mm)</th>
<th>d (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.003</td>
<td>3.22</td>
<td>0.004</td>
<td>0.047</td>
<td>0.106</td>
<td>0.013</td>
<td>1.247</td>
<td>38.27</td>
</tr>
<tr>
<td>B</td>
<td>0.009</td>
<td>4.20</td>
<td>0.003</td>
<td>0.093</td>
<td>0.106</td>
<td>0.004</td>
<td>0.609</td>
<td>21.36</td>
</tr>
<tr>
<td>C</td>
<td>0.002</td>
<td>5.00</td>
<td>0.006</td>
<td>0.059</td>
<td>0.007</td>
<td>0.013</td>
<td>0.820</td>
<td>12.43</td>
</tr>
<tr>
<td>D</td>
<td>0.006</td>
<td>6.30</td>
<td>0.002</td>
<td>0.092</td>
<td>0.009</td>
<td>0.005</td>
<td>1.043</td>
<td>8.94</td>
</tr>
</tbody>
</table>

d is the distance between twins.
constantly reducing velocity profile that assures a constant rate of deformation during the whole test. Testing was carried out at a constant rate of strain of 0.5 sec$^{-1}$, various specimens were deformed to strain levels ranging from 0.2 to 0.8. A number of specimens were drilled to their geometrical centre and a type-K thermocouple was inserted to record temperature changes while testing. All the experiments were conducted using 0.1 mm thick polytetrafluoroethylene (PTFE) tape as lubricant to reduce the frictional conditions at the specimen-tooling interface. The raw load-displacement data was converted into stress–strain curves. Full description of the testing machine and the procedure used can be found elsewhere.$^{11}$ Samples in their as cast condition, and deformed to different levels of strain were prepared for their microstructural examination by optical microscopy (OM) and scanning electron microscopy (SEM) following standard metallographic procedures. The samples were etched with a 2% solution of nitric acid in ethylic alcohol.

3. Results

Figure 1 shows the microstructure of the as cast materials, the average grain size measured by the mean lineal intercept technique is presented in Table 1. A characteristic that was observed in the deformed material was the incidence of extensive twinning. Figure 2 shows the microstructures that were observed in samples compressed to a strain close to 0.8. It is worth noticing in this last figure that the amount of twinning increases with the amount of silicon added to the experimental castings.

Figures 3 and 4 show, respectively, the stress–strain curves for materials A and C, 3.3 and 5 wt% Si, where the reproducibility of the tests can be attested. The experimental data are shown with dots, whereas the continuous line indicates the stress–strain curve that is obtained after assuming that the material follows the constitutive equation described by$^{12}$:

$$\sigma = \sigma_s + (\sigma_y - \sigma_s)e^{-C \varepsilon} \quad \text{....(1)}$$

where $\sigma_s$ is the saturation or steady state stress, $\sigma_y$ the yield stress and $C$ the coefficient determined from experimental data. The full lines that are shown in Figs. 3 and 4 do not correspond to an experiment in particular but were drawn from a statistical model that will be described later on.

Figure 5 shows the stress–strain curves obtained for four different specimens, one for each alloy. The fitted curves, broken in this figure, correspond to the individual tests and were deduced by plotting the instantaneous work hardening rate ($d\sigma/d\varepsilon$) as a function of stress. Figure 6 shows such a construction for the curves from Fig. 5, where it can be seen that the material follows the constitutive relationship given by Eq. (1), as the straight portion of the experimental data follows the relationship:

$$\frac{d\sigma}{d\varepsilon} = C\sigma_y - C\sigma \quad \text{....(2)}$$

therefore, the values of $C$ and $\sigma_y$ are obtained by fitting a straight line through the experimental data points. The values of $d\sigma/d\varepsilon$ were calculated by fitting a succession of second degree polynomials to an even number of points of the stress–strain curve; the procedure used in this work is described elsewhere.$^{13}$ The broken line describing Considere’s criterion, $d\sigma/d\varepsilon = \sigma_y$ in Fig. 6 can be used to obtain the stress at which necking will start to develop in tension in a strain rate insensitive material. The strain for the onset of necking ($\varepsilon_n$) can then be obtained from the stress-strain curves, Fig. 5, or by:

Fig. 1. Optical micrographs of the as-cast material with 3.32 (A), 4.20 (B), 5.00 (C) and 6.30 (D) wt% Si respectively, see Table 1.
Fig. 2. Optical micrographs of samples deformed at room temperature to an equivalent strain of 0.8, 3.32 (A), 4.20 (B), 5.00 (C) and 6.30 (D) wt% Si respectively, see Table 1.

Fig. 3. Stress–strain curves from four specimens of material A tested in compression.

Fig. 4. Stress–strain curves from four specimens of material C tested in compression.
The stress at different values of strain, together with that at Considere's criterion and at saturation are plotted as a function of the silicon content of the alloy in Fig. 7, the slope of 83 : 1 corresponds to the solid solution strengthening caused by the addition of silicon.15)

4. Discussion

Figure 6 indicates that the work hardening rate \( \frac{d\sigma}{de} \) follows the lineal relationship with stress that is associated

\[
\varepsilon_n = \frac{1}{C} \ln \left[ \frac{\sigma}{(\sigma_n - \sigma_0)(1+C)} \right]
\] (3)
with Stage III deformation in single crystals and polycrystal alloys. Such behaviour is maintained from yield ($\sigma_y$) up to saturation ($\sigma_s$), but, as straining progresses beyond the maximum value of stress, the strength of the material diminishes. The amount of reduction in strength beyond the maximum value increases as the strength of the material increases as in Fig. 5. These observations can be explained by the results in Fig. 8, where the temperature increase due to adiabatic heating in samples of materials A and C is plotted as a function of strain. The theoretical adiabatic increase in temperature, given by:

$$\Delta T = \frac{1}{\rho \cdot c_p} \int \sigma \, d\varepsilon$$

where $\rho$ and $c_p$ are the density and heat capacity of the material, and the integral represents the area under the stress–strain curve, which results to be linear. It is worth noticing that the ratio of the theoretical adiabatic rise over the temperature recorded in the tests is close to 0.7 in the higher alloyed material C, in comparison to less than 0.4 in the specimens machined from the material A. The difference in this ratio can be attributed to the decrease in thermal conductivity caused by the increase of Si. The net increase in temperature for samples A and C was of around 60 and 150°C, respectively, therefore the softening detected in the samples with higher amounts of silicon. The value of $c_p$ was set to be equal to 430 J/kg K, whereas the density of the material, in kg/m³, was allowed to change with the amount of silicon:

$$\rho = 7865 - 65 \cdot \%\text{Si}$$

where the amount of silicon is expressed in mass percentage.

Figure 9 shows that the variation of the three parameters ($\sigma_y$, $\sigma_s$, and C) that are used to construct the constitutive relationships of the type given by Eq. (1) depends on the amount of silicon added to the material. The relationships between the amount of silicon and either parameter were used to construct Fig. 10 that represents the variation of strength of the material with respect to the amount of silicon. The experimental curves included in this figure are those in Fig. 5. The full lines in Figs. 3 and 4 correspond to those computed by means of the dependences shown in Fig. 9.

Figure 7 compares the increment of strength that is achieved by straining the samples with different amounts of silicon. It is worth noticing that the reported strengthening due to solid solution of silicon is only valid, on average, to the lowest value of strain (0.05). Punctual strengthening as a function of both straining and silicon content as in Fig. 11 was deduced by derivation of the best fit curves as in Fig. 7. The results shown in Fig. 11 indicate that strengthening by silicon increases with deformation but decreases as the amount of silicon increases, such effect is more noticeable towards the higher range of silicon, at which the occurrence of ordered phases is expected to occur.

Examination of optical micrographs of samples prepared from deformed specimens revealed that the amount of twinning present in the material increases with the level of strain and with the amount of silicon added to the material. The distance ($d$) between twins as in Fig. 2 in specimens deformed to an equivalent strain of 0.8, was obtained by the mean intercept technique and it is reported in Table 1. The values of the saturation stress ($\sigma_s$) for the different samples were plotted as a function of the distance between
the twins as in Fig. 12. The best fit curve, dashed line in Fig. 12, corresponds to the relationship:

\[ \sigma_s = 357.5 + \frac{2178}{d^{0.5}} \]

where \( \sigma_s \) is given in MPa and \( d \) in \( \mu \text{m} \). The correlation between the saturation stress with respect to the metallographic data from specimens deformed to a strain of 0.8 is justified as saturation is well achieved at this strain in all samples, see Figs. 1 to 3.

5. Conclusions

The plastic deformation at room temperature of a series of iron–silicon alloys could be described by constitutive equations in which the characteristic parameters depend on the amount of silicon added to them. The constitutive equations were deduced by plotting the work hardening rate as a function of stress. Some deviations from the expected behaviour were found in samples with the higher amount of silicon, but such behaviour can be explained in terms of the
softening produced by the adiabatic rise in temperature.

It was found that twinning of the material occurred with higher frequency as strain and silicon added to the material increased, being possible to relate the strength at saturation with respect to the inverse square of the distance between the twins present in samples deformed up to strains of 0.8.

It was found that the strengthening caused by the solid solution of silicon varied with the amount of this element and straining. Only the unstrained material values close to those previously reported. The lowest punctual values were found to occur towards the higher range of compositions, where formation of ordered phases is expected to occur.

Acknowledgements

RC acknowledges the support provided by CONACYT, Mexico.

REFERENCES