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# High-temperature creep and structure investigation of nearly stoichiometric $Fe_3Si$

## Part 1 *Creep behaviour of Fe–Si single crystals*

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Single crystals of nearly stoichiometric $Fe_3Si$ were creep-deformed at temperature $T = 450$ to 850° C and applied stress $\sigma = 40$ to 250 MPa. While the temperature dependence of the steady-state creep rate of crystals with less than 25 at% Si can be described by an exponential function $\exp(-\Delta H_{exp}/kT)$, the Fe–26 at% Si samples show an exponential dependence only below 500° C and above 600° C. At intermediate temperatures the dependence is weak. It is suggested that in this intermediate range two phases exist. The experimental results are consistent with the assumption that the phase boundaries do not hinder dislocation movement, and that the disocation velocity in the two phases is different.

## 1. Introduction

Fe–Si alloys of higher silicon-content are of primary interest from the viewpoint of magnetic properties, but the deformation behaviour becomes poor with higher silicon-content. The rollability of Fe–5.7 wt% Si (10.7 at% Si) is therefore greatly influenced by ordering effects, because at this concentration the body-centred cubic-derivative ordered structures B2 and $DO_3$ appear [1].

The present paper is part of an examination of the high-temperature creep of Fe–Si single crystals in the whole range of homogeneity for the $DO_3$ structure from 11 to 26 at% Si [2, 3]. It demonstrated the unexpected mechanical behaviour of samples with excess silicon with regard to the stoichiometric composition Fe–25 at% Si (i.e. $Fe_3Si$). To explain this behaviour, X-ray diffraction experiments were carried out on samples with excess silicon (see part 2 [4]).

The mechanical studies of $Fe_3Si$ published so far by other authors were performed not by creep (constant load) but by constant strain-rate deformation [5–8].

## 2. Experimental procedure

The creep deformation was carried out in a compression apparatus in an inert gas atmosphere (argon, 0.1 MPa) at temperatures ($T$) and applied stresses ($\sigma$) ranging from 450 to 850° C and 40 to 250 MPa, respectively. The specimen heating was performed in a tantalum resistance furnace. The displacement was measured by an inductive displacement transducer. The maximum absolute error of temperature measurement was ± 10 K. The accuracies of stress and displacement determination were better than 10 and 5%, respectively.

The samples were spark-machined ($\sim 3\,\text{mm} \times 3\,\text{mm} \times 8\,\text{mm}$) with two orientations: (i) [2 1 0] compression axis and (2 1 0), $(1\,\bar{2}\,0)$ and (0 0 1) faces, and (ii) [1 0 0] axis and (1 0 0), (0 1 0) and (0 0 1) faces. These orientations were chosen because for slip on $\langle 1\,1\,1 \rangle \{1\,\bar{1}\,0\}$ [9] the corresponding Schmid factor is the same for two or four glide systems. This allows a higher total deformation than the orientation for a single slip. Prior to compression, the shaped samples were finished by grinding and mechanical polishing.

The iron and silicon contents of each sample were determined by electron probe microanalysis with elemental standards. The maximum absolute error of concentration measurement amounted to max ± 1.2 at% Si, but the mechanical properties allow a classification into two groups with different behaviours, denoted "excess iron" and "excess silicon".

To determine the temperature dependence of the creep rate, temperature-change tests were performed. The experimental parameters are given in Table I.

## 3. Experimental results

### 3.1. General information

All registered creep curves showed, after a transient creep stage, a well-established steady-state creep stage. The steady-state creep rate ($\dot{\varepsilon}$) was measurable in the region of temperatures denoted in Table I.

### 3.2. Specimens with excess iron

The steady-state creep rate was found to depend strongly on temperature. A typical result is shown in Fig. 1 at two different applied stresses. The temperature dependence of $\dot{\varepsilon}$ can be described by an exponential function $\exp(-\Delta H_{exp}/kT)$, where $\Delta H_{exp}$ is an apparent activation enthalpy. This behaviour agrees with that of the intermetallic compounds $MgZn_2$ [10] and $V_3Si$ [11].

Because no glide lines were observed on the surface of deformed crystals, the glide planes were determined by dislocation etching on {1 0 0} planes. The results agree with the assumption of {1 1 0} glide planes [12]. The usually-assumed glide direction $\langle 1\,1\,1 \rangle$ could not

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TABLE I Experimental characteristics and results

| Specimen No. | Orientation | Silicon concentration (at %) | $\sigma$ (MPa) | $T$ (°C) | $\Delta H_{exp}$ (eV) | |
|---|---|---|---|---|---|---|
| | | | | | $T < 500°C$ | $T > 600°C$ |
| 6/3 | 210 | 24.2 | 80 | 640 to 705 | 2.2 | |
| | | | 90 | 560 to 706 | 2.4 | |
| 16.1/7.3 | 210 | 26.5 | 40 | 660 to 850 | | 1.4 ± 0.2 |
| 16.1/6.4 | 210 | 26.1 | 140 | 510 to 680 | | 3.4 ± 0.3 |
| 16.1/6.2 | 210 | 26.1 | 150 | 505 to 680 | | 4.1 ± 0.5 |
| 16.1/6.3 | 210 | 26.0 | 180 | 560 to 630 | | 8.7 ± 0.9 |
| 16.1/6.4 | 210 | 26.1 | 190 | 470 to 645 | 2.4 ± 0.3 | 3.7 ± 0.5 |
| 16.1/6.2 | 210 | 26.1 | 200 | 517 to 570 | | |
| 16.1/6.3 | 210 | 26.0 | 250 | 450 to 535 | 1.3 ± 0.1 | |
| 16.1/8.3 | 100 | 26.2 | 80 | 490 to 630 | | 7.6 ± 0.8 |

be confirmed by transmission electron microscopy. In creep-deformed samples the Burgers vector $\frac{1}{2}\langle 1\,1\,0\rangle$ was identified [13].

### 3.3. Specimens with excess silicon

In silicon-rich samples the exponential dependence of the steady-state creep rate on temperature is valid only at temperatures below 500° C and above 600° C (Fig. 2). At intermediate temperatures the dependence is weak. This behaviour appears in [2 1 0] samples as well as in [1 0 0] samples.

## 4. Discussion

In iron-rich samples the activation enthalpy is about 2 eV. The structure in the whole investigated temperature range is $DO_3$ according to the equilibrium phase diagram [14]. This value of $\Delta H_{exp}$ corresponds well to the results of Fe–Si single crystals with ~ 13 and ~ 18 at % Si deformed in the range of $DO_3$ structure [3].

According to Kubaschewski [14] the silicon-rich samples should consist of $DO_3$ phase and some ε-phase with B20 structure up to 700° C. This constitution does not explain the unexpected behaviour at intermediate temperatures, which points to the existence of a phase transition in this range between 500 and 600° C.

In Part 2 [4] it is shown that a low-temperature modification and a high-temperature modification exist, both with $DO_3$ structures, and that the high-temperature modification is probably one with a higher degree of electron binding and with higher interactions between spins. For transition temperatures without mechanical loading see Part 2 [4].

To interpret the creep behaviour we assume the existence of the two $DO_3$ phases in the temperature region from 500 to 600° C under the action of mechanical loading. The dislocations may slip from regions with one modification into those with the other modification only by altering their velocity. The mean

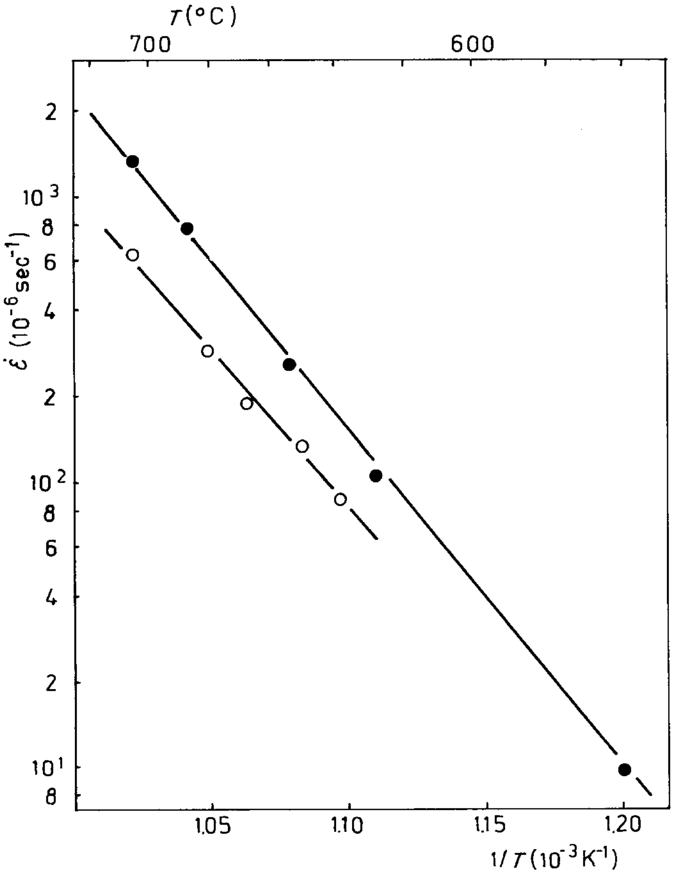

*Figure 1* The influence of temperature ($T$) on the steady-state creep rate ($\dot{\varepsilon}$) in $Fe_3Si$ with excess iron. Specimen 6/3 [2 1 0] with normal stress (○) 80 and (●) 90 MPa.

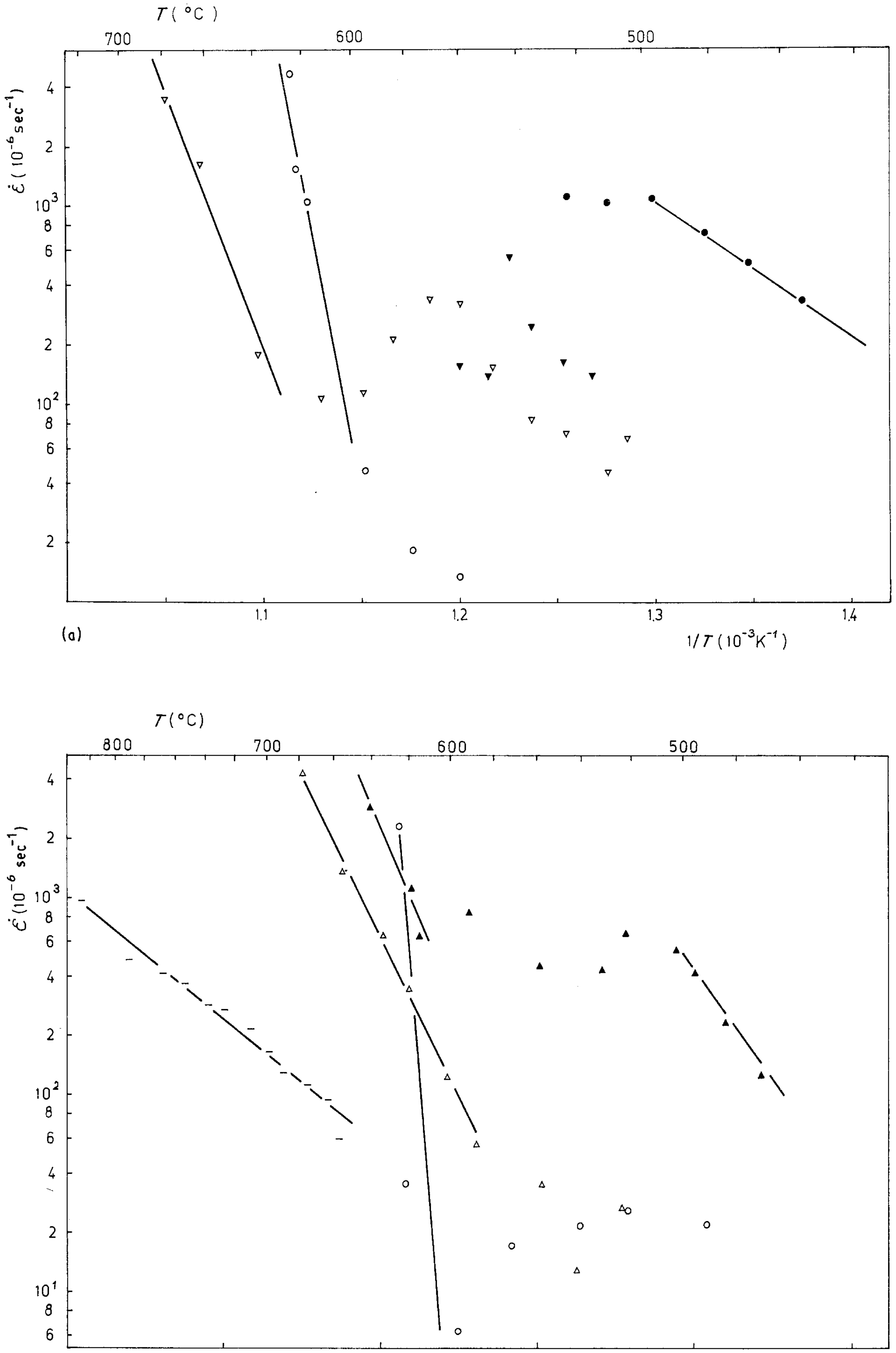

*Figure 2* (a) The influence of temperature ($T$) on the steady state creep rate ($\dot{\varepsilon}$) in $Fe_3Si$ with excess silicon; (▽, ▼) Specimen 16.1/6.2 [2 1 0], (○, ●) Specimen 16.1/6.3; normal stress (▽) 150, (○) 180, (▼) 200, (●) 250 MPa. (b) The same for (—) Specimen 16.1/7.3 [2 1 0], (△, ▲) Specimen 16.1/6.4 [2 1 0], (○) Specimen 16.1/8.3 [1 0 0]; normal stress (—) 40, (○) 80, (△) 140, (▲) 180 MPa.

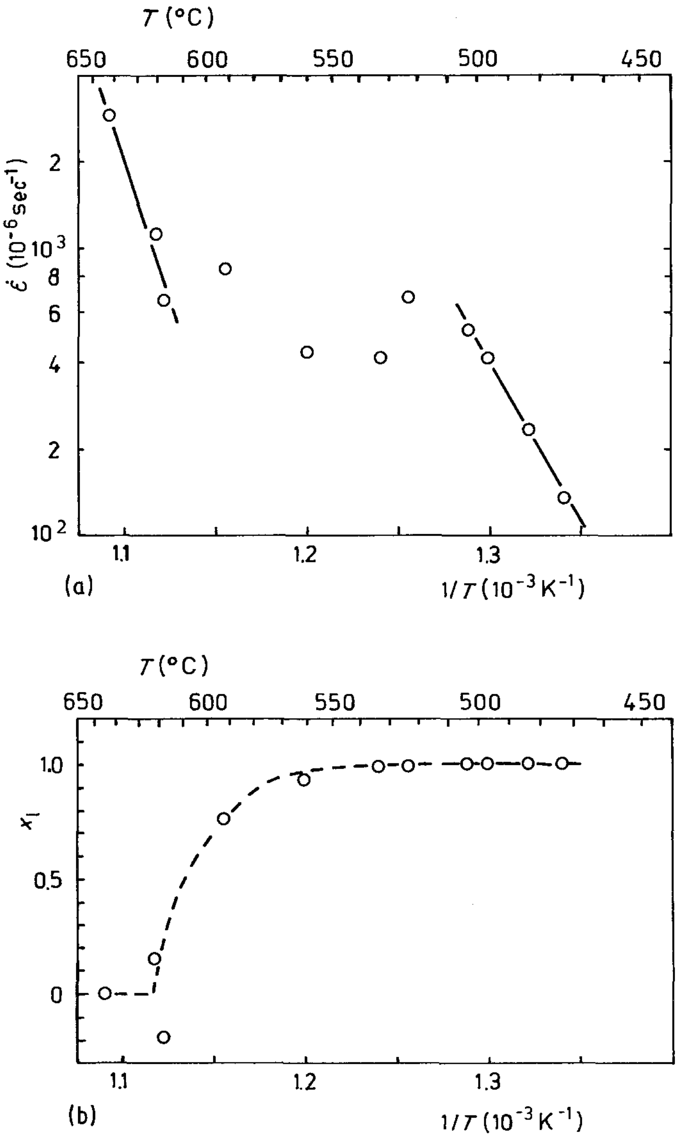

*Figure 3* (a) Steady-state creep rate ($\dot{\varepsilon}$) and (b) calculated volume fraction of the low-temperature phase ($x_l$), plotted against temperature for 16.1/6.4. Normal stress 190 MPa.

velocity $\bar{v}$ results from the velocities $v_l$ and $v_h$ in the low- and high-temperature phases, respectively, according to $1/\bar{v} = x_l/v_l + x_h/v_h$, at which $x_l$ and $x_h$ are the volume fractions of the two phases. If the Schmid factor $\mu$, the dislocation density $\varrho$ and the Burgers vector $\boldsymbol{b}$ are equal in both DO$_3$ phases, it follows from the Orowan equation $\bar{\varepsilon} = \mu\varrho v$ for the total creep rate $\dot{\varepsilon}$ that

$$\frac{1}{\dot{\varepsilon}} = \frac{x_l}{\dot{\varepsilon}_l} + \frac{x_h}{\dot{\varepsilon}_h} = \frac{x_l}{\dot{\varepsilon}_l} + \frac{1 - x_l}{\dot{\varepsilon}_h} \qquad (1)$$

$\dot{\varepsilon}_l$ and $\dot{\varepsilon}_h$ are the creep rates of these phases extrapolated from the regions $T < 500°\,\mathrm{C}$ and $T > 600°\,\mathrm{C}$ according to the formula $\dot{\varepsilon}_{l,h} = \dot{\varepsilon}_{0_{l,h}} \exp(-\Delta H_{\exp_{l,h}}/kT)$.

The values of $\Delta H_{\exp}$ for the two phases are seen in Table I. For $T < 500°\,\mathrm{C}$ the order of magnitude is the same as for the samples with excess iron. For $T > 600°\,\mathrm{C}$ the values are higher, except that determined at the extreme low-stress level of 40 MPa.

A proof of the model was possible for Sample 16.1/6.4 deformed at 190 MPa. $\dot{\varepsilon}_{l,h}$ was found to be given by

$$\dot{\varepsilon}_l = 27.8\,\mathrm{sec}^{-1} \exp(-2.4\,\mathrm{eV}/kT) \qquad T < 500°\,\mathrm{C}$$

$$\dot{\varepsilon}_h = 41.4\,\mathrm{sec}^{-1} \exp(-3.7\,\mathrm{eV}/kT) \qquad T > 600°\,\mathrm{C}$$

For the temperature range between 500 and 600° C the amount of the low-temperature phase $x_l$ was calculated from Equation 1 using the extrapolated values of $\dot{\varepsilon}_l$ and $\dot{\varepsilon}_h$ and the experimental creep rate $\dot{\varepsilon}$. The result of this calculation is seen in Fig. 3. The reduction of $x_l$ with rising temperature shows a satisfactory dependence and gives a certain confidence in the proposed explanation.

It should be noticed that the creep rate is much more sensitive to the phase transformation from the low-temperature modification to the high-temperature modification of the DO$_3$ structure than the physical properties described in Part 2 [4].

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