Dilatation measurements for the study of the $\alpha/\gamma$ transformation in pure iron in high magnetic fields

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**Abstract.** Magnetic field processing is a new promising tool for the structural and functional control of materials. A significant potential exists for tailoring microstructures and impacting kinetics of phase transformation in steels. A high magnetic field modifies the Gibbs free energy. As a result, the phase diagram is shifted upwards so that the $A_c1$ and $A_c3$ temperatures increase as the magnetic field is increased. In this work, a new device for the heat treatment and in situ control of the transformation is described. For the first time, a dilatation measurement is used to study the shift of the ferrite/austenite equilibrium in high magnetic field up to 16 T and to quantify the ferrite concentration during the transformation. Experimental results for the transformations in pure iron are presented. Comparisons are made with the expected values based on the Weiss molecular field model near the Curie point.

1. Introduction

As far as solid state transformations in ferrous alloys are concerned, the magnetic field is not only expected to act on the difference in magnetic moments of the parent and product phases but also on induced magnetic anisotropy [1], transformation kinetics [2] or microstructure of the product phase [3]. As far as the austenite to ferrite transformation in steels is concerned, it was mainly found that the transformation was accelerated [4] and the nucleation rate of ferrite was increased [4,5] by the magnetic field and that a ferrite grain alignment may occur in certain conditions [4]. More recently, thermodynamic calculation based on molecular field theory has been made on phase equilibria of pure Fe [6] and several Fe-C based alloys [7,8] under high magnetic field, showing that the $\gamma/\alpha$ equilibrium temperatures increase with increasing magnetic field. Consequently, a need for experimental evidence of this result has emerged. Hao et al. thermically measured the increase with the magnetic field of the $\gamma/\alpha$ transformation temperature in pure iron and in one Fe-C alloy [9]. Miyauchi et al. used resistivity measurements on pure iron to confirm this effect on both the $\gamma/\alpha$ and the $\alpha/\gamma$ transformation up to 10 T [10].

In this work, we suggest a new experimental technique based on dilatometry under high magnetic field. This technique allows to accurately monitor the solid state transformations in real time, so that the ferrite concentration during the transformation in magnetic field can be easily quantified.

2. Experimental details
The experimental setup used in this study is a home made in-situ dilatometry measurement setup installed in a furnace dedicated to the heat treatments of steels (see figure 1). The whole device is operating under magnetic fields up to 16 T. The room temperature bore available in this magnet is 32 mm large. The system is screened by a copper water-cooled jacket. The resistive furnace is kept under vacuum or controlled atmosphere together with the sample holder. Homogeneous temperature and field are applied to the 1 cm high sample placed in the magnet centre region.

Specimens are kept at 950 °C for 30 min for homogenization before the measurements. The magnetic field is applied during the whole thermal treatment and the furnace in under pure argon atmosphere. The samples are heated with a heating rate of 3 °C/min between 890 and 940 °C. The same heating rate is applied on cooling between 940 and 890 °C. Both the $\alpha$- and $\gamma$-transitions are studied and each measurement is repeated twice. Below 890 °C, the cooling rate is about 30 °C/min down to room temperature.

The dilatation measurement is realized by laser interferometry (Michelson interferometer from the Sageis CSO Company) in the near infrared, with a resolution below 0.1 μm. The laser beam is reflected on a mirror placed on top of the alumina sample holder. It crosses an airtight window on top of the furnace chamber. This direct dilatation measurement method is based on the displacement of optical interference fringes between the reflected beam and a reference beam. The background signal, resulting from the contribution of the sample holder is deduced for each measurement.

Figure 2 shows the dilatation of a pure iron sample during a complete heat treatment. The $\alpha$-transition (respectively $\gamma$-transition) appears as a sharp contraction (respectively dilatation) of the sample. The volume change is about 1%. The dilatation coefficients of each phases (12×10⁻⁶ K⁻¹ for ferrite and 20×10⁻⁶ K⁻¹ for austenite) can be measured.

![Figure 1. Experimental setup.](image-url)
3. Results and discussion

The ferrite volume fraction versus temperature can be deduced from the dilatation curve, as shown on figure 3 for two samples treated with or without magnetic field. For each sample, the $A_{c3}$ and $A_{r3}$ temperatures are measured at the beginning of respectively the heating and cooling curves. Figure 3 clearly shows that both the $\alpha/'$ and $\alpha/\prime$ transformation temperatures increase with increasing magnetic field. The experimental equilibrium temperature defined, in first approximation, by $A_3 = (A_{c3} + A_{r3})/2$ can then be plotted as a function of the magnetic field intensity.

\[ A_{c3}(0\ T) = 914 \, ^\circ C \quad A_{c3}(9\ T) = 919 \, ^\circ C \]
\[ A_{r3}(9\ T) = 912 \, ^\circ C \quad A_{r3}(0\ T) = 906 \, ^\circ C \]

Figure 3. Ferrite volume fraction as a function of temperature deduced from dilatation measurement for two samples treated in 0 T (grey curve) and 9 T (black curve).

Figure 4 shows the shift of the equilibrium temperature, $A_3 = A_3(H) - A_3(0 \ T)$ under magnetic fields. The open circles correspond to the experimental data of this study. Experimental data as measured by Miyauchi et al. [10] are plotted as open squares. The magnetic energy contributes to the
Gibbs free energy of both ferrite and austenite. As ferrite is more magnetic than austenite, this phase is stabilized to higher temperature, so that a shift in the equilibrium temperature is obtained.

Because the $\alpha/\gamma$ transformation in pure iron occurs above the Curie temperature, the ferrite is in the paramagnetic state. The observed shift of the equilibrium temperature is nearly proportional to the square of the magnetic field and amounts to approximately 16 °C in 16 T. The calculated equilibrium temperature, using the Weiss molecular field theory [2] is also shown with solid circles. On figure 4, a very good agreement is observed up to 16 T between the experimental data and the calculated equilibrium temperature.

![Figure 4](image)

**Figure 4.** Shift of the $\alpha/\gamma$ equilibrium temperatures in pure iron as a function of magnetic field intensities. The experimental data (open marks) are compared with the calculated ones (solid marks).

**Conclusion**

In this work, a new experimental method based on in-situ dilatation measurements has been used to monitor the solid state transformations in steels. Both the $\alpha/\gamma$ and $\gamma/\alpha$ transformation temperatures can be deduced from this measurement. The ferrite concentration during the transformations can also be accurately and quantitatively determined. As an illustration, experimental evidence for the increase of the equilibrium temperature with magnetic fields up to 16 T is shown in pure iron. The experimental data are in good agreement with the calculated results based on the Weiss molecular field theory.

**References**

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