Ferrite Transformation in High Magnetic Field Monitored by *in-situ* Magnetic Measurements

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For the first time, *in-situ* high temperature magnetic measurements are used to monitor the solid state transformations in steels. The magnetization provides information about the kinetic of the austenite to ferrite transformation, although care must be taken in the experimental procedure and in the data analysis. The isothermal transformation of ferrite following two regimes under magnetic field is confirmed together with the effect of the magnetic field on the nucleation of this phase.

KEY WORDS: magnetic field effect; ferrite; magnetic susceptibility measurement; nucleation.

1. Introduction

Magnetic fields influence thermodynamic equilibrium when the magnetic energy is comparable to the enthalpy of transformation. Although usually negligible at high temperature in the usual magnetic field intensities, the magnetic term becomes determinant when the transformation is associated with a ferromagnetic transition. Such an effect has been evidenced in solid-gas reactions¹⁾ and in the austenite to ferrite transformation in steels.^{2–4)} In both cases, the stability of the ferromagnetic phase is promoted by the high magnetic field. In steels, the shift of the Fe–C phase diagram to higher temperatures has been calculated.^{5,6)} In this study, austenite to ferrite (γ/α phase) transformation of a Fe+0.15C+1.42Mn+0.77Ni+1.7eqCr steel in magnetic field is investigated by means of *in situ* susceptibility measurement and the effects of magnetic field on transformation behaviour and structures are analysed. *In situ* magnetic measurement is a powerful tool to control materials processing in high magnetic field in real time.^{7,8)} Such measurements in fields up to 2 T have been recently used by Ono *et al.* in a range near the α/γ transformation temperature in steels.⁹⁾ The home made device presented in this work allows measuring the susceptibility in the whole process temperature range³⁾ (thus below the Curie temperature of ferrite) in field up to 4 T.

2. Experimental Methods

The characterization method used in this study is a magnetic susceptibility measurement at high temperature. A home-made device (shown in Fig. 1) enables the measurement of the magnetization during a complete heat treatment. The magnetization is measured by the magnetic force exerted on a sample in a field gradient.^{3,10)} The measuring

device together with the furnace are inserted in the vertical room temperature bore (120 mm diameter) of an 8 T superconducting magnet.

The measurement of the magnetic force derives from the weighing of the sample being freely suspended to the mechanical part of a highly sensitive balance (0.001 g in the range of 1 200 g). When the magnetic field is applied, the weight of the sample corresponds to the addition of the gravity and the magnetic force contribution. The signal of the sample holder is measured for background removal. The samples are about 1 cm high and 5 mm diameter, and the precision of the measurement is below 10^{-6} emu/g for

Fig. 1. Schematic view of the high temperature magnetic susceptibility measurement device (left), schematic view of the local magnetic forces on a ferromagnetic sample (right). The superconducting magnet provides a vertical magnetic field with a maximum value of 8 T in the magnet center and about 4 T on the sample. The magnetization of the sample is derived from the magnetic force measured by an electronic balance.

the susceptibility in the paramagnetic state. Because the steel sample becomes ferromagnetic in the ferritic state, very large forces arise in high fields and special care must be taken to suspend it in the field gradient.

The whole system (including furnace, sample holder and weight measuring device) is under controlled atmosphere (secondary vacuum or inert gas). The electronic part of the balance is kept away from the influence of the magnetic field. The sample, suspended to the balance, is centered inside a hollow molybdenum tube heated by a radio-frequency induction coil (120 kHz, 6 kW). The whole system is screened by a copper water-cooled jacket. Special care must be taken to the position of the heating element. Homogeneous temperatures must be applied to the sample placed in the maximum field gradient. Therefore, the heating element can be vertically translated to meet this requirement. The sample holder is a Boron Nitride cylinder suspended below the balance by two alumina rods also containing the thermocouples.

In the magnet center region, the higher field is located near the edges of the superconducting coil. A sample with positive magnetic susceptibility (ferromagnetic or paramagnetic) is then expelled away from the magnet vertical axis by radial forces and goes into contact with the furnace heating element. In this case and because of friction, any accurate measurement of the vertical magnetic force exerted on the sample is impossible. On the contrary, far above the center field region the magnet is seen as a dipole so that the maximum field is on the vertical axis. A ferromagnetic (*i.e.* paramagnetic) sample is then self-centered on the axis of the magnet, so that it does not go into contact with the heating element. In this position, the sample is submitted to a much smaller magnetic field but the corresponding down-

ward vertical force is very high. **Table 1** summarizes the values for the magnetic field and magnetic field gradient used in this study.

The raw samples have a bainitic microstructure with a small 10% residual austenite content. In the experiments, they are first heated up to 1 000°C for 10 min for austenitization. The heating and cooling rate are 1°C/s. The isothermal ferrite growth takes place on cooling during a dwell at 650°C for 16 h. The samples are submitted to 0.1 or 4 T during the whole heat treatment.

3. Results and Discussion

Figure 2 shows the raw data of magnetization versus temperature for two values of the applied field. In 0.1 T, on heating, the sample magnetization is almost constant until a sharp decrease around 750°C (see part 1 on Fig. 2). However, for the same heating in 4 T, the sample magnetization continuously decreases (from a starting higher value) and is followed by a broad transition around the same temperature. The broadening of the transition in the higher field is typical of a ferromagnetic transition, thus attributed to the ferrite content in the sample. The lower value of the magnetization in 0.1 T indicates that the sample is not fully saturated: the value of the magnetization is then strongly dependent on the demagnetizing field and hence on the shape of the sample. Meanwhile, the magnetic transition corresponding to the bainite/ferrite to austenite transformation can not be evidenced: in 0.1 T, the signal is too weak (compared to the Curie transition) to evidence the transformation and in 4 T, it is masked by the wide ferromagnetic transition.

On cooling, the paramagnetic signal indicates that the

Table 1. Characteristics of the experimental magnet conditions.

Magnetic induction in the center region (T)	Magnetic induction exerted on the sample (T)	Field gradient exerted on the sample (T/m)
0.3	0.152	0.9139
8	4.042	48.23

Fig. 2. Magnetization *versus* temperature in 0.1 T (left) and 4 T (right). (1) Ferromagnetic to paramagnetic transition of the ferrite and bainite/ferrite to austenite transformation. (2) Ferrite growth at constant temperature (650°C). (3) Bainite formation from the remaining austenite.

Fig. 3. Isothermal growth of ferrite versus time in 0.1 T and 4 T. The normalized ferrite fraction 1 corresponds to the starting material.

sample remains in the austenite state down to 650°C where an isothermal 16 h dwell is applied (part 2 on Fig. 2). The magnetization then slowly increases at constant temperature as the ferrite content increases. On further cooling, the large magnetization increase is then attributed to the bainite formation around 400°C (part 3 on Fig. 2).

The ferrite concentration during the isothermal transformation can be derived from the continuous magnetization increase during the 650°C dwell shown on Fig. 2. The estimated ferrite content versus time is presented in Fig. 3 for 2 samples processed in 0.1 T and in 4 T. At 650°C, in a first approximation, we consider the magnetization of ferrite as being the only contribution to the total magnetic signal. This value is proportional to the ferrite fraction in the sample. To overcome the demagnetization field effect and the non saturation in low field (0.1 T), the measured magnetization upon cooling is divided by the value at 650°C during heating to allow quantitative comparison between the samples in low and high field. The estimated value is not then the actual ferrite fraction, but proportional to it. Assuming that no transformation occurs from room temperature up to 650°C, this normalized ferrite fraction is equal to 1 for the starting material (full transformation).

A larger amount of ferrite is formed during the dwell in 4 T as compared to the sample processed in 0.1 T, showing the promotion of ferrite formation by the magnetic field, as already observed in Ref. 3). The increase in the normalized ferrite fraction follows two regimes in both samples. At the beginning of the dwell and for the sample treated in 4 T, the increase is very sharp up to 2.7 h. In the sample treated in 0.1 T, only a minor change of the slope is observed after the same time duration. The faster increase at the beginning of

the dwell is thus correlated to the intensity of the applied magnetic field. Above 2.7 h, the increase is similar in both samples.

In a previous study,⁴⁾ image analysis of polished samples cut after high field isothermal processing showed that the ferrite nucleation rate was promoted by the magnetic field. The number of nuclei was doubled in a field of 16 T (compared to 0 T).

In the present study, it is also confirmed that the appearance of the first ferrite nuclei corresponds to the beginning of the dwell since no ferrite formation shows up before the dwell in the magnetic measurement. In addition, it is shown that the magnetic field has an effect during the first stage of the ferrite transformation, suggesting a larger effect on nucleation rather than on growth.

4. Conclusions

The *in-situ* magnetic measurement is a powerful technique for the control of the ferrite growth during the austenite to ferrite transformation in steels. Care must be taken to avoid demagnetizing shape effects and non saturation of the magnetization to compare results in low field and in high field. The isothermal transformation of ferrite following two regimes is confirmed together with the effect of the magnetic field on the nucleation of this phase.

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