Grain-size dependent austenite–ferrite phase transformation behavior in pure iron

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A process of great interest in the production of steel is the austenite (γ) to ferrite (α) phase transformation during cooling after hot rolling steel slabs. Since this inevitable solid-state transformation controls the final microstructure of the products, and hence the yield strength, it has been studied extensively both from a technological application and a basic scientific point of view [e.g., 1–7].

Many efforts were made to model this transformation in the past decades, but some vital kinetic information was unnoticed. Abnormal $\gamma \rightarrow \alpha$ transformation behavior in substitutional Fe–Co and Fe–Mn alloys (dependent on the grain size of the initial austenite phase) was recently recognized for the first time [8–10]. With the aid of high-resolution dilatometry and differential thermal analysis (DTA), two kinds of transformation kinetics, normal and abnormal, for the $\gamma \rightarrow \alpha$ transformation were detected in the substitutional Fe-1.79 at.%Co alloy [8]. It was found that the normal transformation exhibited one maximum in the transformation rate curve and that the abnormal one showed more than one maximum [8]. Further investigation [9] demonstrated that a large austenite grain size is a precondition for the occurrence of abnormal transformation kinetics.

The $\gamma \rightarrow \alpha$ transformation in the investigated Fe–Co and Fe–Mn alloys was considered as a partitionless transformation, i.e., occurring without any redistribution of the alloying element [8, 9]. It may be questioned if any redistribution of alloying elements (Co, Mn) did occur and was, somehow, the explanation for the occurrence of the abnormal transformation. To rule out such an effect, in this study the $\gamma \rightarrow \alpha$ transformation in pure iron was investigated and it will be demonstrated that the abnormal transformation also occurs in this case.

Bulk iron rods, of 6.3 mm in diameter and a purity of 99.98 wt%, were employed for the present study. The composition of the iron is listed in Table I. The as-received Fe ingots were hammered down to rods of 5.5 mm diameter. In order to achieve a homogeneous microstructure all rods were sealed in a quartz container filled with argon gas at 1.9×10^4 Pa, annealed at 1473 K for 10–100 h (a change in the annealing time at this high temperature leads to a change of the final grain size such that the longer the annealing time, the larger the grain size) and cooled down to ambient temperature within the furnace. The specimens, heat treated at 1473 K for different annealing times, named as A (100 h, 439 μm), B (70 h, 372 μm), C (50 h, 288 μm), and D (40 h, 273 μm), were used to investigate the influence of grain size on the $\gamma \rightarrow \alpha$ transformation behavior of pure iron.

A Baehr DIL 802 differential dilatometer was used to record the length changes of the samples. With this instrument the difference between the sample and an inert reference sample is measured, which results in a high-resolution of about ± 0.01 μm. The dilatometer was calibrated according to the method described in Ref. [11]. The measurements were performed under flowing high purity argon to protect the specimens from oxidation.

The ferrite grain sizes after each heat treatment cycle of the fully transformed Fe specimens were analyzed using light microscopy. The ferritic grain boundaries were revealed by etching with a 2.5 vol% nital solution. The line-intercept method [12] was employed in three different directions along the cross section to determine the mean grain size.

Pure iron specimens with different grain sizes were prepared for isochronal dilatometric measurements. The length changes recorded for specimens of different grain size, during one heating and cooling cycle, are shown in Fig. 1a. The initial length (L_0) of the samples was about 10 mm. During continuous heating, the specimen expands gradually before the onset of the $\alpha \rightarrow \gamma$ transformation, which is associated with a length contraction. After completion of the transformation, the normal thermal expansion and contraction of austenite occurs upon continued heating and subsequent cooling. After completion of the $\gamma \rightarrow \alpha$ transformation upon cooling, associated with a length increase, the normal thermal contraction of ferrite can be observed upon continued cooling down to room temperature.

The high temperature part of the curves are shown in Fig. 1b. The transformation upon heating is associated with the (inhomogeneous) build up of the α/γ misfit deformation energy. After completion of the $\alpha \rightarrow \gamma$ phase transformation upon heating, this misfit strain energy relaxes, which corresponds to the length reduction on top of the length increase due to thermal expansion (see arrows in Fig. 1b). Therefore the slope of the recorded length change of austenite upon continued heating after the $\alpha \rightarrow \gamma$ transformation is not constant. After holding at 1223 K for 30 min after the heating up, the misfit strain energy is fully relaxed. Then, upon subsequent cooling, only normal contraction of

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TABLE I Chemical composition of the iron used (unit: ppm, as provided by Aldrich Chemical Company)

Element	Si	Cu	Ti	C	Fe
Content	13	1	0.6	14	Balance

austenite occurs and the correspondingly recorded dilation data reflect (only) the thermal linear contraction of austenite.

As pointed out in Ref. [13], the small length change ($<0.15\ \mu$m in L_0) due to one measurement cycle, i.e., $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformation, is not accompanied by a density change of the sample. The growth of the product phases during transformation, i.e., γ and α phase, is not completely isotropic: the growth of the phases along the axis direction is slightly different from that in other directions of the cylindrical sample.

For the small overall change of the sample after one measurement cycle, the dilatation of the sample during phase transformation can be considered to be largely isotropic. Then, from the data of relative length change, $\Delta L/L_0$, during the (nonisothermal) transformation, the fractions of γ and α phase can be calculated by the lever rule. To investigate the influence of grain size on the $\gamma \rightarrow \alpha$ transformation kinetics, the dilation behaviors of Fe specimens with different initial austenite grain sizes were measured.

The values determined for the ferrite fraction, f_α, are illustrated for different Fe specimens as a function of temperature in Fig. 2. The corresponding transformation rate, $\mathrm{d}f_\alpha/\mathrm{d}t$, for the different pure iron specimens are shown in Fig. 3a and b as a function of temperature and ferrite fraction, respectively.

According to the $\mathrm{d}f_\alpha/\mathrm{d}t$ data (Fig. 3), two kinds of transformation kinetics, abnormal and normal, are easily recognized. Specimen A, with the largest grain size, exhibits distinctly abnormal transformation behavior as revealed by the occurrence of three maxima in the transformation rate curve (cf. the earlier results reported for Fe–Co in Ref. [8]). Specimens C and D, with the smallest grain size, exhibit only one maximum for $\mathrm{d}f_\alpha/\mathrm{d}t$, which is typical for normal transformation behavior.

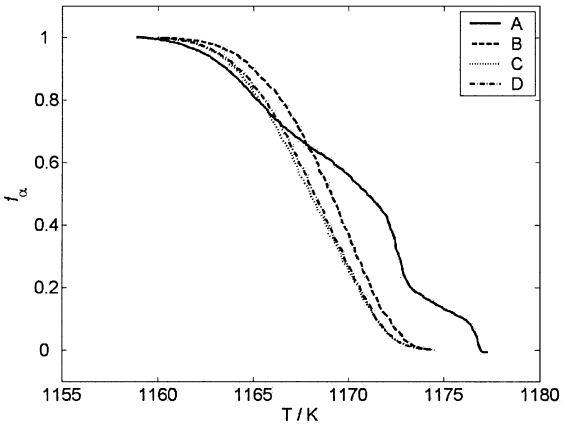

Figure 2 The ferrite fraction, f_α, as a function of temperature, T, calculated from dilatometric measurements of different Fe specimens subjected to cooling from the austenite-phase field.

The starting temperature of the abnormal $\gamma \rightarrow \alpha$ transformation (specimen A) is slightly higher than that of the normal transformation (specimens C and D). The first maximum in the first part of the $\mathrm{d}f_\alpha/\mathrm{d}t$ curve of specimen B corresponds to the abnormal transformation behavior (Fig. 3).

It follows that a relatively large austenite grain size is a precondition for the occurrence of the abnormal transformation kinetics; the kinetics changes from abnormal to normal with decreasing (austenite) grain size of the pure iron.

In conclusion, the occurrence of both abnormal and normal transformation kinetics was recognized for the first time for the $\gamma \rightarrow \alpha$ transformation in iron. Abnormal transformation kinetics is exhibited by the presence of more than one maximum in the transformation rate curve, as compared to the presence of only one maximum in the transformation rate curve for normal transformation kinetics. The observation of abnormal transformation kinetics for pure iron excludes a decisive role for solute drag and solute diffusion in the occurrence of abnormal transformation kinetics. A prerequisite for the emergence of abnormal transformation kinetics is a large initial austenite grain size. The transformation behavior changes from abnormal to normal upon decreasing austenite grain size.

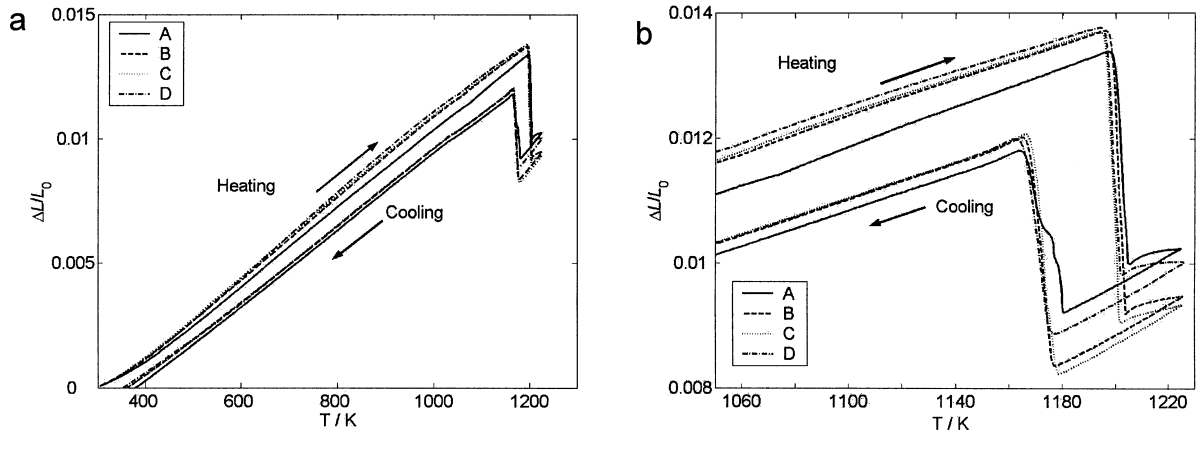

Figure 1 Measured relative length change, $\Delta L/L_0$, of pure iron with different grain sizes (coded as A, B, C, and D) as function of temperature, T: (a) entire range and (b) expanded view of transformation region.

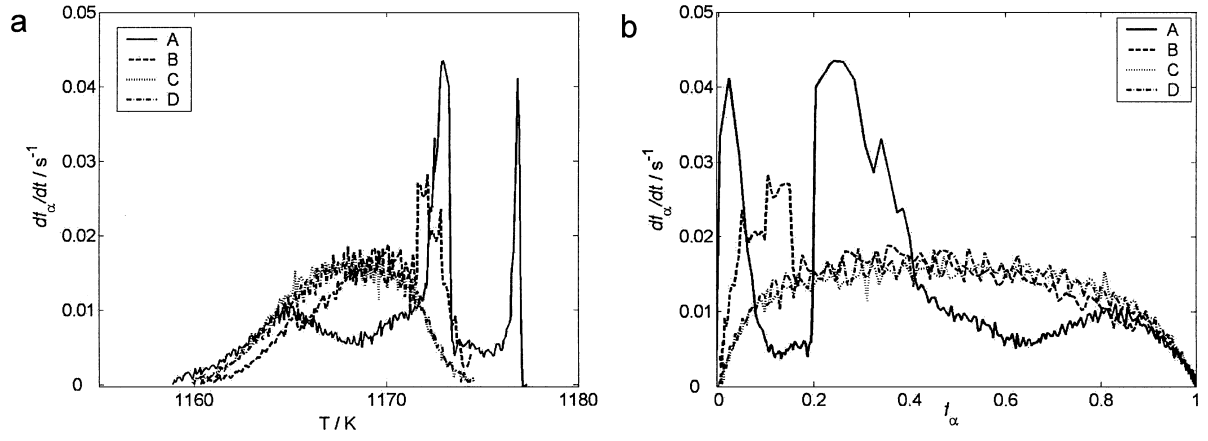

Figure 3 The ferrite transformation rate, $\mathrm{d}f_\alpha/\mathrm{d}t$, as a function of (a) temperature, T, (b) ferrite fraction, f_α, of different Fe specimens A, B, C, and D as determined from dilatometric measurement (corresponding to the data shown in Fig. 2).

Acknowledgments

The authors are grateful to the Foundation for the Author of National Excellent Doctoral Dissertation of PR China (FANEDD), and the National Natural Science Foundation of Tianjin City for grant and financial support.

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Received 15 December 2003
and accepted 12 January 2004