

Dilatometric Analysis of Irreversible Volume Change during Phase Transformation in Pure Iron

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Abstract: One assumption underlying the conventional dilatometric analysis based on the lever rule is that the volume of the specimen changes isotropically during phase transformation, which conflicts with the irreversible length change shown in actual measurements. The contribution of this irreversible effect to the dilation data of pure iron upon heating and cooling was respectively quantified via conversion equations based on lattice parameters. A model considering the elastic strain and creep deformation was established for both the interpretation of the irreversible volume change and the discrepancy between the results measured by a dilatometer and a micrometer.

Key words: dilatometry; irreversible volume change; austenization; austenite decomposition; lattice parameter

Dilatometry is widely used for studying solid-solid phase transformation in steels, because it could monitor the dimensional change of the sample in real time, thus providing the details of the relationship between phase transformation and the given thermal history^[1,2]. The quantitative calculation of phase fractions from the dilatometric curve based on the lever rule is a common method for validating phase transformation theoretical models^[3,4]. Generally, the dilatometric curve of ferrous metal specimens upon heating or cooling could be divided into three segments: the linear segment before the phase transformation, the non-linear segment during phase transformation, and the linear segment after the phase transformation. In the lever rule, the two linear segments below the transformation starting temperature and above the transformation finishing temperature are extracted from the dilatometric curve and linearly fitted respectively. Thus, fractions of the transformed and transforming phases could be well evaluated from the relative positions of the measured curve and those fitted lines^[5].

Assuming that the isotropical volume changed during phase transformation, several scholars built

verification models and applied them to calculate the phase fractions by analyzing the average atomic volumes based on the lever rule^[6-12]. However, the volume change would not occur isotropically in dilatometric specimens during phase transformation in actual measurements, which conflicted with the important assumption in previous analysis models. The neglecting of the irreversible effect may cause a critical error of phase fractions calculated from a dilatometric curve with the lever rule, thus making the dilatometric validation of phase transformation theoretical models unreliable. The irreversible length change is commonly observed during thermal cycling of two-phase materials due to the difference in thermal expansion coefficients between the phases^[13]. Previous studies have reported that transformation plasticity is responsible for the dimensional change of specimens undergoing thermal cycling^[14], but the knowledge of the irreversible volume change still remains controversial.

In order to avoid the unnecessary influence of carbon redistribution on lattice parameters and the difficult confirmation of the pearlite transformation start temperature^[15], the irreversible volume change

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in pure iron specimens undergoing different numbers of thermal cycles was studied in this paper. The necessity of considering the irreversible contribution into dilatometric analysis was highlighted and a model considering both elastic and plastic deformation was established to interpret the cause of irreversible volume change.

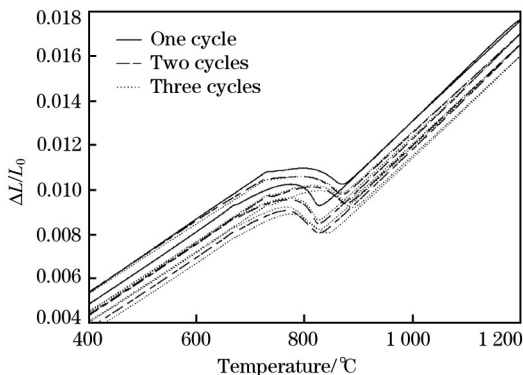
1 Experimental Procedure

Cylindrical specimens were machined from a rolled plate of DT4 pure iron ($C \leq 0.04\%$, $99.50\% \leq Fe \leq 99.90\%$) along the same direction and then polished to a length of 10 mm and a diameter of 5 mm (± 0.001 mm). The dilatometric measurements were performed using a dilatometer (German, NETZCH. Corp, DIL402) equipped with a radiation furnace for heating. The length change was measured using a linear variable displacement transducer (LVDT). The dilatometric specimens were heated to 1200 °C and cooled to room temperature at a rate of 0.2 °C/s as a thermal cycle. The thermal cycle was executed once, twice, and three times, respectively. The results were rectified to eliminate the error caused by the dilation of the sample holder according to the baseline acquired from the measurement of a standard sample (Al_2O_3) undergoing the same thermal history, ensuring a measurement error less than 1.25 nm.

2 Results and Discussion

2.1 Dilatometric curves

As shown in Fig. 1, similar trends are manifested in the measured dilatometric curves of pure iron specimens undergoing repeated thermal cycles, but small deviations along both the temperature and the relative length scales are shown at each thermal cycle. The open loops of each dilatometric curve indicate that the specimen length decreased after thermal



ΔL —Length increment; L_0 —Initial specimen length.

Fig. 1 Dilatometric curves of pure iron specimens undergoing different numbers of thermal cycles

cycles, which directly displays the irreversible contribution to the dilatometric curve. A slight increase of A_{c3} temperature (austenite finish temperature) after each thermal cycle was observed. The same phenomenon has been reported in previous dilatometric studies^[14,16]. The reason is not clear, and probably is the microstructure change.

As shown in Fig. 2, the final specimen length measured with a micrometer after thermal cycling is compared with that obtained from the dilatometric curve. The amount of the irreversible length change is not the same at each thermal cycle. However, a larger reduction of the specimen length is consistently shown in dilatometric measurement compared to that of micrometer. Similar results were reported by other researchers^[14,15]. The reason for this discrepancy will be discussed later.

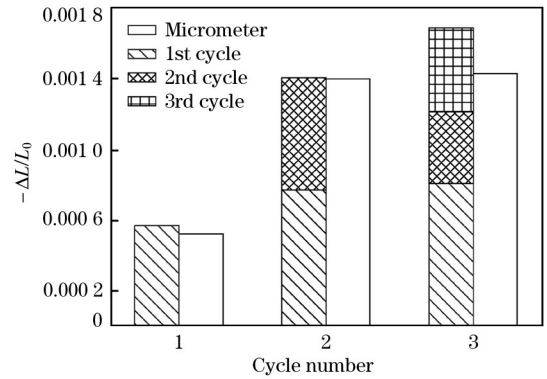


Fig. 2 Irreversible length change of tested specimens

2.2 Irreversible length change upon heating and cooling

For pure iron, the average atomic volume can be represented by a linear combination of the atomic volumes of the constituent phases as follows:

$$V = f_a V_a + f_\gamma V_\gamma \quad (1)$$

where, V is the average atomic volume; V_a and V_γ are the average atomic volumes of the ferrite and austenite phases, respectively; and f_a and f_γ are the fractions of these phases, respectively.

Compared to the initial volume, the volume change during transformation is small and could be assumed isotropic, thus the relationship of the average atomic volume change (ΔV) and the length change is described as follows:

$$\frac{\Delta L}{L_0} = \frac{1}{3} \cdot \frac{\Delta V}{V_0} = \frac{1}{3} \left(\frac{V}{V_0} - 1 \right) \quad (2)$$

where, V_0 is the reference average atomic volume.

According to Eq. (2), ΔL is expressed as follows:

$$V = a V_0 \left(\frac{3\Delta L}{L_0} + 1 \right) \quad (3)$$

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