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Modified dilatometric technique for determining the austenitizing kinetics of a low alloy Mn–Mo–Ni pressure-vessel steel

P. G. H. PISTORIUS, G. T. VAN ROOYEN
Department of Materials Science and Metallurgical Engineering, University of Pretoria, Pretoria 0002, South Africa

While dilatometry is a powerful technique to study the kinetics of phase transformations, the fact that both the thermal expansion (or contraction) and dimensional changes due to phase transformations are measured simultaneously limits the sensitivity of this method. As the thermal expansion is largely linear, it is possible to compensate for this contribution to the dimensional change of the specimen. A technique based on this approach was developed and used to study the austenitizing kinetics of a low alloy Mn–Mo–Ni pressure-vessel steel. Owing to the increased sensitivity it was possible to establish that austenitizing started at a temperature much lower than the temperature normally assumed to be the A_{c1}. This has implications for the maximum tempering temperature. Possible metallographic evidence for austenitization at a temperature lower than the temperature normally assumed to be the A_{c1} is presented.

1. Introduction

Dilatometry is the obvious technique to establish the coefficient of thermal expansion [1], measure thermal strain during the thermal cycling of materials [2] and determine the internal stresses in metal matrix composites [3]. It is one of the preferred techniques for studying the kinetics of phase transformations where volume changes are associated with the transformation [4]. This wide range of applications also restrict the sensitivity of this technique for the study of phase transformations. If the sensitivity of the recording apparatus is increased, the range of the signal representing the change in length quickly exceeds the range of the recording instrument. This is not a major problem if only a narrow temperature range is studied. Normally the onset of a phase transformation is taken to be the temperature at which the first deviation from the straight line is discernible on an X–Y plot of temperature and dimension. The presence of a substantial linear region in the dilatometric plot is, therefore, essential for the reliable interpretation of the recorded data. Restricting the range over which the temperature is recorded in order to achieve a higher sensitivity to small changes in the specimen length is therefore of dubious value. One possible solution to this problem would be to differentiate the signal representing the change in length. This can be done by either using an operational amplifier in the differentiation mode or by real time digital processing. A differentiated signal, regardless of how it is generated, is highly sensitive to noise in the original signal.

This problem became relevant during the study of the austenitizing kinetics of a low alloy Mn–Mo–Ni pressure vessel steel which was necessary as part of an investigation of the upper nose temper embrittlement of this steel [5]. The tempering behaviour of a steel would change drastically if the austenitization temperature, A_{c1}, was exceeded during the tempering heat treatment. It was therefore important to have a reliable indication of the temperature at which reaustinitizing starts on heating.

2. Experimental procedure and results

A cylindrical sample some 15 mm long of the steel under consideration was used in a resistance-heated vertical tube furnace dilatometer. Table I gives the chemical composition of the material used in this study. The material was initially in the quenched condition. The length of the tube furnace was 250 mm. The temperature of the specimen was measured continuously by 0.1 mm thick noble metal thermocouple wires spot welded to the top and bottom of the cylindrical specimen. The length change was measured by a silica glass tube of 3 mm outside diameter which served as a push rod supporting the core of an a.c. linear variable differential transformer (LVDT) some 300 mm above the top of the tube furnace. The specimen and the push rod, as well as the LVDT core, were contained in a second silica glass tube which was sealed at the lower end. The open top end of this container tube was hermetically sealed by a rubber stopper. The specimen was copper coated for protection against decarburization. The spacing of the resistance wire of the tube furnace and the vertical position of the specimen in the furnace was adjusted so that the temperature difference under steady state conditions over the length of the specimen was less than 5 °C.

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TABLE I Chemical composition of material used in this study (weight %).

C	Mn	P	S	Si	Ni	Mo	Cu	Cr	Al	B
0.15	1.4	0.012	0.003	0.26	0.52	0.53	0.1	0.2	0.030	3 p.p.m.

This was checked after the construction of the furnace by using a differential thermocouple. The temperature of the tube furnace was controlled by a commercial programmable controller and a separate central thermocouple immediately below the specimen holder. For this study, the furnace was initially programmed to heat to 500 °C at 25 °C min^{-1}, followed by heating to 1000 °C at 1 °C min^{-1}. The latter heating rate corresponded to industrial practice. On reaching 1000 °C, the tube furnace switched off and cooled down naturally.

A typical X–Y dilatometric plot generated by plotting the thermal expansion on the horizontal axis and the signal from the Pt–Pt 13% Rh (R-type) thermocouple on the vertical axis is shown in Fig. 1. This deviation from the convention of plotting the temperature on the horizontal axis made it more convenient to establish the zero point for the channel used for recording the thermocouple signal. The formation of large amounts of austenite at around the A_{c1} temperature is evident. This procedure was done for 10 different specimens. The A_{c1} temperature was finally estimated to be 707 ± 4 °C (95% confidence limit). This was in fair correlation to the A_{c1} temperature of 696 °C calculated from an established empirical equation [6]. The change in slope of the dilatometric plot at lower temperatures probably corresponds to the change in heating rate from a fairly high one (where transient temperature gradients in the sample were possible) to a fairly low one (where the temperature difference across the length of the sample might be considerably smaller).

Of possible significance in the context of this study is a slight change in the slope of the dilatometric curve noticed occasionally some distance below the A_{c1} temperature. It was possible that this feature was associated with early austenitization. It could not be studied further, due to the constraints imposed by the thermal expansion on the conventional dilatometric technique.

An alternative technique to determine the austenitizing temperature of steel with a high hardenability is to soak a sample in a dilatometer at a specific temperature followed by rapid cooling. By recording a dilatometric curve during cooling the presence of any partial transformation to austenite is revealed by the subsequent martensitic transformation. This can be clearly discerned as an abrupt change in the slope of this curve, due to the fact that the coefficient of thermal expansion for austenite and ferrite differs considerably. The volume change associated with the formation of martensite is therefore considerably larger than that associated with the ferrite to austenite transformation occurring during equilibrium transformation. It is therefore easier to establish whether austenitization occurred at the soaking temperature by establishing whether martensite forms on cooling.

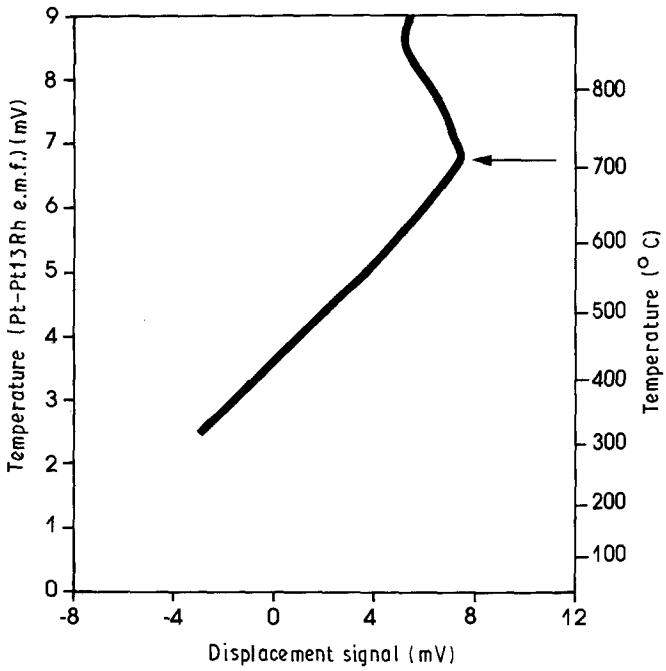

Figure 1 Conventional dilatometric plot of heating of low alloy Mn–Mo–Ni pressure-vessel steel. The formation of austenite at around 705 °C is clearly visible.

The austenitization temperature is experimentally determined by systematically increasing the soaking temperature until the presence of martensite is discerned during cooling.

There are obvious limitations to this method. The magnitude of the systematic increase in the soaking temperature determines the accuracy with which the A_{c1} temperature can be determined. This method is not suitable for low hardenability steels. Probably the single most important objection to this method is that one of the major advantages of dilatometry as a technique for studying phase transformations, namely that it enables one to monitor the extent of reaction as the phase transformation occurs, is lost. This method is therefore only applicable for determining the A_{c1} temperature. It is less suitable for studying the kinetics of the ferrite–austenite transformation or of any other phase transformation.

A modification to the standard dilatometric technique was developed to improve the sensitivity. With this modification, the expansion of the sample was no longer used as an indicator of the presence of a phase transformation. For this purpose the thermocouple e.m.f. was subtracted from a fraction of the voltage representing the change in length of the sample. It is convenient to adjust the fraction of the signal voltage from the LVDT by using a simple potentiometer so that this voltage was of the same order of magnitude as that of the thermocouple e.m.f. The details of this modification are shown in Fig. 2. If the magnitude of these two signals – the fraction of the LVDT signal

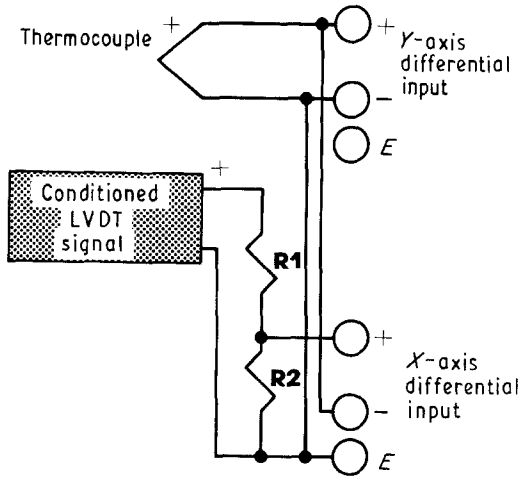

Figure 2 Modification to dilatometric technique to increase sensitivity to non-linear dimensional changes.

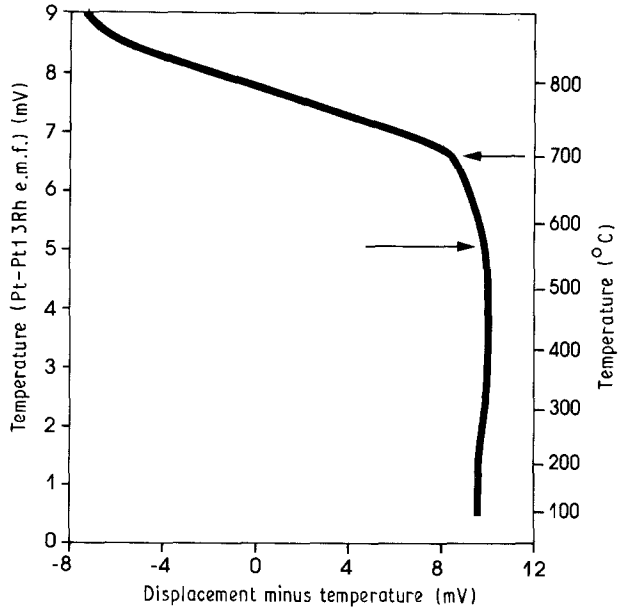

Figure 3 Modified dilatometric plot of heating of steel, showing the definite change in slope at around 560 °C and the onset of large-scale austenitization at around 700 °C.

and the thermocouple e.m.f. – was properly balanced, the difference between these two signals stayed quite constant during thermal expansion. This made it possible to use a much decreased attenuation of the X-channel input to the recorder. The sensitivity to changes in sample length which are associated with phase changes during a particular temperature interval could therefore be greatly increased, typically by a factor of 50.

This modified procedure was used to study the austenitization of 10 samples. The heating cycle was modified to a heating rate of 15 °C min^{-1} to 300 °C, followed by heating to 800 °C at 1 °C min^{-1}. A typical result is shown in Fig. 3. Again the formation of large amounts of austenite at a temperature around 700 °C is evident. The A_{c1} temperature for 10 samples was 700 ± 4 °C. This is slightly lower than the temperature as determined using the conventional dilatometric method. The difference might be attributed to the higher sensitivity of the modified method to non-linear thermal expansion effects. Of more importance to this study is the fact that the slight change in the slope of the conventional dilatometric curve was shown to be a definite change in slope at a temperature determined to be 585 ± 8 °C.

3. Discussion

The change in slope might be interpreted as the start of a ferrite–austenite phase transformation or as evidence of a tempering reaction. Tempering during the heating of a quenched steel also results in deviations from linear expansion. This behaviour can be used to study tempering reactions. These deviations generally occur at considerably lower temperatures [7, 8]. It therefore seems reasonable to assume that this change in slope at, on average, 585 °C indicates the start of austenitization.

Metallographic evidence to support the last hypothesis – that austenitization was observed at temperatures above 585 °C – is presented in the transmission electron micrograph in Fig. 4. A small island of austenite (arrowed) is visible in the matrix of low alloy Mn–Mo–Ni material with the same chemical composition as that used in this study. This material has been tempered at 670 °C for 1 h, well below what is generally assumed to be the A_{c1} temperature for this material. Given the morphology of this austenite island and the temperature and the duration of the tempering heat treatment, it is doubtful that this represents austenite which has been retained during quenching.

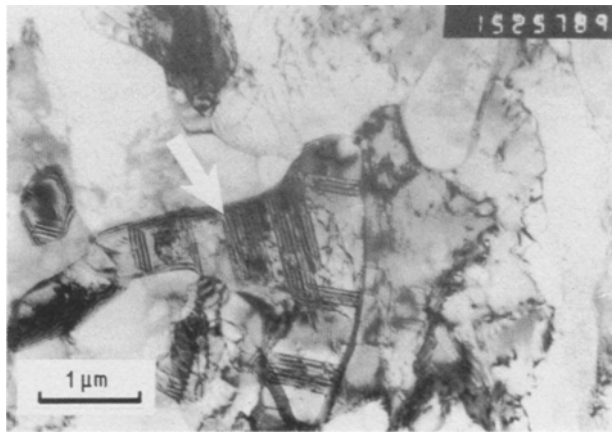

Figure 4 Island of residual austenite in low alloy Mn–Mo–Ni pressure-vessel steel sample annealed at 670 °C for 60 min. The annealing cycle was followed by air cooling.

The technique by which the sensitivity of a dilatometer is increased by largely compensating for the linear thermal expansion has other possible applications in the study of phase transformations. If this technique is implemented when generating continuous cooling dilatometric curves, the kinetics of the phase transformation under consideration can be quantified with a much higher degree of accuracy. The accuracy with which phase transformation temperatures are determined can also be increased to some extent. The dilatometric study of the phase transformation behaviour of alloys with a high linear coefficient of thermal

expansion, for example aluminium alloys, may also become more convenient. This technique would also yield benefits if the higher order coefficients of thermal expansion must be determined. In all of these cases, the technique can be refined even further by using some technique to linearize the thermocouple signal before subtracting it from the signal representing the change in length, either by using a compensating amplifier or digital data processing.

It must be noted that this arrangement is more sensitive to electrical noise than the conventional dilatometer, as the amplification ratio in one channel is much higher than normal. Much more care with regard to the earthing of the recorder, the LVDT signal processing apparatus and the furnace driving circuit was necessary.

4. Conclusions

1. A modification to the conventional dilatometric technique was developed by largely compensating for the linear thermal expansion of the sample. This greatly increased the sensitivity to changes in dimension associated with phase transformations.

2. This technique was used to study the austenitization of low alloy Mn–Mo–Ni pressure-vessel steel. This provided evidence for the onset of austenitization some 130 °C below the conventionally determined A_{c1} temperature for this steel. Metallographic evidence supports the hypothesis that austenitization did, in fact, start at temperatures much lower than the conventionally determined A_{c1} temperature.

3. This technique may be applied to other dilatometric studies of phase transformation and thermal expansion behaviour.

Acknowledgements

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References

1. W. D. DROTNING, *Int. J. Thermophys.* **9** (1989) 849.
2. E. G. WOLFF, B. K. MIN and M. H. KURAL, *J. Mater. Sci.* **20** (1985) 1141.
3. D. MASUTTI, J. P. LENTZ and F. DELANNEY, *J. Mater. Sci. Lett.* **9** (1990) 340.
4. M. TAKAHASHI and H. K. D. H. BHADESHIA, *ibid.* **8** (1989) 477.
5. W. M. DE VILLIERS, H. J. DE KLERK and G. PIENAAR, *Int. J. Pres. Ves. & Piping* **50** (1992) 215.
6. K. W. ANDREWS, *J. Iron Steel Inst.* **203** (1965) 721.
7. Y. TOMITA, *Mater. Sci. Technol.* **4** (1988) 977.
8. LIU CHENG and E. J. MITTEMEIJER, *Metall. Trans.* **21A** (1990) 13.

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