Retraction

Retraction: Effect of thermal cycling on microstructure and hardness of carbon steel (*IOP Conf. Ser.: Mater. Sci. Eng.* 1145 012081)

Published 23 February 2022

This article (and all articles in the proceedings volume relating to the same conference) has been retracted by IOP Publishing following an extensive investigation in line with the COPE guidelines. This investigation has uncovered evidence of systematic manipulation of the publication process and considerable citation manipulation.

IOP Publishing respectfully requests that readers consider all work within this volume potentially unreliable, as the volume has not been through a credible peer review process.

IOP Publishing regrets that our usual quality checks did not identify these issues before publication, and have since put additional measures in place to try to prevent these issues from reoccurring. IOP Publishing wishes to credit anonymous whistleblowers and the Problematic Paper Screener [1] for bringing some of the above issues to our attention, prompting us to investigate further.

[1] Cabanac G, Labbé C and Magazinov A 2021 arXiv:2107.06751v1

Retraction published: 23 February 2022
Effect of thermal cycling on microstructure and hardness of carbon steel

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Abstract. The present investigation deals with design of thermal cycling schedule to enhance hardness of 0.26%C carbon steel using thermo-mechanical simulator Gleeble® 3800. The specimens were thermal cycled at 775°C for 3 and 7 cycles. Optical micrographs, scanning electron micrographs, X-Ray diffraction patterns of as-received steel specimen as well as thermal cycled specimens were reported. The thermal cycling heat treatment causes the refinement of microstructure and is responsible for enhancement in hardness of steel specimen.

Keywords: Carbon steel, Thermal cycling, Grain refinement, Enhanced hardness

1. Introduction
Steel is an important engineering material because it experiences numerous phase changes leading to various range of microstructural development and their analogous mechanical properties [1]. Carbon steels are cost-effective engineering material because they contain only residual concentrations of impurities other than carbon and a little manganese [2]. Even though being cost effective, however, unalloyed carbon steel comes across two main problems, specifically, poor strength and hardness [3,4].

In order to resolve these issues researchers are doing conventional heat treatment, thermo-mechanical processing and thermal cycling. Thermal cycling or cyclic heat treatment is an effective alternate that requires repetitive thermal cycling about a critical temperature proving to reason numerous innovative microstructural modifications, thus affording superior mechanical properties in steel [5–10]. The thermal cycling effects mainly can be concise as the quickening of process kinetics, origination of defects, and structural modification [11–14].

In the current investigation a classic thermal cycling process is done on a carbon steel (containing 0.26% C) using Gleeble® 3800 comprises of repetitive small duration (1min) holding at 775°C (above Ac1 temperature) shadowed by fast cooling. The microstructural development and its result on hardness have been reported in details.

2. Experimental procedure

2.1. Material
The chemical composition of the as-received (ASR) steel obtained by Thermo Jarrell Ash spark emission spectroscope (Model No.13160901) is given in Table 1.
Table 1. Chemical composition of as-received steel (wt.%)

|   | C   | Si  | Mn  | P   | S   | Al  | Fe  |
|---|-----|-----|-----|-----|-----|-----|-----|
|   | 0.263 | 0.001 | 0.654 | 0.026 | 0.026 | 0.017 | Balance |

2.2. Dilatometry and Thermal cycling of steel

To find the valuable critical temperatures such as ferrite to austenite beginning conversion temperature (Ac₁), ferrite to austenite end conversion temperature (Ac₃), austenite to ferrite beginning conversion temperature (Ar₃) and austenite to ferrite end conversion temperature (Ar₁) dilatometry test was done in the Gleeble® 3800 thermo-mechanical simulator. The specimen dimensions for dilatometry test are 6 mm diameter and 30 mm long. This specimen has been heated up to 1100°C with heating rate of 5°C/s, hold for 2 min and cool down to room temperature with the cooling rate of 5°C/s.

Gleeble® 3800 thermo-mechanical simulator was employed for thermal cycling. Cylindrical specimens had a thermocouple spot welded at the centre along the length of the specimen and placed inside copper round grips. Figure 1 displays the schematic demonstration of the designed thermal cycling curves for different number of cycles, viz. 3-cycle and 7-cycle. Each cycle consisted of heating the specimen at a rate of 5°C/s in intercritical region at a temperature 775°C (above the Ac1 temperature, 725°C) and holding for 1 min (short duration), followed by fast cooling at a rate of 30°C/s.

![Figure 1: A schematic diagram of thermal cycling curves.](image)

2.3. Metallography

As received steel specimen as well as thermal cycled specimens were segmented to tiny pieces and polished with succeeding grades of emery papers up to 3000 grit size shadowed by cloth polishing with colloidal alumina solution. These specimens were etched by 2% nital. A Leica DMI 5000 M light optical microscope (LOM) furnished with digital imaging was employed for optical micrographs and a Carl Zeiss EVO 18 scanning electron microscope (SEM) was employed for scanning electron micrographs. To verify the phases Rigaku Smart Lab X-Ray Diffractometer (XRD) was used. The average ferrite grain size was calculated using the linear intercept method according to ASTM E112-13 standard [15].

2.4. Hardness testing

A FIE VM50 Vickers hardness tester with 10kg load and 15s of dwell time was used to measure macro hardness on polished surface. Average of 10 readings are taken at dissimilar locations and its standard deviation is also reported.

3. Results and discussion
3.1 Dilatometry test

Figure 2. illustrates the dilatometry curve for as-received (ASR) steel obtained through Gleeble® 3800. In this curve during change of phase slope of the curve changes because of reduction and increase of specific volume in the altered phases. From figure 725°C, 885°C, 728°C and 528°C are the values of $\text{Ac}_1$, $\text{Ac}_3$, $\text{Ar}_3$ and $\text{Ar}_1$ obtained respectively.

![Dilatometry curve for ASR steel obtained through Gleeble® 3800.](image)

3.2 Microstructure evolution

The optical micrographs of the specimens exposed to dissimilar number of thermal cycles are presented in Figure 3. (a)-(c). In the figure P refers of pearlite and F refers to Proeutectoid ferrite.

![Optical micrographs of the specimens exposed to dissimilar number of thermal cycles](image)

**Figure 3.** Optical micrographs of the specimens exposed to dissimilar number of thermal cycles (a) ASR (0-cycle), (b)3-cycle, (c)7-cycle. P-pearlite, F-proeutectoid ferrite.
Figure 4. Change of ferrite grain size with number of thermal cycles.

Change of ferrite grain size with number of thermal cycles is shown in Figure 4. Table 2 also reports the grain size of ferrite for dissimilar number of thermal cycles. It is inferred from the figure that as the number of thermal cycles increases ferrite grain size decreases.

Table 2. Grain size of ferrite for dissimilar number of thermal cycles

| Number of thermal cycles | Grain size (µm) |
|-------------------------|----------------|
| 0                       | 26.47          |
| 3                       | 25.08          |
| 7                       | 7.32           |

Figure 5. SEM secondary electron images of the specimens subjected to dissimilar number of thermal cycles (a) ASR (0-cycle), (b) 3-cycle, (c) 7-cycle. P-pearlite, F-proeutectoid ferrite.

As depicted in Table 2, ASR steel has average ferrite grain size of 26.47 µm. After 3-cycles average ferrite grain size reduced slightly to 25.08 µm. Drastic reduction in average ferrite grain is found to be 7.32 µm for 7-cycles.
In all the thermal cycling process, when the ASR steel is heated to 775°C, the ferrite-pearlite interface acts as potential site for austenitisation [16,17]. Simultaneously, austenitisation takes place at the ferrite-cementite interfaces in pearlite regions. In both the cases, transformation from ferrite phase to austenite phase takes rapidly in a diffusionless polymorphic transformation at 775°C [18]. On continued holding at this temperature, closure of cementite in the austenite phase is a diffusion regulated gradual process. In the present study, austenitisation at 775°C for an inadequate period of 1 min causes incomplete dissolution of pearlitic cementite appearing in the ASR steel microstructure [19-24]. The presence of any un-dissolved cementite stops the austenite evolution during holding at 775°C. On subsequent cooling, the inadequately grown austenite experiences more under-cooling due to fast cooling rate transforms to numerous ferrite nuclei, whose growth ceases rapidly to produce finer grains.

The SEM secondary electron images of the specimens subjected to various thermal cycles are shown in Figure 5 (a)-(c). P refers to pearlite and F refers to proeutectoid ferrite in the figure. To proceed for microstructural analysis, the ASR steel (0-cycle) microstructure (Figure 5(a)), being an initial microstructure revealing lamellar pearlite with ferrite, has been chosen as a reference microstructure; so that the features developed after different thermal cycles can be characterised through comparison with this reference microstructure. Based on this concept, a close look at Figure 5 (a) and (b) no significant change in average ferrite size, but lamellar pearlite changed to flowery pearlite. From Figure 5 (c) flowery pearlite can be seen in case of specimens subjected to 7-cycles.

![Figure 6](image.png)

**Figure 6.** X-ray diffraction patterns of the specimens subjected to dissimilar number of thermal cycles (a) ASR (0-cycle), (b) 3-cycle, (c) 7-cycle.

The XRD patterns of the specimens subjected to dissimilar number of thermal cycles are shown in Figure 6 (a)-(c). All these specimens display the presence of two phases, α-ferrite and cementite. Analogous XRD patterns were noticed in previous studies, too [9,10].

### 3.3 Hardness development

Variation in hardness with number of thermal cycles is shown in Figure 7. Results of hardness test were reported in Table 3, hardness of ASR steel (0-cycle) was found to be 132 and increased drastically after 3-cycles to 167 due to change in microstructure from ferrite, lamellar pearlite to refined ferrite, flowery pearlite, even though there is no significant change in average ferrite grain size from 26.47µm to 25.08 µm.
Figure 7. Variation in hardness with number of thermal cycles.

After 7-cycles further increase of hardness to 186 because of drastic reduction in ferrite grain size of 7.32μm.

Table 3. Results of hardness test

| Number of thermal cycles | Hardness (VHN) |
|--------------------------|----------------|
| 0                        | 132 ± 8        |
| 3                        | 167 ± 2        |
| 7                        | 186 ± 3        |

4. Conclusions
The following conclusions can be drawn from the present study:

- Thermal cycling process causes refinement of ferrite grain size and pearlite morphology.
- Average hardness value increased from 132 to 186 VHN with implementation of thermal cycles in the present study. This is attributed to reduction in ferrite grain size as well as refinement in pearlite morphology.
- Maximum average ferrite grain size of 26.47μm is seen for ASR specimen, whereas minimum average ferrite grain size of 7.32 μm is seen for the specimen subjected to 7-cycles.

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