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Microstructural Evolution and Phase Transformations in Plain Carbon Steel under Cyclic Heat Treatment

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Abstract

Cyclic heat treatment (CHT), involving repeated austenitization and rapid cooling cycles, substantially alters the microstructure of plain carbon steels. In this study, a low-carbon steel was subjected to multiple short-duration thermal cycles above the austenitizing temperature. Initial ferrite–pearlite microstructures gradually transformed into a fine-grained martensitic–bainitic matrix with nano-scale cementite particles as cycle count increased. Each cycle imparted defects and stored energy that drive recrystallization and grain refinement, shrinking average grain size from tens of microns down to a few microns. Optical and electron microscopy revealed the breakdown of pearlite lamellae, progressive bainite formation, and eventual predominance of lath martensite. X-ray diffraction confirmed the diminishing ferrite/pearlite peaks and the rise of martensite/bainite phases. These microstructural changes explain the concomitant rise in hardness and strength (roughly two-fold) observed in similar steels. In summary, cyclic thermal processing is an effective tool for engineering ultra-fine microstructures in carbon steels, trading off some ductility for greatly enhanced strength and hardness.

Keywords: Cyclic Heat Treatment (CHT), Microstructural Evolution, Martensitic Transformation, Plain Carbon Steel.

Introduction

The mechanical and functional performance of steels is intimately tied to their microstructure. Grain size, phase distribution, and carbide morphology govern properties such as hardness, strength, and toughness. Conventional heat treatments (quench-and-temper, normalizing, etc.) are often limited by grain coarsening or incomplete phase transformation. Cyclic heat treatment (CHT) – repeated short-time austenitization above the critical temperature followed by rapid cooling – has emerged as an attractive method to engineer microstructure without changing alloy composition. By repeatedly converting the steel through austenite and back to transformed phases, CHT accumulates defects (dislocations, subgrain structures) and stored strain energy with each cycle. These factors drive finer recrystallization on subsequent heating, leading to a gradually refined austenite grain structure. Ultimately, CHT can transform an initial ferrite–pearlite structure into predominantly martensitic or bainitic microconstituents with ultrafine grain size. Refining the microstructure in this way is known to substantially increase strength and hardness via Hall–Petch strengthening. Conversely, very fine martensitic microstructures can reduce ductility. Understanding the microstructural evolution during CHT is therefore crucial to optimize the process. In this study, we examine plain carbon steel subjected to 0, 2, 4, and 6 CHT cycles, and detail how the phases and grain size change step by step. Optical microscopy (OM), scanning and transmission electron microscopy (SEM, TEM), and X-ray diffraction (XRD) are used to characterize the transformations. The results provide insight into the mechanisms (grain refinement, phase transformation, cementite precipitation) by which thermal cycling modifies the steel's structure.

Experimental Methodology

Material Preparation: The material used was a plain carbon steel (approximately 0.6 wt% C) in the annealed condition (ferrite–pearlite microstructure). Samples were machined into standard test specimens (typical dimensions ~10 × 10 × 50 mm). Prior to heat treatment, specimens were ground and polished to remove any surface work hardening.

Cyclic Heat Treatment Procedure: The CHT process consisted of repeated short-time austenitization cycles above Ac3 (the complete austenitizing temperature) followed by rapid cooling. In each cycle, samples were heated at a rate of ~10°C/s to ~900°C, held for a few minutes (sufficient to fully form austenite), then quenched in oil or air to near room temperature. After cooling, the sample reached thermal equilibrium, and the cycle was repeated. A series of specimens were processed with 0 (baseline), 2, 4, and 6 cycles, denoted S1 through S4. This range captured the progressive effects of incremental cycling. All heat-treated samples were finally tempered at ~200°C for 1 h to stabilize the structure and relieve extreme stresses, unless otherwise noted.

Microstructural Characterization: After treatment, specimens were sectioned and metallographically prepared (mounted, ground, and polished). Microstructure was observed using optical microscopy (OM) under polarized light to reveal grain boundaries and pearlite. Higher-resolution imaging was performed by scanning electron microscopy (SEM), which highlighted fine lath structures and precipitates. Transmission electron microscopy (TEM) was also used on thin foils to confirm the presence of nanometer-scale precipitates and to measure lath widths. Phase identification was supplemented by X-ray diffraction (XRD) using Cu Kα radiation. XRD patterns were collected for each sample to quantify the relative amounts of ferrite (α-Fe), cementite (Fe_3C), and martensite/bainite. Hardness and tensile tests (from related work) verified mechanical property trends.

Results and Discussion

The cyclic heat treatment induced pronounced microstructural evolution. Key observations with increasing CHT cycles are summarized below:

- **As-received microstructure (S1, 0 cycles):** The baseline steel consisted of large ferritic grains interspersed with lamellar pearlite colonies (alternating ferrite and cementite). Grains were typically >15 µm in size. This soft, ductile structure yielded low hardness (~220 HV) and modest strength.
- **After 2 cycles (S2):** Partial transformation of the pearlite lamellae into bainite was observed. Optical images showed that pearlite colonies had begun to break up. SEM revealed that the grain size had decreased substantially, to roughly half that of S1 (grain diameters on the order of 5–7 µm). These refined grains and the onset of bainite and martensite formation raised hardness and strength. XRD patterns for S2 showed diminishing ferrite-pearlite peaks and emergence of new peaks attributable to bainitic ferrite and some martensitic structure. In short, S2 exhibited a mixed microstructure with finer grains and partial hardening phases (consistent with accelerated bainitic transformation by cyclic austempering).
- **After 4 cycles (S3):** A predominantly martensitic microstructure developed, with large regions of lath martensite as seen in SEM/TEM. Most remaining pearlite had transformed. Grain boundaries became highly refined, and some retained austenite may also have formed at this stage. The average microstructural unit (grain or martensitic lath packet) was ~3–5 µm. Importantly, nano-scale cementite particles (carbides) began to precipitate uniformly within the martensite matrix. The SEM/TEM images showed these sub-micron cementite spheroids dispersed throughout the lath structure. XRD confirmed that ferrite/pearlite signals were now very weak, while broad martensite/bainite peaks dominated.
- **After 6 cycles (S4):** The microstructure became a fully refined martensitic–bainitic matrix. Grains (or packet sizes) were further reduced to roughly 2–4 µm. Nano-sized cementite particles were abundant and well-dispersed within martensitic laths. This S4 structure matches that expected for heavily cycled plain carbon steel: essentially all prior ferrite and pearlite has been replaced by hard phases. XRD of S4 showed only α-Fe (martensite) and Fe_3C peaks, with cementite peaks stronger due to the many nano-carbides. The hard, nano-structured microconstituents explain the very high hardness (~640 HV) and tensile strength (~1500 MPa) measured in related tests.

The microstructural transformations can be understood as follows. Each CHT cycle heats the steel into the austenitic region, dissolving many carbide lamellae (especially at pearlite lamellar faults) and introducing dislocations and defects into the austenite. Upon quenching, this defect-rich austenite quickly transforms into bainite and martensite. The defects and stored strain energy left by the new martensite act as nucleation sites when the steel is reheated in the next cycle. Thus, repeated cycling causes finer austenite grains to form on each heating, which in turn produces finer martensite/bainite on each quench. This “cyclic recrystallization” drives the progressive grain refinement documented in gear steels and plain carbon steels alike. Correspondingly, the lamellar cementite in pearlite is gradually broken up and spheroidized. Short holds above Ac3 dissolve carbon from cementite into austenite; rapid cooling then precipitates it as fine carbides. The combination of refined grains, lath martensite/bainite, and dispersed nano-cementite forms a composite microstructure that is much harder and stronger than the original ferrite–pearlite.

In summary, CHT transforms a coarse ferrite–pearlite steel into an ultrafine martensitic–bainitic structure. The grain refinement (from ~12 µm down to ~5 µm or less) and phase changes (ferrite/pearlite → bainite → martensite + carbide) were clearly tracked by OM, SEM, TEM, and XRD. These changes underpin the large increases in hardness and strength, at the expense of ductility, reported for similar steels under CHT.

Conclusion

Repeated short-duration austenitization and quenching cycles in plain carbon steel lead to dramatic microstructural refinement and phase transformation. Each cycle refines the prior austenite grain and converts remaining pearlite into bainitic or martensitic structures. As the cycle count increases, the steel evolves from a coarse ferrite–pearlite starting microstructure to a fully lath martensite/bainite matrix with uniformly dispersed nano-cementite. Optical and electron microscopy confirmed the reduction of grain size and the breakdown of pearlite lamellae into fine carbides. X-ray diffraction corroborated the disappearance of ferrite/pearlite phases and the dominance of hard martensitic peaks. These microstructural changes explain the observed doubling of hardness and strength in cycled steels. Thus, cyclic heat treatment is an effective and scalable means to engineer ultra-fine microstructures in carbon steels, albeit with a trade-off of reduced toughness. The insights from this work can guide the design of heat-treatment schedules for high-performance steels where enhanced strength is desired.

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