Heat treatment optimization of an automotive stamping die steel with low energy consumption

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Abstract. It has innovatively treated an ultra-high carbon steel via a novel technique with low energy consumption for automotive stamping die application in this study. Formation mechanism of isothermal transformation product at low temperatures in an ultra-high carbon steel has been investigated by the means of X-ray diffraction (XRD) analysis and metallographic examination on the resulting microstructure, which is optimally designed to achieve an excellent combination of high impact toughness and high hardness. It is indicated that microstructure morphology of resulting bainite isothermally formed at temperatures below 200℃ is observably different from water-quenched shear-transformed martensite; in the case of isothermal transformation above 200℃ for a long time, it obtains bainite sheaf which is composed of slender bainitic ferrite plates separated by parallel parent phase films; bainite sheaf is achieved by nucleation and growth of new parallel bainite plates in the vicinity of both body sides of the already formed bainite plate.

1. Introduction
Ultrahigh carbon steels with a carbon content of 1.0~2.1 wt.% were previously neglected in industrial application in last century because the continuous thick carbide networks precipitated along grain boundaries upon cooling from high temperature to intermediate temperature with great embrittlement and could be the location where cracks might initiate and readily propagate under stress resulting in premature failure [1]. Thanks to development of material manufacturing technology and requirement of high mechanical-property steels with low cost, ultrahigh carbon steels have received more significant attention to investigations on the superplasticity behavior. Professor Sherby from Stanford University researched on ultrahigh carbon steels with ultrafine grains [2,3] and achieved the related patent [4]. Soon afterwards, investigators all over the world [5,6] began with further research in succession. Recent years thermo-mechanical treatment [7] and multiple heat treatment processes [8,9] generally produce ultrafine ferrite and dispersed globular carbide to decrease embrittlement [10-11]. However, the thermo-mechanical treatment brings about disadvantage in noise pollution, while the multiple heat treatment consumes very much energy resource. In this study, corresponding to low emission policies, a novel technique has been developed to achieve excellent combined performance of high toughness and high hardness in an automotive stamping die steel with ultrahigh carbon content via isothermal transformation at low temperatures. Phase transformation characteristic has been investigated to attempt to provide some experimental evidence for manufacturing technology of new environmental-protection ultrahigh carbon steel with low energy consumption.
2. Materials and experiments
The experimental ultra-high carbon steel (Fe-1.5C-0.3Si-1.5Cr-0.7Mn-0.7Al wt.%) was produced by electro-slag refining process in a ZG-25 vacuum mid-frequency induction furnace. The cast ingot was forged into a bar with a diameter of Φ60 mm. The specimens were machined with a final dimension of 12 mm×12 mm×57mm. The martensite-start temperature (MS) was determined using a Formastor-Digit 2 dilatometer, and MS = 22.8℃ was obtained, corresponding to austenitizing at 880℃. The specimens were austenitized at 880℃ for 20 min in an SRJX-4-10 furnace, and quenched in water, or isothermally transformed in oil bath for various durations and followed by water-cooling to ambient temperature, respectively.

The specimens were mechanically polished up to 1 mm considering to completely remove the oxidization and decarburization layer. The hardness test and impact test were carried out using HRC-150 hardness tester and JB30A 30/15 pendulum impact toughness tester on V-notched specimens in 10 mm × 10 mm × 55 mm, respectively. The metallographic microstructure was revealed by 4 vol.% nital solution and observed by a Ziss Axio Scope A1 optical microscopy. X-ray experiments were conducted using a D/Max-2500V Japanese Science X-ray diffractometer (XRD) with CuKα radiation at a voltage of 40 kV and a current of 100 mA, and the results were analyzed by Jade Software.

3. Results and discussion
3.1. Mechanical properties
Table 1 presents the hardness test results of specimens isothermally transformed for various holding durations at low temperatures from 880℃ austenitization, and another specimen quenched in water which is just for informative reference only. It is obviously seen that the hardness is decreased with increasing the holding time. This most probably implies that with a short isothermal transformation time very a fewer bainite could be allowed to form while more martensite could be obtained from the retained austenite in the subsequent water quenching to ambient temperature which contributes higher hardness. This point would be double-check demonstrated by resulting microstructures in the following sections. The hardness reduces faster and goes down lower when isothermally transformed at 200℃, in comparation with temperatures above or below. It suggests that a faster bainitic transformation could be achieved at 200℃, in another word, the shortest incubation period of bainitic transformation is over there or around. Increasing the isothermal transformation time, more bainite, a little bit lower hardness and much higher impact toughness take place concurrently as interpreted in Table 2 which describes the impact absorption energy of the accordingly related specimens for hardness test. Particularly, it can reach up to 45 J/cm² when isothermally transformed at 200℃ for 1h.

| Heat Treatment Process | Isothermal transformation duration |
|------------------------|-----------------------------------|
|                        | 10min    | 20min    | 30min    | 60min    |
| Isothermally transformed at 150℃ | 64.2 HRC | 63.5 HRC | 62.9 HRC | 60.0 HRC |
| Isothermally transformed at 200℃ | 63.5 HRC | 61.8 HRC | 59.7 HRC | 58.1 HRC |
| Isothermally transformed at 250℃ | 64.0 HRC | 63.4 HRC | 61.6 HRC | 59.5 HRC |
| Quenched in water       |          |          |          | 64.5 HRC |

Table 2- Charpy impact test results of specimens

| Heat Treatment Process | Isothermal transformation duration |
|------------------------|-----------------------------------|
|                        | 10min    | 20min    | 30min    | 60min    |
| Isothermally transformed at 150℃ | 11 J/cm² | 12 J/cm² | 15 J/cm² | 30 J/cm² |
| Isothermally transformed at 200℃ | 14 J/cm² | 18 J/cm² | 32 J/cm² | 45 J/cm² |
| Isothermally transformed at 250℃ | 12 J/cm² | 16 J/cm² | 21 J/cm² | 34 J/cm² |
| Quenched in water       | 10 J/cm² |          |          |          |
3.2. Analysis of transformation temperature

Figure 1 shows transformation curve of initial bainite after austenitizing at 880°C. It can be evidently seen that bainite can be generated with the shortest incubation period of 120 seconds in accordance with isothermal transformation at 200°C. It investigates the formation characteristic of transformation product phases in the specimens isothermally transformed at 150°C, 200°C and 250°C, respectively.

![Figure 1](image1)

Figure 1 Transformation curve of initial bainite at the medium isothermal temperatures after austenitizing at 880°C

XRD patterns of the specimens isothermally transformed at 150°C, 200°C and 250°C, respectively, for 30 min, and also an annealed specimen are presented in Figure 2. Note that α phase peaks vary: (110)α peak of the annealed specimen meets up with 2θ=44.559°, while the specimens transformed at 150°C, 200°C and 250°C, 2θ=43.94°, 44.459° and 44.419°, respectively. Intensity and shape of the peaks are particularly different around 2θ=65°, where the former has the highest intensity peak, while the latter lower successively as the isothermal temperature increases. It is indicated that existence states of α phase in the specimens differ with each other.

![Figure 2](image2)

Figure 2 XRD patterns of annealed specimen (a) and specimens isothermally transformed at (b) 150°C, (c) 200°C, and (d) 250°C

3.3. Microstructures of transformation products

Microstructures of the annealed specimen and specimens isothermally transformed at 150°C, 200°C and 250°C for 30 min are shown as Figure 3. Figure 3a illustrates microstructure of the specimen transformed at 150°C, composed of randomly distributed bainitic ferrite plates (as arrowed) and bright carbide particles. Mass sheaf-like microstructures of bainite can be seen observed in Figure 3b
(amplification of the rectangle region is shown in the right top corner), while independent plates become less. The bainite sheaves distribute uniformly, and some distinct spaces exist between neighboring plates in a bainite sheaf. It is suggested that each plate nucleates and growth independently. Nevertheless, the imprint of bainite sheave on matrix austenite is not obvious at all. The mixture microstructure transformed at 250°C, as shown in Figure 3c, is constituted by a great quantity of independent bainite plates, a spot of bainite sheaves, some carbide particles and the residual austenite. The bainite sheaf transformed at 250°C has a smaller quality of grain side and plates number than that of transformed at 200°C. Figure 3d presents the microstructure of the annealed specimen. The single arrow shows carbide particle, and the double arrow shows pearlite. Note that quantity of particles in isothermally transformed specimens is less than that in annealed specimen. Moreover, the carbide particles decrease with increasing transformation temperature. It is indicated that mass carbon is excessive in α phase solution when isothermally transformed. With lower transformation temperature, stronger crystal distortion is formed by larger carbon supersaturation. As a result, XRD peak of α phase migrates or changes into a diffusing shape, which is identical with the patterns in Figure 2.

![Figure 3 Microstructures of specimens: isothermally transformed at (a) 150°C, (b) 200°C, (c) 250°C and (d) the annealed specimen](image)

According to details of Figure 4, microstructures of the specimens isothermally transformed at low temperatures for a long time are evaluated, and the advisable temperatures at which bainite sheaf can be generated by isothermal transformation are estimated as well. There is a common phenomenon in Figure 4a and b that no bainite sheaf can be found in specimens by lower-temperature isothermal transformation at 150°C or 175°C for even 60 min, the observation is only filled with individual bainite plates, while it is completely opposite in the case of higher-temperature isothermal transformation at 225°C or 250°C, as shown in Figure 4c and d. It can be concluded that lower transformation temperatures below 200°C are beneficial to forming individual plates rather than bainite sheaf, while higher transformation temperatures above 200°C advantage formation of bainite sheaf, but individual plates.
Figure 4 Microstructures of specimens: isothermally transformed at (a) 150℃, (b) 175℃, (a) 225℃, and (b) 250℃ for 60min

In accordance with the XRD analyses and metallographic observation above, product of phase transformation might be pearlite, bainite or martensite in sequence as transformation temperature decreases, and corresponding ferrite morphology is layer, plate, or acicular. With decreasing transformation temperature, formation of bainite changes form sheaves to single plates, and carbon supersaturation becomes larger. It is inferred that bainitic transformation is different from that of martensite, neither pearlite, and it shall not be briefly regarded as a single diffusion or shear mechanism.

3.4. Formation model of bainite sheaf

In order to contribute to the formation model of bainite sheaf in the experimental steel, this paper focuses on analysis and discussion on the microstructure characteristics of specimens isothermally transformed at 200℃ for different times, of which the XRD patterns have been shown as Figure 5, in company with that of the water quenched specimen. The change in location of α phase diffraction peak should be respected. With increasing the holding time from 2 min to 60 min, the value of 2θ pertain to (110)α peak increases from 43.882° to 44.266°, each of which is more than that of the quenched specimen. The quenched specimen has a double peak feature of (002)α and (200)α around 2θ=65°, where the specimen holding for 2 min also has a similar tendency. As the holding time increases, the feature of double peak wears off. For example, in the case of holding for 60 min, only a feature of (200)α single peak can be found. It is demonstrated that nature of ferrite phase in specimens holding for different times is also variational. For the specimen holding for only a short time, α phase mainly reflects martensite transformation mechanism, while holding for a long time reveals bainite transformation mechanism.

Figure 5 XRD patterns of specimens: (a) quenched in water and transformed at 200℃ for (b) 2 min, (c) 5 min, (d) 10 min, (e) 20 min and (f) 60 min
Figure 6 shows that in the quenched specimen, it consists of dark acicular martensite and light matrix austenite, and boundary of martensite is more distinct than that of bainite. Because of lattice distortion accompanied with shear transformation, acicular martensite can be easily etched to present a dark contrast. A mixture microstructure is obtained in the specimen held at 200°C for 2 min, which is composed of acicular martensite, bainite plates, and residual austenite. Because of very short holding time, only a small fraction of austenite transforms to bainite, and the retained austenite could transform to martensite during the subsequent cooling to ambient temperature. Figure 6c–f represent the formation of bainite sheaf as a function of holding time. With increasing holding time, more and more bainite plates are generated, meanwhile, less and less acicular martensite can be formed. Note that almost entire observation is filled with bainite sheaves when holding for 60 min.

![Figure 6 Microstructures of specimens: (a) quenched and transformed at 200°C for (b) 2 min, (c) 5 min, (d) 10 min, (e) 20 min and (f) 60 min](image)

An appropriate carbon concentration caused by thermodynamical component fluctuation in austenite nearby the bainite plates, as well as activation energy of phase transformation in a particular range, is prerequisite for the formation of bainite sheaves. Such conditions are exactly right provided at temperatures above 200°C. It has been confirmed that the bainitic ferrite plate is carbon supersaturated which is obtained by low temperature transformation [16-18]. Carbon concentration in bainitic ferrite is considerably higher than pearlitic ferrite, but much lower than martensite. The parent austenite extremely bordering on bainite/austenite interface contains excessive carbon. Carbon distribution is regular, along with the boundary of bainite plate. Bainite is not able to nucleate in these slender carbon-rich zones, but in the normal austenite zone with an appropriate carbon concentration. New bainitic ferrite plates nucleate repeatedly in this way, and grow via a displacive mechanism in nature [19]. New plates do not distribute randomly, but keep a coordinated orientation relationship with the already formed plates, as result of influence of carbon concentration distribution. Such parallel plates in the form of colony grow into a bainite sheaf, so that bainite sheaves occur. It can be concluded that formation of bainite sheaf is most possibly a consequence of evolution of new repeated plates generated, with an approximately same orientation relationship, in vicinity of body sides of the already formed plates.
4. Conclusions
During the cooling from austenitizing temperature of 880°C, the parent austenite can be possibly transformed into pearlitic ferrite, or bainitic ferrite, or martensite, as a function of transformation temperature. In this study, it has innovatively heat-treated an ultra-high carbon steel with low energy consumption for automotive stamping die application by austempering process at low temperatures as consequence of crystal nucleation and growth via a displacive mechanism in nature, which excels in contributing an excellent toughness property concurrently with a very high hardness. The shortest incubation period of bainitic transformation occurs at 200°C, above which temperature the bainite sheaf can merely forms. In this given condition, the most feasible formation model of bainite sheaf is that new repeated plates are generated in vicinity of body sides of the already formed plates and grow up with an approximately same orientation relationship.

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