Effect of two-step austempering process on carbidic ductile iron

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Abstract. The two-step austempering process when applied to ductile iron, results in significant improvement in mechanical properties due to the strengthening of the ausferitic matrix. The present work was undertaken to investigate the effect of the two-step austempering process on carbidic ductile iron. Carbidic ductile iron is a new member to the ductile iron family, it is produced by alloying the ductile iron composition with chromium. In the present work, the carbidic ductile iron thus produced, was austempered by both conventional and two-step austempering route. For both processes, all specimens were austenitized at 975°C and soaked for 180 min. In the conventional process, the specimen was quenched in salt bath held at to 350°C, 400°C, and 450°C and soaked at respective temperatures for 180 min. Whereas, in the two-step austempering process, specimens were first quenched in salt bath held at 250°C. After stabilization at 250°C, the salt bath temperature was raised to 350°C, 400°C, and 450°C. After the respective temperature was attained, the specimens were soaked for 180 min. The austempered samples obtained by both processes were analyzed with the help of SEM, XRD, and TEM. Two-step process resulted in finer ausferitic matrix than the conventional process. The XRD analysis revealed an increase in carbon content of austenite and volume fraction of Carbon Stabilized Austenite (CSA) in sample austempered by a two-step process. The TEM analysis indicated that the lath width of austenite and ferrite was small in samples austempered by two-step process than the conventional process, due to a high degree of super-cooling.

1. Introduction
Austempered Ductile Iron (ADI) is known to be very useful in many engineering applications as automobile parts (camshafts). The ADI has unique property of high strength at very low cost [1]. The properties of ADI are attributed to its unique structure comprising of acicular ferrite and carbon stabilized austenite called as “ausferrite” (2). ADI is produced by austempering of ductile iron.

Carbidic austempered ductile iron (CADI) is the newest member in the cast iron family. As the name suggests, it has carbides embedded in the ausferitic matrix. The presence of carbides improves the hardness and wear resistance of alloy but on the other hand reduces the toughness [1, 2].

The carbide can be introduced in matrix ADI by various methods [1, 3-6]. In this investigation, chromium was added into the melt to obtained carbide in ADI matrix.

Putatunda introduced the concept of two-step austempering process [7, 8]. Many researchers have studied and compared the two-step and conventional austempering processes. They reported many advantages of two-step process over the conventional process [9-16].

The aim of present study was to study the effect of two-step austempering, on the microstructure and structural characterization using XRD and TEM analysis of CADI.
2. Experimental details

2.1. Metal casting and heat treatment
The carbidic ductile iron was produced in a medium frequency induction furnace. The molten metal was treated with 5% Mg-Fe-Si alloy using the sandwich technique. The molten metal was inoculated with foundry grade ferrosilicon with nominally 75% silicon. After spheroidising treatment, the molten metal was poured into mould. The casting was produced in cylindrical (φ20 mm, L = 300 mm) form.

The chemical composition (wt. %) of the ductile iron alloyed with chromium is given in table 1.

| Element | C   | Si  | Mn  | Cu  | Ni  | Cr  | CE* |
|---------|-----|-----|-----|-----|-----|-----|-----|
| Wt.%    | 2.42| 1.88| 0.86| 0.57| 0.57| 3.63| 3.1 |

*CE (carbon equivalent) = %C + 1/3 %Si

The carbidic ductile iron was austempered by both conventional and two-step austempering route. All specimens were austenitized at 975°C for 180 min. In the two-step process, specimens were quenched to 250°C. After stabilization at 250°C, the salt bath temperature was raised to 350°C, 400°C, and 450°C. After the respective temperature reached, the specimens were soaked for 180 min. In the conventional process, the specimens were quenched at 350°C, 400°C, and 450°C and soaked for 180 min.

The SEM analysis was carried out on JEOL 6380A make machine. X-ray diffraction analysis was carried on X-pert-Pro diffractometer. For X-ray, Cu Kα radiation at 40 mA and 45 kV was used. The XRD analysis volume fraction of CSA, the carbon content of austenite and ferrite cell size were estimated as described in previous literature [12,17-21].

3. Results and discussions

3.1. Microstructural analysis
The figure 1 shows the microstructure of the alloy in the as-cast condition.

![Figure 1. As as-cast carbidic ductile iron (2% Nital)](image)

The SEM images of conventionally and two-step austempered samples of the alloy at three different temperatures are shown in figures 2 and 3 respectively.

In the as-cast condition and in samples austempered by either of the processes, the coarse eutectic carbides, the carbides having globular and blocky morphologies were also observed. The similar observations have been also reported by other researchers, after the conventional austempering of CADI [2,22].

The ausferitic matrix gets coarser, with increasing austempering temperature in both processes. For given austempering temperature, a fine ausferitic matrix was observed in samples process by two-step austempering route than conventional austempering route.
3.2. XRD analysis

The representative XRD plots for samples austempered by conventional and two-step process austempered at 400°C are given in figures 4 and 5 respectively. Along with austenite and ferrite peaks, some peaks corresponding to carbide like Fe$_3$C$_7$ and Cr$_7$C$_3$ were also observed in both processes. The austenite peak observed at (111) at 43.4° in conventional austempering, shifted slightly toward left in sample austempered by two-step austempered indicating a change in the lattice parameter of austenite. The lattice parameter of austenite changes due to the presence of carbon in austenite [20]. The intensity of austenite peak (111) was more in samples austempered by two-step than that of the conventional austempering process. This suggests an increased volume fraction of CSA in two-step than that of conventional austempering process.
The comparison of ferrite cell size, the carbon content of austenite and volume fraction CSA for both austempering is given in figures 6-8 respectively. From figure 6, it clear that the two-step process results in smaller ferrite cell size when compared to that of conventionally austempered samples. It is also observed that the ferrite cell size increases with austempering temperature in both processes. Both observations are in correlation with the observation in SEM images.

![Figure 6. Comparison of Ferrite Cell Size for Conventional and Two-Step Austempering.](image1)

![Figure 7. Comparison of Carbon Content of Austenite for Conventional and Two-Step Austempering.](image2)

![Figure 8. Comparison of volume fraction of CSA for conventional and two-step austempering.](image3)

The carbon content of austenite and volume fraction of CSA was increased in samples austempered by two-step process than that of conventionally austempered samples (figure 7 and figure 8).

3.3. TEM analysis

TEM analysis was carried out on JEOL JEM 2100. The accelerating voltage was 200 kV.

The TEM images are shown in figure 9 (Conventional austempering treatment) and figure 10 (Two step austempering treatment).

It was observed that in case of conventional austempering, average lath width of ferrite was 306 nm and that of austenite was 315 nm. As against that the two step austempering process produced ferrite with an average lath width of 248 nm and austenite with a width of 260 nm.

In a conventional process, less number of ferrite nuclei gets nucleated compared to that of two-step process due to higher quenching temperature. As number of ferrite nuclei is less, they can grow much bigger in given austenite grain and vice versa. In both process, austenitization temperature is 975°C.
and hence the prior to quenching condition is same, i.e. size of austenite grain and austenite carbon [23-25]. Thus, in this case, austempering or quenching temperature plays an important role in final microstructure. Hence, the two-step austempering process produces the finer ausferitic matrix than the conventional process.

**Figure 9.** TEM analysis for conventionally austempered samples at 400°C for 180 min. **Figure 10.** TEM analysis for two-step austempered samples at 400°C for 180 min.

Further, large number of ferrite nucleation result in more carbon atom rejected from ferrite to get loaded into surrounding austenite in sample processed by two-step route. This result in higher carbon content in austenite, in samples austempered by two-step process than conventional process. Higher carbon content in austenite increases the amount of carbon-stabilized austenite (CSA). Hence, the CSA volume fraction and its carbon content is more in sample austempered by two-step process than that in case of the conventional process.

4. Observations and conclusions

The carbides having globular and blocky morphologies were observed in samples austempered by conventional and two-step austempering process. The XRD analysis confirms the presence of iron carbide (Fe₃C₇) and chromium carbide (Cr₇C₃). The area fraction of carbide is reduced after austempering from 22–28% to 15–18%.

The TEM analysis shows the ferrite and austenite lamellar structure in ausferite matrix of CADI along with blocky carbide. This lamellar structure was found in both samples austempered by both routes. SADP analysis confirms the presence of carbides.

The fine ausferritic matrix was observed in SEM images of samples austempered by two-step route when compared to the SEM images of samples austempered by conventional route. The lath width of ferrite and austenite is smaller in sample austempered by two-step process than that of conventional process. The lower quenching temperature in two-step favors the high nucleation rate of ferrite. Consequently, a large number of ferrite gets nucleated and the fine ausferitic matrix was obtained.

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