

THE INFLUENCE OF AUSTENITISING TEMPERATURE ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF LOW-ALLOYED Ni-Mo-Cu AUSTEMPERED DUCTILE IRON

VPLIV TEMPERATURE AUSTENITIZACIJE NA MIKROSTRUKTURU IN MEHANSKE LASTNOSTI MALOLEGIRANE NiMoCu AUSTEMPRANE DUKTILNE ŽELEZOVE LITINE

**Branko Radulovic¹, Branka Bosnjak², Richard Harding³, Kiro Pop-Tonev⁴,
Vanja Asanovic¹**

¹ University of Montenegro, Department of Metallurgy and Technology, Cetinjski put bb 81 000 Podgorica, Montenegro, Yugoslavia

² University of Montenegro, Rektorat, Cetinjski put bb 81 000 Podgorica, Montenegro, Yugoslavia

³ University of Birmingham, IRCM, Edgbaston, Birmingham B15 2TT, UK

⁴ University "Rudjer Boskovic", Department of Technology and Metallurgy, 91 000 Skopje, Macedonia

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In the present study we have investigated the effect of austenitising temperature and austempering time on the microstructure and mechanical properties of low-alloyed Ni-Mo-Cu ductile iron. The specimens were austenitised at 850, 875, 900 and 930 °C for 120 min, then austempered for 10, 30, 60, 120, 240 and 360 min at 350 °C, and examined by optical and scanning electron microscopy. The volume fraction of retained austenite, bainite, martensite and austenite-carbon-content results were then correlated with microstructural changes, the kinetics of transformation and impact toughness.

Following short periods of austempering time, the carbon content of the retained austenite is low and on subsequent cooling to room temperature it transforms to martensite, resulting in low impact values. The carbon content of the austenite was evaluated by measuring the lattice parameters using X-ray diffraction.

Optimum properties were obtained at 900 °C for intermediate austempering periods (120-240 min).

Keywords: austempered ductile iron, retained austenite, austempering, austenitising

Raziskan je vpliv temperature austenitizacije in austempering časa na mehanske lastnosti malo legirane NiMoCu duktilne litine. Vzorci so bili ogrevani 120 min pri 850, 875, 900 in 930 °C ter nato austemprani 10, 30, 60, 120, 240 in 360 min pri 350 °C ter preiskani v optičnem in v vrstičnem elektronskem mikroskopu. Volumski delež zadržanega austenita, bajnita, martenzita in vsebnosti ogljika v austenitu so bili korelirani s spremembami mikrostrukture, kinetiko premene in udarno žilavostjo.

Po kratkih časih austenitizacije je vsebnost ogljika v zadržanem austenitu majhna in pri ohlajanju na sobno temperaturo spremeni v martenzit, kar povzroča majhno žilavost. Vsebnost ogljika v austenitu je bila določena z določitvijo parametra mreže z difrakcijo X žarkov.

Optimalne lastnosti so bile dosežene pri 900 °C za vmesne čase austempranja (120-140 min).

Ključne besede: austemprana duktilna litina, zadržani austenit, austempranje, austenitizacija

1 INTRODUCTION

The term austempered ductile iron (ADI) refers to a new family of materials whose mechanical properties can be varied by the correct selection of heat treatment variables. The wide range of properties offered by austempered ductile iron makes ADI suitable for a wide variety of applications in automotive, rail and heavy engineering industries¹⁻³. The commercial use of this new family of iron is continually increasing and the recent establishment of standards such as ASTM A897M:1990 will encourage further applications.

The heat treatment of the austempering process consists of austenitising ductile iron, during which time the matrix becomes fully austenitic, quenching to the austempering temperature for a controlled time, and then cooling to room temperature (**Figure 1**).

During the austempering process, ADI undergoes a two-stage transformation process³⁻⁶. In the first stage, the austenite (γ) decomposes into bainitic ferrite (α) and carbon enriched austenite (γ_{hc}), a product known as ausferrite. This reaction can be expressed as:



If the casting is held at the austempering temperature for too long, then the carbon enriched austenite (γ_{hc}) further decomposes into ferrite (α) and carbide.



The optimum mechanical properties are achieved with completion of the first reaction, but before the second reaction. The second reaction produces carbides, which makes the material very brittle. This reaction is,

therefore, undesirable and must be avoided. The time period between the end of stage I and the onset of stage II is called the processing window.

In ductile iron, tight control of austenitising temperature is imperative if consistent properties are to be obtained. The variations in austenitising temperature shift the processing window over which the optimum properties can be attained. In general, increasing the austenitising temperature delays the austempering transformation, and shrinks the size of the processing window.

The austenitising temperature has a significant effect on the hardenability and transformation behavior at higher temperatures, the matrix carbon content increases and the austenite grain size is enlarged. This change increases hardenability and reduces the rate of reaction and thus permits larger castings to be treated or less efficient quenching baths to be used⁷.

The austenitising temperature should be selected to ensure sufficient carbon transfer from the graphite nodules to the austenite matrix. The carbon solution process is both time and temperature dependent, thus high austenitising temperatures shorten the time necessary to attain a uniform carbon content in the matrix structure. However, scaling and casting distortion may result if austenitising is carried out at higher temperatures⁸.

Lower austenitising temperatures may be specified for castings which are to be machined after austempering. Lowering the austenitising temperature lowers the volume of untransformed austenite. Under the higher pressures involved in machining, the untransformed austenite transforms to martensite, thus reducing the machinability. On the other hand, a decrease in austenitising temperature frequently results in incomplete austenitisation and associated problems with the microstructure and mechanical properties.

In this study, the austenitising temperatures investigated were 850, 875, 900 and 930 °C. It is believed that such an investigation will help to establish the optimum austenitising temperature. Previous studies suggest that this temperature would be within the proposed range.

2 EXPERIMENTAL

The ductile iron used in this investigation was produced in a medium-frequency induction furnace of 100 kg capacity. The charge consisted of 50% pig-iron ingot and 50% low-manganese steel scrap. The melt was treated with 1.8% of magnesium-ferrosilicon alloy using the sandwich technique. The melt was then doped with 0.67% foundry-grade Fe-Si containing nominally 75%Si and cast at 1400 °C into a standard Y-block mould, which ensured a sound casting. The chemical composition of the low-alloyed ductile iron used in the present work is given in **Table 1**.

Table 1: Chemical composition of treated alloy, wt-%

C	Si	Mn	P	S	Cu	Cr	Ni	Mo
3.14	2.67	0.07	0.023	0.003	0.30	0.12	0.80	0.25

Image analysis techniques were used to measure the volume fraction of ferrite, pearlite, carbides and graphite and the nodule count in the as-cast structure.

Specimens for mechanical investigations were machined from the test section of the Y-block.

Unnotched Charpy specimens (55 x 10 x 10 mm) were machined from the bottom section of the keel block in order to avoid segregation effects and porosity.

Test specimens were initially austenitised for 2 hours at 850, 875, 900 and 930 °C in a muffle furnace and then immediately austempered in a molten-salt bath at 350 °C for, 10, 30, 60, 120, 240, 360 minutes and finally air-cooled to room temperature.

After austempering, surface grinding removed any decarburised skin which formed during the heat treatment. In order to avoid the transformation of any metastable austenite to martensite, the grinding speed was kept as low as possible.

The austempered, unnotched, Charpy specimens were tested at room temperature. At least two samples were tested for each heat-treatment condition.

For microstructural analysis, samples were taken from the impact-test specimens at positions far from the fractured area, others were taken from near the fractured area in order to determine the transformation of unstable austenite to martensite by mechanical stress. Specimens for light microscopy were prepared by a standard metallographic technique.

In order to determine the volume fraction of retained austenite, X-ray diffraction analysis was carried out with Cu-K α radiation at 40kV and 20mA. The diffractometer was equipped with chart recorder to cover the angular-2 θ range from 20-120°. The profiles were computer analyzed to obtain the peak positions and the integrated intensities⁹. The volume fraction of retained austenite (X_γ) was determined by the direct-comparison method using integrated intensities of the (110) and (211) peaks of ferrite, and the (111), (220) and (311) peaks of austenite. The carbon content of the austenite was determined using the relationship¹⁰.

$$a_\gamma = 0.3548 + 0.0044 C_\gamma \quad (3)$$

where a_γ is the lattice parameter of austenite in nanometres and C_γ is its carbon content (wt%). The (111), (220) and (311) peaks of austenite were used to estimate the lattice parameters.

The heat tinting of the prepared samples, after their light etching in 2% nital, improved phase identification. Heat tinting involved heating in an oven with no protective atmosphere at 260 °C for 5.5h and cooling to room temperature. With this technique the various phases appear in different vibrant colors. These colors are: untransformed austenite - light blue, reacted high

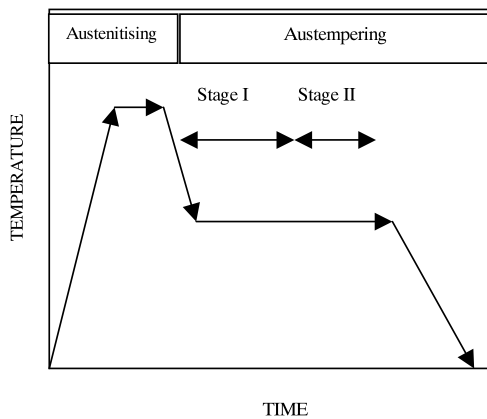


Figure 1: Schematic diagram of the austempering heat-treatment process;

Slika 1: Shema procesa austempering toplotne obdelave

carbon austenite – light brown, ferrite – beige, eutectic carbide – white or cream, martensite – dark blue.

During point counting at last 2000 points were counted at a magnification of x500. The number of counts was increased with the decreasing volume fraction of martensite to maintain a low standard deviation.

3 RESULTS AND DISCUSSION

3.1 Characteristics of as-cast irons

The as-cast microstructure consists of graphite nodules surrounded by ferrite in a pearlitic matrix. Image analysis measurements for nodule count, amount of ferrite, pearlite and carbide are given in Table 2.

Table 2: Quantitative measurement of structural features of as cast iron

Graphite, vol.-%	Ferrite, vol.-%	Pearlite, vol.-%	Carbide, vol.-%	Nodule count, mm ⁻²
12.7	41.0	46.3	0.05	120

The austemperability results are presented in Figure 2 as the critical bar diameter that can be austempered without pearlite formation. Voight and Loper¹¹ who, have examined austemperability, suggest the following regression function for calculating the critical bar diameter:

$$D_c = 124C_\gamma^\circ + 27(\%Si) + 22(\%Mn) + 16(\%Ni) + 25(\%Mo) + 1.68 \times 10^{-4}T_a^2 + 12(\%Cu)(\%Ni) + 62(\%Cu)(\%Mn) + 88(\%Ni)(\%Mo) + 11(\%Mn)(\%Cu) + 127(\%Mn)(\%Mo) - 20(\%Mn)(\%Ni) - 137 \quad (4)$$

where C_γ° is the carbon content of the matrix austenite at the austenitising temperature and T_a is the austempering temperature. The austenite carbon content depends on the austenitising temperature (T_γ) and the iron

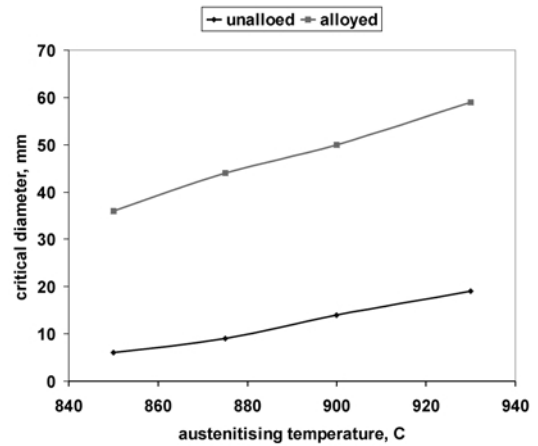


Figure 2: Variation of critical bar diameter that can be austempered without pearlite formation as function of austenitising temperature for Ni-Mo-Cu iron and unalloyed iron austempered at 350 °C (Ref. 13)

Slika 2: Sprememba kritičnega premera palice, ki se lahko austempra brez nastanka perlita v odvisnosti od temperature austenitizacije za NiMoCu litino in nelegirano litino austemprano pri 350 °C (Ref. 13)

composition. This dependence is frequently described by the approximate expression¹²:

$$C_\gamma^\circ = T_\gamma / 420 - 0.17(\%Si) - 0.95 \quad (5)$$

and the results are given in Table 3.

The second curve (Figure 2) represents some previous measurements on unalloyed ductile iron austempered at 350 °C¹³. The critical diameters of the ductile iron increase significantly as the content of alloying additions and austenitising temperature increase.

3.2 Microstructural changes after different austenitising treatments

The morphology of the matrix after austempering depends on the austenitising temperature and austempering time. Figures 3 (a-d) show the microstructures taken from impact-test specimens austenitised at 850, 875, 900 and 930 °C, respectively, and austempered at 350 °C for 120 min. All the microstructures exhibit upper bainite in the form of relatively coarse ferrite needles in an austenite matrix. The microstructure of the specimen austenitised at 850 °C contains various proportions of pro-eutectoid ferrite, bainitic ferrite and austenite (Figure 3a). The pro-eutectoid ferrite occurs as a result of a low austenitising temperature, i.e. as a result of austenitising the iron in the austenite-ferrite-graphite region of the phase diagram (Figure 4)¹⁴.

It can be seen from Figure 5a that as the austenitising temperature increases from 850 to 930 °C the retained-austenite content increases from 27 to 41 %.

The increase in austenite content with austenitising temperature can best be explained by referring to the free-energy-composition diagram for austenite, ferrite

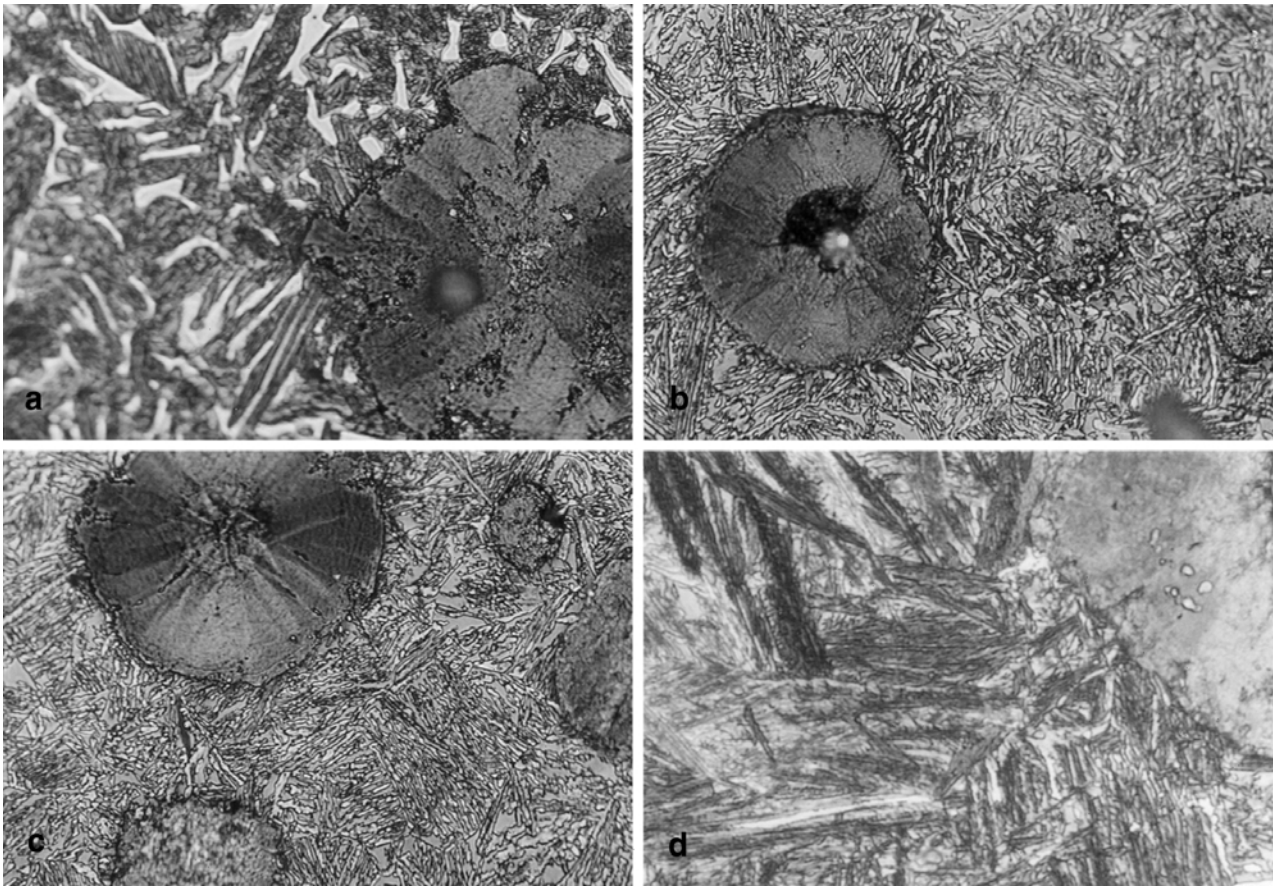


Figure 3: Effect of austenitising temperature on the microstructure of specimens austempered at 350 °C for 240 min, etched in 2% nital (optical) 850 °C (x500); b) 875 °C (x350); c) 900 °C (x350); d) 930 °C (x500)

Slika 3: Vpliv temperature austenitizacije na mikrostrukturo vzorcev, ki so bili austemprani 240 min. pri 350 °C, jedkano z nitalom, optično. 850 °C (x500); b) 875 °C (x350); c) 900 °C (x350); d) 930 °C(x500)

and cementite phases. **Figure 4** shows that if the matrix-carbon content decreases then the driving force from austenite to the metastable product, ferrite, and high-carbon austenite increases. This is indicated by a-a' increasing to b-b'. The higher driving force allows more nuclei to form and leads to a more rapid transformation. Thus for a given austempering temperature and time, the bainite transformation reaction is more advanced in those structures austenitised at the lower temperature, consequently these structures are more homogeneous and exhibit improved mechanical properties.

Figure 5b shows that as the austenitising temperature decreases from 930 to 850 °C the martensite content decreases from 33 to 19% for a 10 min austempering time. It has been shown that the rate of bainite formation increases as austenitising temperature decreases. On the other hand, the more advanced the bainite reaction the more carbon is rejected, resulting in a more stable retained austenite. Thus this austenite is less likely to transform to martensite as the specimen cools to room temperature or as stress is applied to it. **Figure 3d** shows that the structure austenitised at 930 °C contains martensite, and as a consequence, the transformation of

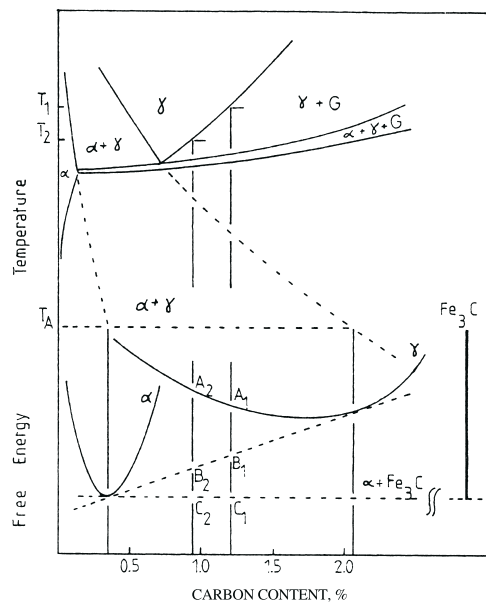


Figure 4: Schematic Fe-C-Si phase diagram and free energy curves for ferrite (α), austenite (γ) and cementite (Fe_3C) phases

Slika 4: Shematičen Fe-C-Si fazni diagram in krivulje proste energije za ferit (α), austenit (γ) in cementit (Fe_3C)

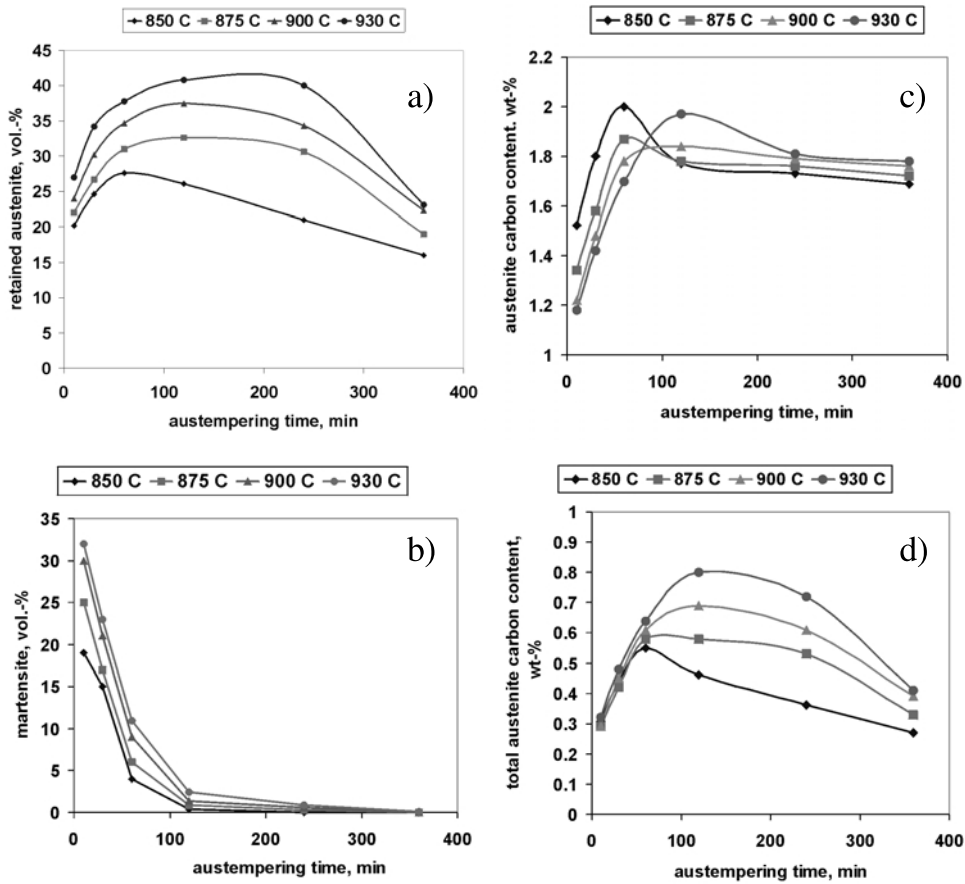


Figure 5: Variation in a) volume of retained austenite; b) martensite; c) austenite carbon content; d) total austenite carbon content; with austempering time for iron austempered at 350 °C and austenitised at 850, 875, 900 and 930 °C for 120 min

Slika 5: Sprememba v a) volumnu zaostalega austenita; b) martenzita; c) vsebnost ogljika v austenitu; d) skupna vsebnost ogljika v austenitu s časom austempranja pri 350 °C in po austenitizaciji 120 min. pri 850, 875, 900 in 930 °C

unstable austenite to martensite upon cooling to room temperature or the application of stress. In austempered,

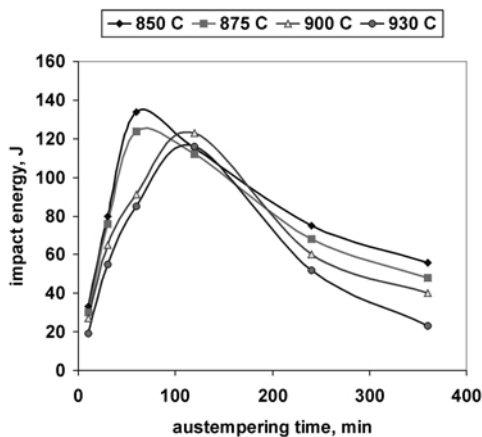


Figure 6: Variation of impact energy with austempering time after austenitising at 850, 875, 900 and 930 °C for 120 min

Slika 6: Sprememba udarne energije s časom austempranja pri 850, 875, 900 in 930 °C v trajanju 120 min

ductile iron it is difficult, even for the expert eye, to identify the various phases with black-and-white photomicrographs. To assist in the identification of the phases the heat-tinting technique was used. Martensite, which appeared as dark blue was evident.

This accounts for the low impact-energy results.

Another important point is that with the higher temperature austenitising more carbon is taken into solution by the original austenite (**Table 3**). Thus the austenite remaining after austempering may be stabilized even if a smaller percentage of ferrite is formed. As can be seen from **Table 3**, increasing the austenitising temperature from 850 to 930 °C increases the carbon content at the austenitising temperature from 0.63 to 0.81%. The actual carbon content of the austenite was made using X-ray diffraction techniques (**Figure 5c**).

Table 3: Matrix austenite carbon content after austenitisation

Austenitising temperature, °C	850	875	900	930
Carbon content in austenite, vol %	0.63	0.68	0.74	0.81

3.3 Kinetics of transformation

The kinetics of the process can be revealed by the structural parameters, austenite volume fraction (X_γ) and austenite-carbon content (C_γ), both determined by X-ray diffraction. The variations of X_γ and C_γ versus austempering time for the treated ductile iron are shown, respectively, in **Figures 5 (a, c)**. It is observed that a high austenitising temperature of 930 °C results in a relatively larger volume of retained austenite when compared to the iron austenitised at lower temperatures. However, this iron has the lowest austenite-carbon contents for all the austempering times. In contrast, the iron austenitised at 850 °C contains lower levels of retained austenite but the highest carbon contents in the retained austenite. This austenite is thermally and mechanically stable which leads to optimum impact properties (**Figure 6**).

In **Figure 5d** the total austenite-carbon contents in the original austenite matrix ($X_\gamma C_\gamma$) are plotted versus austempering time. The figure shows that the magnitude of the product $X_\gamma C_\gamma$ for an austenitising temperature of 930 °C increases to a higher maximum value than for the sample treated at 850 °C. Therefore, less original austenite carbon is dissolved within the austenite matrix at the lower austenitising temperature.

Increasing the austenitising temperature to 930 °C increases the austempering time needed to reach the maximum retained austenite. At 930 °C, the maximum is reached for about 120 min, but at 850 °C the maximum is reached for less than 60 min. This is in agreement with the theory, and is explained by the free-energy diagram.

The austempering time has an important influence on the mechanical properties and microstructure. As the austempering time is extended to 120 min, the volume of retained austenite starts to decrease. This decrease is a result of the second-stage reaction in which the high-carbon austenite starts to decompose into ferrite plus carbides. As shown in **Figure 5c**, the austenite-carbon content is also lowered that leading to further transformation to bainitic ferrite and carbides. The precipitation of carbides reduces impact-energy values.

4 CONCLUSIONS

The critical diameters of ductile iron increase significantly as the content of alloying additions and austenitising temperature increase.

Decreasing the austenitising temperature slightly decreases the volume of retained austenite but increases the austenite-carbon content and optimized impact toughness. The reduced carbon content of the matrix austenite increases the driving forces for the stage I reaction and reduces the austempering time needed to obtain high-carbon austenite. This results in a more uniform structure.

Heat treatment at 850 °C resulted in pro-eutectoid ferrite in the microstructure. This is attributed to the lower austenitising temperature which reduces the solubility of carbon in austenite and introduces a three-phase region of ferrite, austenite, and graphite in the Fe-C-Si equilibrium diagram.

The drop in impact energy at an austenitising temperature of 930 °C appeared to be caused by a reduction in austenite-carbon content as a result of carbide precipitation.

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