

# Investigation of Structure and Properties of Austempered Materials Produced by Traditional Austempering and Newly Developed Hard Surface Austempering

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## Abstract

This study was conducted to compare the improvement in the properties of ductile iron with a newly developed austempering process by conventional austempering. Patents have been obtained for this new method and the materials produced (TPE Patent Number: TR 2020-22197 B and TR 2020-22199 B). The new surface hardening austempering process (HSA) and conventional austempering processes (A) were applied under similar conditions. The samples were austenitized at 900 °C for 60 minutes and tempered at 350 °C for 30 and 240 minutes. While ductile iron material with an ausferritic structure was produced with conventional austempering, ductile iron with an ausferritic interior, but martensitic surfaces with HSA. As a result of the experiments, it was seen that the properties of the DI samples were improved with both heat treatments, but the highest properties were obtained at the end of the HSA processes.

**Keywords:** New heat treatment process, Hard surface austempering, Ductile iron.

## 1. Introduction

The quality of technologically required materials is also improving with technological developments. The superior properties of cast iron and steel can be further improved by heat treatments. One of these heat treatments is austempering. Therefore, the most common types of materials that have higher toughness, ductility and strength compared to other types of metallic materials are austempered cast iron and steel materials. The austempering process creates an ausferritic matrix structure in cast irons and a bainitic matrix structure in steels. These structures improve the material properties to a great extent (1-6). As in steels, the matrix structure in cast irons is called bainite due to the similarity in shape [1-5]. However, it has been shown by studies that the microstructures of austempered ductile iron (ADI) and steel (AS) are not the same [6-8]. Some of these studies have proven that ADI is an ausferritic structure consisting of ferrite + high carbon austenite, and other researchers have also supported this view [7-11]. ADI materials have been standardized in five material groups by ASTM (ASTM 897-90). The mechanical properties of ADI vary greatly depending on the austempering process parameters, namely austempering temperature and time, and alloy addition [5, 12-15]. ADI's chemical composition, especially with its high C content, and its

matrix structure, which allows for a quick transformation, allow it to be transformed much more quickly than steel. Equivalent strength to steel, wear resistance properties, and a 10% lower density are important advantages of ADI materials [16]. In addition, ADIs are better than wrought aluminum in terms of strength-to-weight ratio, and better than steel in terms of impact and tensile strength, since they contain graphite [2-4, 11, 17-29].

Since it has an ausferritic (carbon enriched ferrite + austenite) matrix, ADI has better mechanical properties compared to ductile iron. This is due to the matrix structure. This structure is formed as a result of a two-step heat treatment. In the first step, the DI structure is completely austenitized at 850–950 °C. In the second step, it is cooled to the target tempering temperature (250–450 °C) and tempered there for a certain period of time. This waiting is an isothermal process and the ferrite + high-carbon austenite structure is formed. In the first stage of tempering, high-carbon austenite is formed depending on the process temperature. The tempering time is very critical. Isothermal heat treatment (tempering) is performed within a process window (process range) in which the austenite is completely transformed [1-3, 7-11, 18-26]. ADIs are unique engineering materials that combine strength

and ductility [4–13, 22–33]. Studies show that the wear properties of ductile and brittle materials are similar to other cast irons and steels [14–17]. There are many studies on austempered cast iron and steel materials [22–47]. Literature reviews show that a secondary heat treatment is required when a harder surface than ADIs is desired [31–43]. With the hard surface austempering (HSA) heat treatment developed and the subject of this article, it is possible to produce bainitic/ausferritic iron-based materials by austempering the hard surface and core in a single process without the need for austempering and subsequent surface hardening. In this study, the differences between conventional austempered (A) and hard surface austempered (HSA) ductile iron (DI) material production processes with a single heat treatment under similar production conditions and the differences in the materials produced by both methods are presented comparatively.

## 2. Materials and Methods

The chemical compositions of ductile iron used in the present study are given in Table 1. The ductile iron was produced in an induction furnace in an industrial foundry. The wire addition method was treated with a 1000 kg melt iron with MgFeSi alloy at 1460 °C. Two-stage inoculation was carried out with a FeSi alloy. The melt at 1420 °C was cast into green sand molds. In this study, standard Y-block part castings were made. Austempered and hard surface austempered samples prepared are subjected to austenitizing treatment at 900°C for 60 minutes and then to an isothermal transformation process at 350°C for 30 and 240 minutes in a salt bath of 50% KNO<sub>3</sub> and 50% NaNO<sub>3</sub> mixture. Temperature was measured during heat treatment, using a K-type thermocouple spot-welded to the sample. The austenitizing treatment was performed by embedding samples into their chips to prevent decarburization. A summary of the heat treatment applied is given in Table 2. After standard polishing processes of the samples were completed, an optical microscope was

used for metallographic and quantitative analysis.

Table 1. Chemical composition of ductile iron samples produced for this study (%)

| C     | Si    | Mn    | P     |
|-------|-------|-------|-------|
| 3.96  | 1.74  | 0.293 | 0.026 |
| S     | Ni    | Cu    | Cr    |
| 0.018 | 0.039 | 0.96  | 0.042 |

Table 2. Heat treatment conditions

| Specimens Code | Heat Treatment Condition            |
|----------------|-------------------------------------|
| N1 (As-cast)   | No heat treatment                   |
| N2 (ADI)       | 900 °C-1 hour + 350 °C -30 minutes  |
| N3 (ADI)       | 900 °C-1 hour + 350 °C -240 minutes |
| N4 (HSADI)     | 900 °C-1 hour + 350 °C -30 minutes  |
| N5 (HSADI)     | 900 °C-1 hour + 350 °C -240 minutes |

Test samples were prepared by machine from pieces taken from standard Y-block. Using standard metallographic procedures, sand and permanent mold cast ductile iron samples were prepared for microstructural examination. 2% nital was used to reveal microstructures. Jeol 6060 LV 0.5-30 kV and 15X-100.000X scanning electron microscope (EDS faz analyze) and Leica DMI 5000M optical microscope were used for microstructural characterization. The count of microstructural features (matrix structure, graphite nodule and volume fraction of ledeburite/carbides in the cast parts was measured quantitatively with Leica Application Suite version 4.6 image analysis software. Hardness tests were conducted in Emcotest Duravision 200 universal hardness tester with Rockwell C hardness method (150 kgf load and 120° diamond cone indenter). Hardness was scanned from all areas. Three specimens were measured for each condition. Tensile tests were applied to samples taken from thin and thick sections. The tests were performed by connecting an extensometer to a 5-ton capacity Instron 3369 tensile test machine with a brand computer-controlled device. % elongation, fracture energy, tensile and yield strength values were obtained from at least 3 samples tested for each product.

## 3. Results and Discussion

### 3.1. Evaluation of Structural Properties

As an alternative to conventional austempering heat treatment, ductile iron materials that have been subjected to hard surface (material exterior) austempering (HSA) heat treatment are called Hard Surface Austempered Ductile Iron (HSADI). In this study, ADI and HSADI materials produced by ductile iron, conventional austempering (A) and hard surface austempering (HSA) processes were compared in terms of structural and mechanical properties. These new methods and materials have been patented (Patent Number: TR 2020-22197 B and TR 2020-22199 B) [44,45]. First of all, it is important to understand the fundamental difference between ADI and HSADI materials. The structural structure of both materials is shown schematically in Figure 1. While the ADI material (on the right) has an ausferritic structure from the outermost surface to the center of the material, the HSADI material (on the left) has a martensitic structure on the outer part (surface and 1-5 mm inward) and an ausferritic structure from the inner part to the center. Therefore, HSADI materials are hard-surface materials. When HSADI is examined especially in terms of hardness, the hardness of the inner part can be low (approximately 25-45 HRC) and the outer surface hardness can be high (40-60 HRC for a distance of 1-5 mm or more, depending on the request and the applied heat treatment conditions). This is provided by this new heat treatment, HSA.

When examining this subject, many researchers may wonder whether other processes can provide the structure and properties of ductile iron. Yes, especially the surfaces of austempered ductile irons (ADI) can be hardened, the outer part can be produced with a martensitic structure, and their surface hardness can be increased compared to the inner parts.

Martensitic structure in iron alloys is formed due to a non-diffusion structural transformation, due to rapid cooling. This process is generally known as martempering. Martensitic structure increases the hardness significantly due to its high strength and high C content against the forces applied to the

material. However, this situation also causes a decrease in ductility and toughness. Serious brittleness occurs especially in cast irons. Therefore, after austempering of cast irons, it is preferred to apply surface hardening process (induction, flame, etc.) again [33-42]. However, in this study, a newly developed HSA heat treatment was applied to eliminate these negativities of martempering and surface hardening with a secondary process. HSA heat treatment allows the surface of the material to be martensitic (like martempering) and the inner parts to be ausferritic (like austempering). Therefore, while martensitic structure is formed in the outer parts (Figure 1-3) (from the surface to the desired depth), ausferritic structure is formed in the inner parts. While also improving its toughness and ductility, the ausferritic structure [5-20] increases the strength and hardness of cast iron. Therefore, since only the outer surface is martempered compared to fully martempered material the negative effects on the outer surface are reduced.

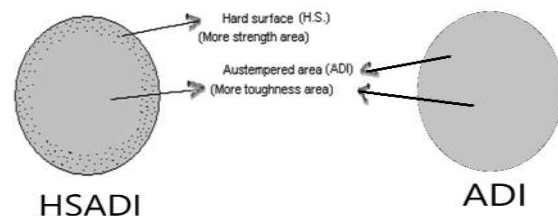


Figure 1. Schematic representation of the change in the material structure of the sample as a result of classical and new type heat treatment application.

On the other hand, an advantage of this new method is that there is no need to use additional materials (C, N, B, etc.) to obtain a harder surface than traditional austempered materials. On the other hand, these materials can also be used if desired. In other words, there is no need for surface hardening processes such as nitriding, cementation, boronization and carbon-nitriding. The processes are carried out completely according to the traditional austempering process conditions. Only a new process approach has been added to increase the surface hardness. The application stages and the resulting structures are given in Figure 2.

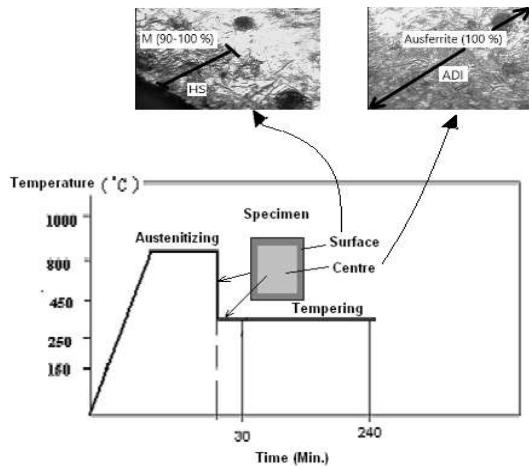


Figure 2. Schematic representation of the new heat treatment process (Hard Surface Austempering (HSA)) on the conventional austempering heat treatment cycle chart

In traditional austempering, the DI material is directly placed in a salt bath after austenitization and subjected to the tempering process. However, in the HSA method, just before the DI material is placed in the salt bath after the austenitization process, only the surfaces are suddenly cooled below the  $M_s$  temperature for a short time with suitable and special cooling equipment/devices. During this cooling, a martensitic structure is formed on the surfaces (optionally 1-5 mm). Here, the cooling rate and duration are of great importance in order to form a martensitic region with the desired thickness. The material is immediately immersed in the salt bath to prevent the formation of unwanted structures (pearlite, etc.) just below the surface. Thus, the formation of the desired ausferrite (in cast irons)/bainite (in steels) structure is guaranteed (Figure 3).

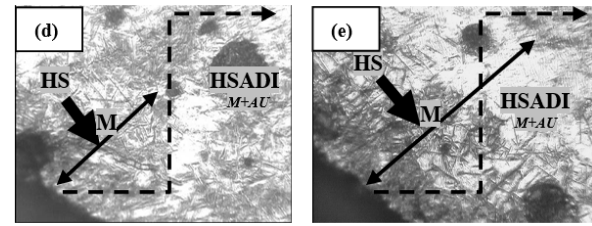
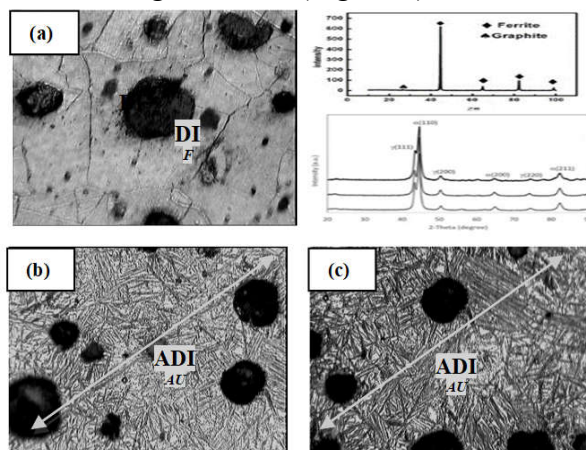


Figure 3. Images of specimen microstructure produced by austempering at different heat treatment conditions a) N1 (As-cast Ferritic DI), b) N2 (30 min. tempering ADI), c) N3 (240 min. tempering ADI), d) N4 (30 min. tempering HSADI) and e) N5 (240 min. tempering HSADI) materials (DI: Ductile Iron, HS: Hard Surface, HSADI: Hard Surface Austempered Ductile Iron, ADI: Austempered Ductile Iron, F: Ferrite, M: Martensite, AU: Ausferrite)

### 3.1. Evaluation of Structural Properties

Figure 3 shows that the DI material (sample 1) is completely ferritic, the traditional austempered materials (samples N2 and N3) are ausferritic, and the HSA-treated materials (samples N4 and N5) have martensitic surfaces and ausferritic interiors. A single heat treatment can produce materials with different (martensitic) surface parts and different (ausferritic) interiors. However, it is seen that a second heat treatment must be performed to produce a material with a different exterior structure [31-36,40-43]. This success constitutes the first of the most important advantages of this new method. Secondly, this structure will also cause the exterior to be harder and more resistant to wear. Because as the hardness increases, the wear properties improve [33,36-38]. However, hardness also brings about brittleness. In the new HSADI material produced with this new heat treatment, the brittleness of the exterior is differentiated by the ausferritic structure in the interior, which increases its toughness. As is known, in materials with a hard exterior, it is generally preferred that the inner part has high toughness. For these purposes, surface hardening processes are applied with secondary processes [34-39,41-43].

Table 3 and Figures 4 and 5 show the mechanical properties of DI, ADI HSADI materials. DI materials have better mechanical properties than many cast irons, nonferrous alloys and some steels. DI materials can have 400-800 MPa strength, 140-300 HB hardness, 2-18% elongation and

5-20 J toughness depending on their matrix structures and alloy combinations. Especially DI materials with ferrite matrix have high toughness and ductility as well as good strength. Therefore, they are preferred [20-22,29-32,46-48]. In this study, ferritic DI material was preferred as the starting material without adding the alloying element effect and not having a different effect on the heat treatment results. Ferritic DI (sample N1) material turns into ADI material when subjected to conventional austempering heat treatment. Depending on the heat treatment conditions, it can have 900-1600 MPa strength, 280-420 HB hardness, 7-24% elongation and 8-30 J toughness. The most important parameters affecting these properties are especially the ausferritic structure and the tempering temperature and time that affect it. This is called the process window. During tempering, the austenitic structure transforms into the ausferritic structure. While the temperature determines the formation of fine or coarse ausferrite structure, the tempering time determines the volume ratio of ausferrite and untransformed austenite. These structural properties also affect the mechanical properties [4-12,29-35]. In this study, the effect of the process window was tried to be observed in both ADI and

HSADI materials by changing the tempering time. In conventional austempered materials (ADI samples N2 and N3), the matrix structure consists mostly of untransformed austenite (85%) and ausferrite (15%) in a 30 min tempering time, while at the end of 240 min, there is no untransformed austenite left since a completely ausferritic (100%) matrix is formed. This structural transformation has completely increased the mechanical properties compared to DI. There has been an increase of 80-150% in strength, 20-50% in hardness, 20-50% in elongation and 25-45% in toughness. Austempering studies [2-4,8-12,20-23,27-31,46-48] have also shown that the mechanical properties increase at varying rates depending on the heat treatment time and ausferritic transformation. Because the high carbon austenite and ferrite mixture in the ausferritic structure improves both strength and hardness, as well as ductility and toughness. On the other hand, it has been shown that carbide precipitation reduces toughness and ductility at the end of much longer tempering times. For this reason, the process interval of 30-240 minutes was selected to prevent carbide precipitation.

Table 3. Mechanical properties of DI, ADI and HSADI specimens

| Specimens    | Tensile Strength,<br>(Mpa) | Yield Strength,<br>(Mpa) | Hardness<br>(HRC) | Elongation<br>(%) | Toughness<br>(J) |
|--------------|----------------------------|--------------------------|-------------------|-------------------|------------------|
| N1 (As-cast) | 461                        | 397 ↑                    | 8 ↑               | 5 ↑               | 15 ↑             |
| N2 (ADI)     | 907 ↑                      | 794 ↑                    | 30 ↑              | 7 ↑               | 24 ↑             |
| N3 (ADI)     | 1009 ↑                     | 812 ↑                    | 34 ↑              | 10 ↑              | 32 ↑             |
| N4 (HSADI)   | 1182 ↑                     | 951 ↑                    | 32* ↑<br>40** ↑   | 8 ↑               | 44 ↑             |
| N5 (HSADI)   | 1293 ↑                     | 1093 ↑                   | 35* ↑<br>43** ↑   | 12 ↑              | 48 ↑             |

\* Hardness in sample center - \*\* Hardness in sample surface

For the purpose of comparing with ADI material, the same heat treatment conditions were preferred for similar reasons in the heat treatment of HSADI material. When these samples (N4 and N5) were examined, it was determined that the material surface was martensitic up to a depth of approximately 2.4 mm and the inner parts were composed of varying proportions of ausferritic (15% ausferrite+85% untransformed austenitic in 30

minutes of tempering and 100% ausferritic matrix in 240 minutes of tempering). In HSA heat treatment, the outer part was quickly cooled to below the Ms (martensite start) temperature in very short periods just before the material was immersed in the salt bath after austenitization. As a result, martensitic transformation occurred on the surfaces of both N4 and N5 materials. It was observed that the martensite formed was the lath type frequently formed in cast irons. This

martensite type is also frequently seen in surface hardening processes [24-28,36-43]. Due to this structure, the surface hardness in the N4 and N5 samples to which the HSA process was applied is approximately 20% higher than the hardness in the 3 mm inner part. In these inner parts, the hardness is lower because the structure is an ausferritic matrix (Table 3, Figures 5 and 6).

The mechanical properties of cast irons depend on conditions such as chemical analysis, cooling conditions, casting conditions and heat treatment. Some alloying elements increase strength and hardness by forming compounds and/or solid solutions in cast irons, while others improve ductility and toughness. Mechanical properties also change depending on the change in cooling rate during production. In particular, the number of grains increases with rapid cooling, increasing strength and hardness. Heat treatment application is one of the main factors affecting the development of mechanical properties in cast irons. Depending on the formation of hard and brittle or, on the contrary, soft and ductile structures, heat treatment conditions determine mechanical properties [3-9,15-18,33-42]. Austempering stands out as a heat treatment in materials where mechanical properties of opposite characters (both high ductility and hardness) are desired. On the other hand, there may be situations where it is desired to obtain more strength and hardness without reducing toughness and ductility. To achieve this goal, it is also common to apply surface hardening as an additional process after austempering. This additional process creates additional requirements such as energy and cost. Due to all these needs and conditions, this new HSA heat treatment was developed. In this study, the HSA process, which can be used to obtain structural differences in cast iron by only changing the surface cooling rate during the austempering heat treatment, was compared with ADI materials and the changes in structure and

mechanical properties were examined (Table 3, Figures 4, 5 and 6).

When the mechanical properties obtained were examined, the tensile strength of ferritic ductile cast iron was improved by 97-119%, yield strength by 100-105%, hardness by 275-325%, elongation by 40-100% and toughness by 60-113% with conventional austempering. Since the change in the elongation of ADIs is affected by the heat treatment conditions (process window or tempering time), a single tempering temperature was selected in this study. Austempering increases the mechanical properties of iron alloys. The ferrite and/or pearlite matrix structures of these materials can provide mechanical properties up to a certain value. On the other hand, ausferrite structure is formed in cast irons and bainite structure in steels with austempering. Ausferritic structure is also in a layered arrangement like pearlite [31-40]. Ausferritic structure formed as a result of austempering consists of high carbon austenite and ferrite. This structure is much more resistant to loads and ductile than ferrite and/or pearlite. Therefore, ADIs have much higher properties than DI materials. Mechanical properties can be improved by 40-325% with austempering. There are many studies on how these properties are provided, their production, tests and comparisons, and these issues have been examined in detail [21-33]. When the properties obtained in the HSADI samples produced as a result of the newly developed HSA heat treatment were compared with DI, it was determined that the change was much greater. The hardness in the HSADI material was measured from two different regions. Because the outer part was rapidly cooled and the inner part was only an austempered material, the hardness was measured in this way. When DI and HSADI are compared, the inner hardness of the material has improved by 331-337% and the outer hardness has improved by 400-437%.

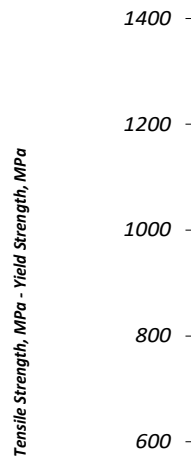


Figure 4. Elongation, tensile and yield strengths of DI, ADI and HSADI samples

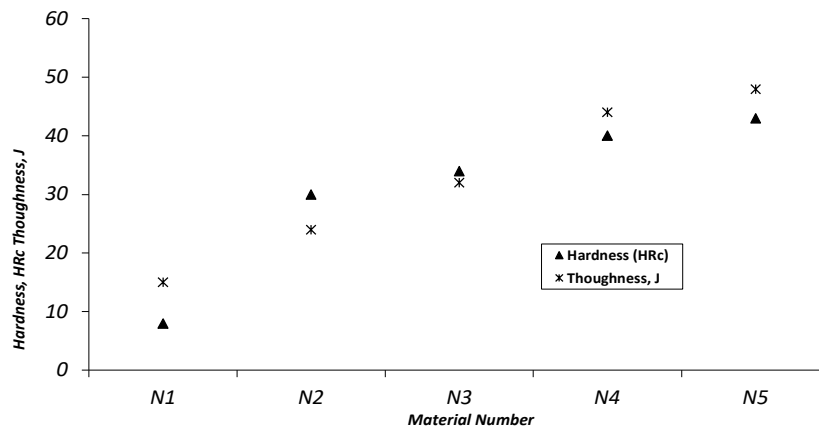


Figure 5. Hardness and toughness of DI, ADI and HSADI samples

When iron alloys are cooled rapidly (e.g. quenching), the material structure becomes martensitic. This structure increases hardness and strength, while reducing elongation and toughness. The tempering process positively affects ductility and toughness. The changes provided by tempering the martensitic structure of iron alloys and its contributions to material properties are known from the studies conducted [33-39]. In this study; While HSA process is performed, the surface of the HSADI sample was cooled very rapidly compared to the traditional ADI sample and a martensitic layer (Figure 3) was formed. While the martensitic structure reduces the deformation and flexibility capabilities of iron alloys, it increases the strength. This layer increases the mechanical properties requiring strength, while slightly reducing the properties requiring flexibility. Since high carbon content increases brittleness,

quenching and tempering are preferred especially for steels with low carbon content. Although limited studies have been conducted on cast irons, ductility decreases as in steels [38-44]. In this study, a slight decrease in ductility is observed in the HSADI sample since the surface part is cooled rapidly, similar to martempering. On the other hand, this decrease is not as much as in traditional martempering. On the other hand, unlike martempering, especially toughness increases in austempered materials. As can be seen from this study, ductility and toughness increase in the HSADI sample. Because toughness and ductility increased in the ADI sample with austempering compared to the DI sample. Under the same conditions, these properties also increased in the HSADI sample, which was partially cooled rapidly but generally austempered, compared to the DI sample. This situation shows that the ausferritic structure is

effective in increasing toughness, the hard layer on the surface provides preliminary resistance to the initial forces on the material, and the ausferritic structure in the lower (inner) part absorbs the impact to a large extent. In this study, the effect of the process window was tried to be observed by changing the tempering time in both ADI and HSADI materials. In HSA materials (HSADI samples N4 and N5), the matrix structure mostly consists of untransformed austenite (94%) and ausferrite (8%) in the 30-minute tempering time, while at the end of the 240-minute tempering time, a completely ausferritic (100%) matrix is formed, so there is no untransformed austenite left. This structural transformation further increased the mechanical properties compared to ADI. There was an increase of 28-30% in tensile strength, 20-35% in yield strength, 3-7% in hardness (26-33% in surface hardness), 14-20% in elongation and 50-100% in toughness. Conventional austempering studies [9-15,17-20] also show that

the mechanical properties increase at varying rates depending on the heat treatment time and ausferritic transformation. Because the high carbon austenite and ferrite mixture in the ausferritic structure improves both strength and hardness, as well as ductility and toughness. On the other hand, carbide precipitation has been shown to reduce toughness and ductility at the end of much longer tempering times. Therefore, a treatment interval of 30-240 minutes was chosen to prevent carbide precipitation. This paper is the first study on HSA heat treatment, and detailed studies are ongoing. However, from all these results, it can be seen that HSADI material is unique and has higher properties compared to not only DI but also ADI material. Studies on processing temperature and time are ongoing for further development of this material. At the same time, research on the effects of the environment during austenitization is ongoing.

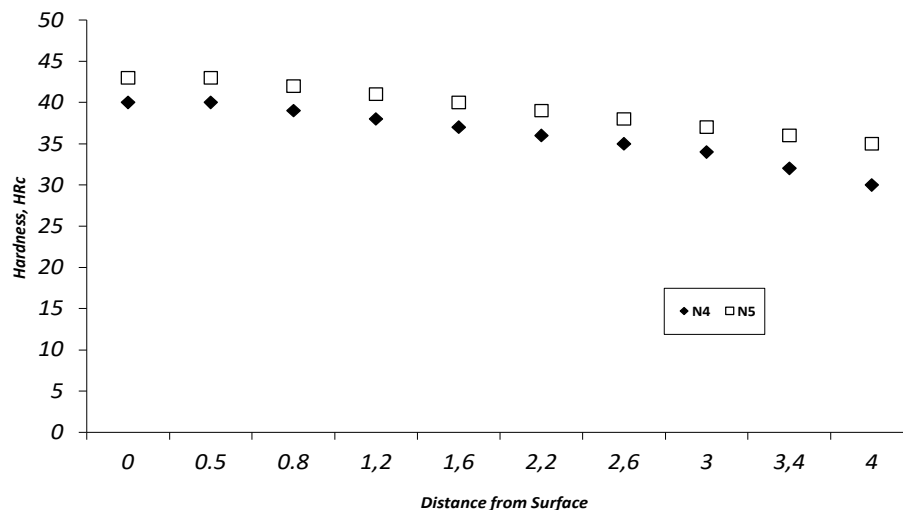


Figure 6. Change in hardness from surface to interior in HSADI samples

### 3. Conclusions

The following results were obtained as a result of the experiments:

- ✓ As a result of the austempering heat treatment, the mechanical properties of the materials obtained with the traditional austempering process are generally 40-325% more improved than the DI material.
- ✓ The hard surface austempering (HSA) process was developed by making a modification of the austempering heat treatment. The outer surface of the material produced in this process has a martensitic structure and the inner parts have an ausferritic structure.
- ✓ In this study, ductile iron materials were examined using the HSA process and it was determined that the general properties of the HSADI material were much superior to both DI and traditional ADI materials.
- ✓ It was determined that the HSADI materials produced with this new process could have 3-100% more improved mechanical properties

than ADI materials. On the other hand, it was determined that they had 43-425% better properties than DI materials.

- ✓ Improvements were made in all properties, not just certain properties. However, especially the much higher surface hardness will bring much more advantages to this material in areas such as wear, corrosion, fatigue.
- ✓ This new HSADI material, which is generally superior to both DI and ADI materials, has the potential to be further improved by changing the process parameters.

#### Data Availability Statement

This article contains all the data produced or analyzed during this investigation. Further inquiries should be forwarded to the corresponding author.

#### Ethics Approval

Not applicable.

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#### Declaration Of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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