

The surface layer of austempered ductile iron investment castings properties

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Abstract

The article presents a unique process of carbonitriding and nitriding the precision casting surfaces of austempered ductile iron. The results of the research are pointing that adequate process parameters allow to obtain multiple increase of wear resistance and a significant increase of corrosion resistance. Also, changes of cast microstructure and hardness are presented.

Keywords: Austempered Ductile Iron, Nitriding, Carbonitriding, Wear resistance, Corrosion resistance

1. Introduction

ADI is generally known to possess high hardness, good wear resistance, high toughness and fatigue resistance [1]. It has also been proved that all these properties of ADI can be additionally improved when the formation of compressive stresses is induced on its surface through application of proper mechanical surface treatment, like shot peening or surface rolling, or a conventional operation of machining, i.e. turning, boring, grinding, and milling [1,2,3].

These types of treatment, and hence the initiation of transformation of a unstable austenite into martensite, besides a very favorable effect of improving the ADI properties, make its machining very difficult. Therefore, quite paradoxically, rather lower grades of this material are said to be preferred in production (the grades characterized by lower tensile strength and higher ductility) because of their much better machinability [9]. The above mentioned types of surface modification of ADI (shot peening and surface rolling included) require some machining which is usually also required after the heat treatment, since austenitisation causes decarburizing of the ADI surface layer and loss of the very advantageous properties. In a similar way, the

corrosive effect of salt baths commonly used for austempering has its negative impact on the casting surface. Mechanical working with tools is, however, quite troublesome in the case of complex or not readily accessible surfaces, which are to be cast in a near-net shape and ready-for-use condition. In such cases some special methods of the, so called, direct austempering of the ductile iron can be applied [7], or surface treatment other than the mechanical working can be useful, e.g. PVD process [4].

Thermo-chemical surface treatments, like nitriding or carbonizing, are the methods used commonly for modification of the surface layer in products made from various materials. These processes are usually combined with preheating of the treated element. In the case of ADI, the temperatures applied in conventional thermo-chemical treatments would destroy the ausferritic microstructure present in the ductile iron matrix and hence would deteriorate its properties.

Quite an interesting variation of the thermo-chemical treatment was a PVD process used by Feng et al. [4]. Due to this treatment, onto a specific grade of ADI castings the layers of TiN and TiCN were applied. The presence of these layers made it possible to increase the hardness in a surface layer and raise the ADI fatigue resistance to 22%, compared with the same material but untreated.

The layers were, however, very thin ($1\div 2\mu\text{m}$) and bound to the substrate by the forces of adhesion which limited the use of ADI castings to some special applications only.

It seems that to avoid the unfavorable transformation changing the microstructure of ADI matrix it could be possible to apply a thermo-chemical treatment but in a mode slightly different than the traditional process. It is a well-known fact that to produce ADI the castings made from ductile iron should be subjected to a heat treatment which consists in austenitising at a temperature of $850\div 950^\circ\text{C}$ and austempering at a temperature of $230\div 400^\circ\text{C}$. During austenitising it is usually necessary to provide in the furnace a protective atmosphere counteracting the surface decarburizing of castings. Allowing the castings to get decarburized makes machining after the heat treatment necessary to remove the undesired layer. On the other hand, the temperature of austenitising amounting to 850°C creates the possibility of an effective application of the process of carbonitriding case hardening in the cycle of ductile cast iron heat treatment by producing an appropriate active atmosphere in the furnace (Fig.1). So, it seems possible to produce simultaneously in ADI castings the required microstructures - in the core and on the surface layer. In the case of this specific thermo-chemical treatment, after the process of carbonitriding case hardening the surface of a casting should be ready for its immediate operation.

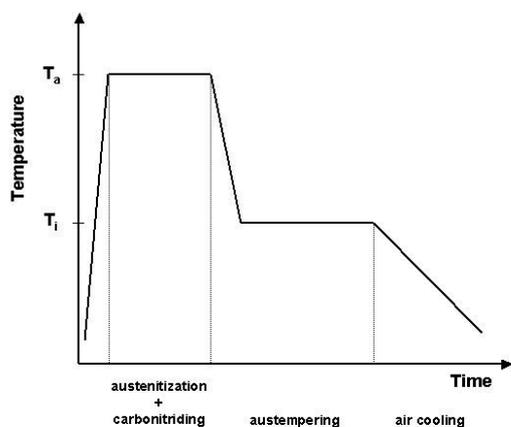


Fig.1. Schematic representation of the "surface-and-core" treatment of ductile cast iron; T_a – austenitizing temp., T_i – austempering temp.

The article presents the process of carbonitriding and nitriding adapted to thermal treatment, in which the austempered ductile iron is obtained. The research on microstructure and properties was performed; those are microhardness distribution, corrosion resistance and wear resistance.

2. Investigation methods

Precision castings of the austempered ductile iron were made at Department of Ferrous Alloys, Foundry Institute of Cracov, by melting the metal in the induction furnace of middle frequency and 500 kg capacity, with spheroidizing NiCuMg17 using the

"sandwich" method. The stepped castings presented at fig. 2 were made in ceramic moulds cooled in the air after pouring. Then, the castings with chemical composition presented at table no. 1 were cleaned off by sand blasting and subjected to the next process after cutting the gating system. Then, the ductile iron was subjected to a thermo-chemical treatment of nitriding and carbonitriding at Institute of Precision Mechanics. The parameters of treatment are presented at table 2.

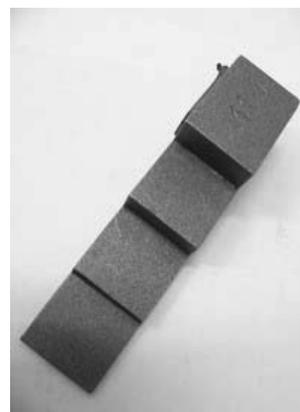


Fig.2. Stepped precision casting of ductile iron

After thermal treatment combined with thermo-chemical treatment, the castings were subjected to microhardness test with use of the Vickers $\mu\text{HV}-100\text{mg}$ microhardness tester and microstructure examination with use of the OLYMPUS IX-70 light microscope.

Table 1. Chemical composition of stepped precision castings [% by weight]

C	Si	Mn	P	S	Ni	Mg	Cu	Mo
3,40	2,80	0,28	0,035	0,015	0,02	0,055	0,72	0,27

Wear resistance test of specimens using "three rollers-cone" method was performed at Institute of Precise Mechanics with use of I-47-K-54 type machine, according to PN-83/H-04302, procedure no. 2-B/ZW-3/06. Wear marks on specimens were measured with use of the SAGEM N°133 device and 45 Steel counter-specimens. The specimens were tested at load of 100 MPa. The linear wear of specimens and the wear path were calculated. The diagrams of linear wear vs. time were made.

Corrosion resistance was tested with use of voltamperometric method. Measurements were carried at room temperature in $0.1\text{M Na}_2\text{SO}_4 + 0.1\text{M NaCl}$ oxygenated solution, $\text{pH}=3$ (pH corrected by sulphuric acid), with use of tri-electrode electrochemical vessel:

- hot electrode – surface of 0.39 cm^2
- reference electrode – saturated calomel electrode (SCE) connected with capillary through electrolytic bridge
- auxiliary electrode – platinum electrode (grid)

Before the measurement, specimens were degreased with acetone in an ultrasonic cleaner. Anodic voltamperometric curves were recorded at a rate of the potential change of 1 mV/s .

Measurement procedure was following:

- settling the resting potential (OCP), 60 min.
- recording the anodic curve from 0 V vs. OCP to 1.5 V vs. SCE or

recording the full polarization curve for chosen specimens from - 0.3 V vs. OCP to 1.5 V vs. SCE.

Table 2. Technological parameters of thermo-chemical treatment

Austenitization						Fluid bed austempering		Sign
No.	Temperature [°C]	Decarburization [h]	CH ₄ [%]	NH ₃ [%]	Duration [h]	Temperature [°C]	Duration [h]	
Carbonitriding								
1	860	-	5	10	1.5	370	1	ADI CN1
2	860	1	5	10	2.5	370	1	ADI CN2
3	860	2	5	10	3.5	370	1	ADI CN3
Nitriding								
4	840	-	-	100	1.5	370	1	ADI N1
5	860	-	-	100	1.5	370	1	ADI N2

3. Results and discussion

The results of static tension test (table 3) proved, that each of the treated materials comply with European and American standards for austempered ductile iron – ADI [6]. By examining the microstructure of ADI surface layer after carbonitriding process, it can be found that dark layer is occurring in every case (fig. 5). In carbonitriding process (ADI CN), the thickness of this layer is increasing along with extending the time of isothermal transformation. However, this does not take the effect of increasing the hardness measured from surface deep into material (fig. 6).

Surface layer look a little different after nitriding (fig. 3). Higher temperature of process allows creating a layer with less thickness in comparison with the temperature of 840 °C, but with hardness larger than the hardness of the core. The results of the strength test also show that the temperature of 840 °C for obtaining austempered ductile iron produce significantly lower strength at a maintained yield level, like for higher austenitization temperature.

Table 3. Mechanical properties of ductile iron after thermal treatment and thermo-chemical treatment

	ADI CN1	ADI CN2	ADI CN3	ADI N1	ADI N2
T.S. [MPa]	1068	1169	1148	875	1058
Y.S. [MPa]	776	874	837	559	759
El. [%]	9,7	10	9,7	10,3	9,7

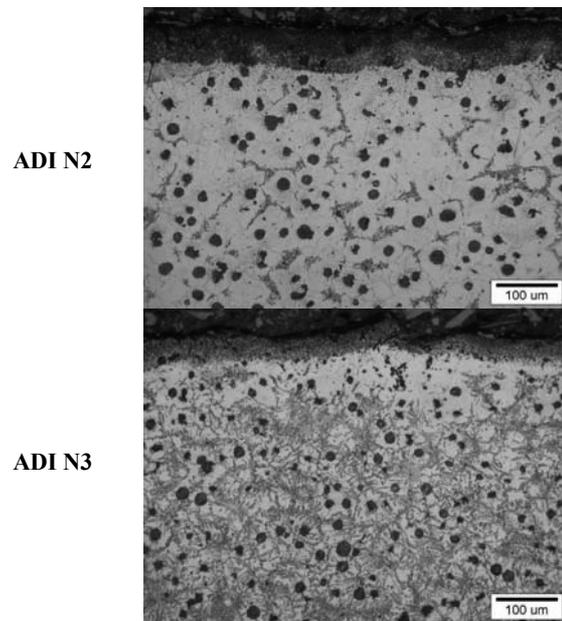


Fig. 3 Microstructure of surface layer in ADI castings after nitriding

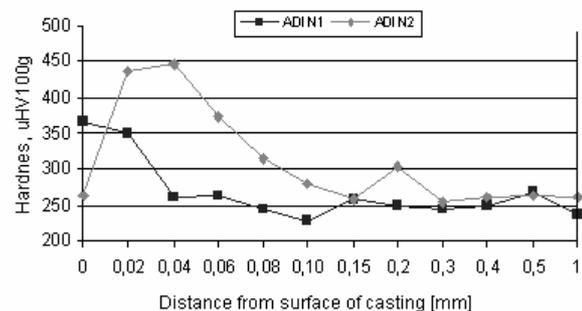


Fig. 4. Microhardness $\mu\text{HV}_{0,1}$ measured from the surface of ADI casting after carbonitriding

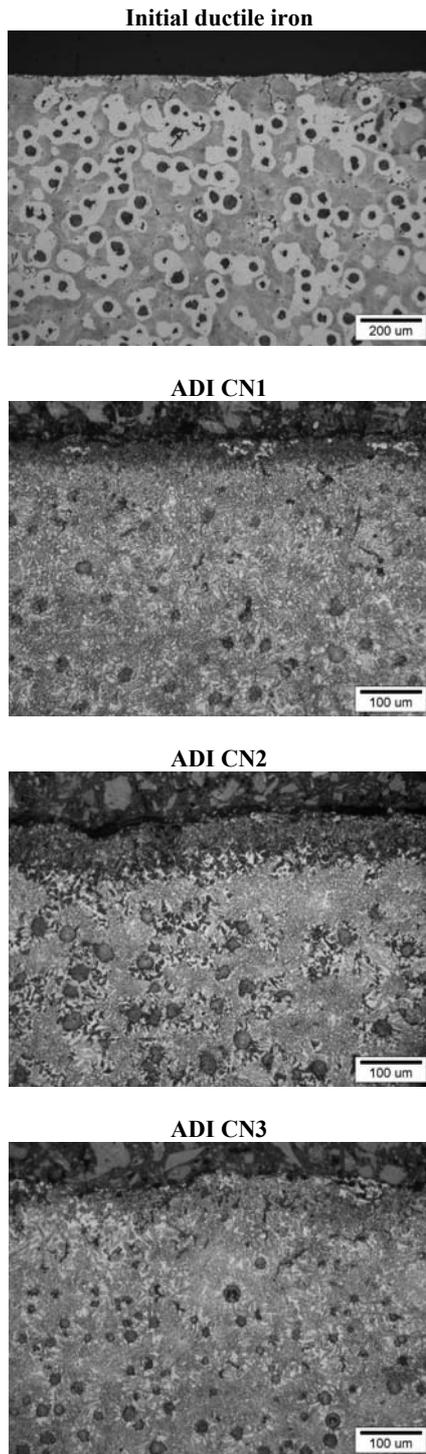


Fig. 5. Microstructure of surface layer in initial casting and carbonitrided castings – ADI CN: a) initial; b) ADI CN1; c) ADI CN2; d) ADI CN3

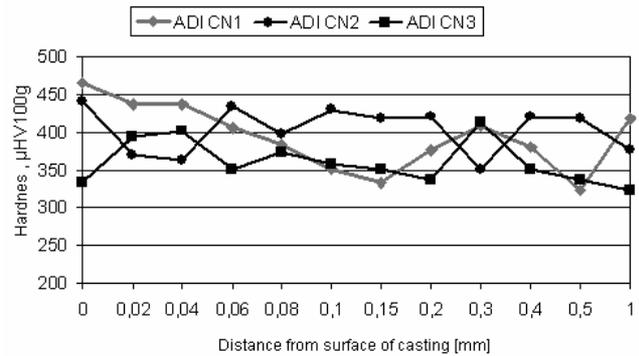


Fig. 6. Microhardness $\mu\text{HV}_{0,1}$ measured from the surface of ADI casting after carbonitriding

The results of wear resistance tests show that the wear resistance is higher in a cast iron processed with proposed carbonitriding and nitriding processes (fig. 7). Austempered ductile iron nitrided at the temperature of 840°C is characterized by highest wear resistance. This wear resistance is comparable with resistance of surface after carbonitriding at the temperature of 860°C during 90 min. Following diagrams show that those castings will have several times higher resistance comparing to conventional austempered ductile iron.

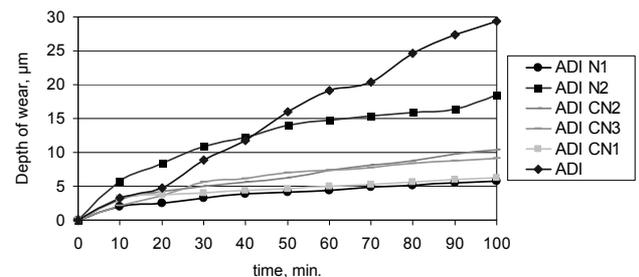


Fig. 7. Depth of wear evolution of the surface conventional ADI, nitrided and carbonitrided (consequently ADI, ADI N, ADI CN)

Anodic curves recorded at examined sulphate-chloride medium do not show features typical for pitting corrosion. Appearance of anodic curves indicates occurrence of general corrosion at given conditions (fig. 8).

Among the specimens, we can distinguish those with higher or lower susceptibility to corrosion. Anodic behaviour of pure austempered ductile iron without surface modifications at examined solution is a reference here. The base dissolution rate, including size of area subjected to anodic reaction of Fe dissolution, is characterized by values of current density for partial anodic reactions occurring at surface. In order to range tested materials in respect of susceptibility to corrosion, the values of current density for each specimen were evaluated at chosen potential, +30 mV away from stationary potential (within active dissolution range without side effects) (fig. 9). Then, values of

current density were evaluated at potential -300mv, which is after further polarization. Results are contained on following diagrams: Fig. 9 and 10.

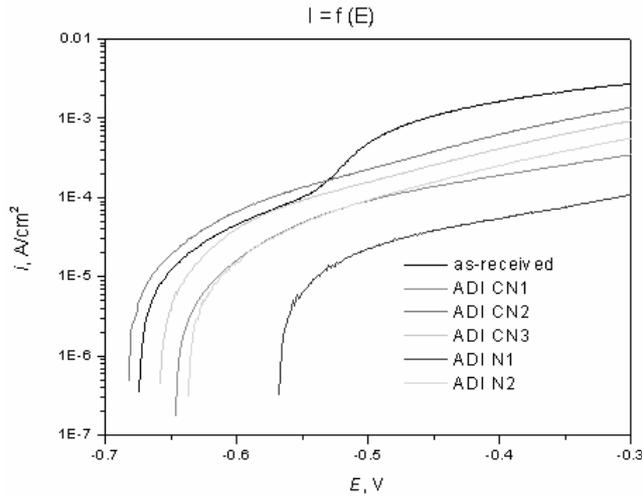


Fig. 8. Voltamperometric anodic curves at 0.1M Na₂SO₄ + 0.1M NaCl (pH=3) for austempered ductile iron carbonitrided (ADI CN) and nitrided (ADI N) specimens

First diagram (fig. 9) allows evaluating material and intensity of dissolution. The smaller the values of current density, the smaller the dissolution rate. On this basis, few specimens with values of current density smaller in comparison with austempered ductile iron without thermo-chemical treatment can be selected. Those specimens are: ADI CN1 and ADI N1. During further polarization at potential of -300 mV (fig. 10), above mentioned materials are also distinguished by better corrosion resistance in comparison to cast iron with non-modified surface.

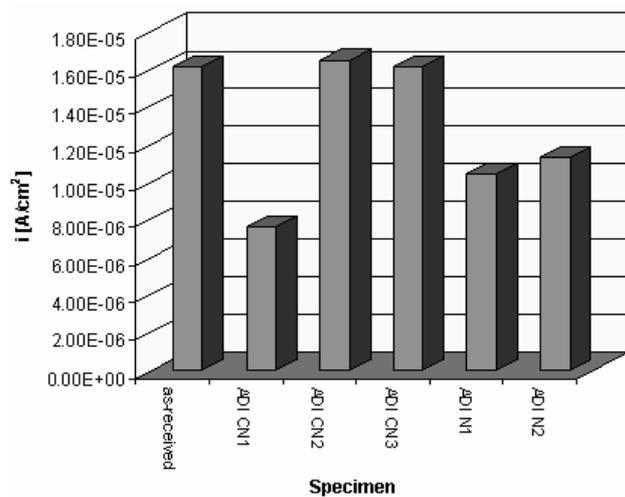


Fig. 9. Values of current density at potential of +30 mV away from stationary potential, for each of tested specimens

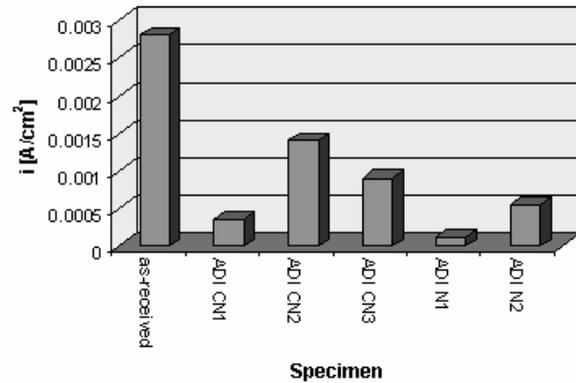


Fig. 10. Values of current density at potential of -300 mV (about +350 mV from stationary potential), for each of tested specimens.

Recorded anodic polarization curves allowed to range tested specimens in respect of susceptibility to corrosion. The modification of surface has most favourable effect on materials signed by numbers: ADI CN1 and ADI N1. The second group of materials is created by specimens, which resistance is decided by corrosion products formed in the consequence of dissolution: ADI N2. The weakest group, in respect of improvement of resistance through modification of surface, is created by remaining specimens, those are: ADI CN2, ADI CN3.

4. Conclusions

The results of performed research show that thermo-chemical treatment have an influence on structure of surface layer in austempered ductile iron precision castings, thus on their properties. The most important conclusions, coming from performed research are following:

1. The carbonitriding increased wear resistance in all tested specimens.
2. Selected proportions of carbonitriding atmosphere (NH₃ i CH₄) and the temperature of 860°C increased corrosion resistance in most effective way at conditions without additional treatments of activating the cast iron surface, that is decarburization, during shortest test time of 90 min.
3. The nitriding at the temperatures of 840°C and 860°C changed cast iron structure of surface layer; however, corrosion resistance has increased only in case of lower temperature.
4. The nitriding at the temperature of 840°C caused the most effective increase of wear resistance; however, in comparison to higher temperature of 860°C, castings had definitely lower strength at the same level of elongation.
5. Case of nitriding at the temperature of 840°C and carbonitriding at the temperature of 860°C proved similar characteristic of positive modification of precise casting surface layer in comparison with tempered ductile iron without surface treatment. However, for the sake of considerably higher resistance of castings treated thermally

at the temperature of 860°C, the carbonitriding will be more predestined to apply as the method of tempered ductile iron surface treatment.

Acknowledgements

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