



EFFECT OF AUSTEMPERING PARAMETERS ON THE MICROSTRUCTURE, MECHANICAL PROPERTIES, AND MACHINABILITY OF AUSTEMPERED DUCTILE IRON

By

Mostafa Ahmed Abdel Hamed Ahmed

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
In

<Metallurgical Engineering>

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT

2015

EFFECT OF AUSTEMPERING PARAMETERS ON THE
MICROSTRUCTURE, MECHANICAL PROPERTIES, AND
MACHINABILITY OF AUSTEMPERED DUCTILE IRON

By

Mostafa Ahmed Abdel Hamed Ahmed

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE (or DOCTOR OF PHILOSOPHY)
in
< Metallurgical Engineering >

Under the Supervision of

Prof. Dr. Abdel-Hamid Ahmed Hussein

Professor of Metallurgy
Mining, Petroleum, and Metallurgical
Department
Faculty of Engineering, Cairo University

Prof. Dr. Elsayed Mahmoud Elbanna

Professor of Metallurgy
Mining, Petroleum, and Metallurgical
Department
Faculty of Engineering, Some University

Prof. Dr. Mohammed Abdel-Wahab Waly

Professor of Metal Casting
Foundry Technology Laboratory
Central Metallurgical for R&D Institute (CMRDI)

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT

EFFECT OF AUSTEMPERING PARAMETERS ON THE
MICROSTRUCTURE, MECHANICAL PROPERTIES, AND
MACHINABILITY OF AUSTEMPERED DUCTILE IRON

By

Mostafa Ahmed Abdel Hamed Ahmed

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in

< Metallurgical Engineering >

Approved by the

Examining Committee:

Prof. Dr. Adel Abdel-Moniem Saleh Nofal, External Examiner
Central Metallurgical R&D Institute (CMRDI)

Prof. Dr: Mohammed Mamdouh Ibrahim, Internal Examiner

Prof. Dr: Abdel-Hamid Ahmed Hussien, Thesis Main Advisor

Prof. Dr: Elsayed Mahmoud Elbanna, Supervisor

Prof. Dr: Mohamed Abdel Wahab Waly, Supervisor

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT

2015

Engineer: Mostafa Ahmed Abdel Hamed Ahmed
Date of Birth: 27 / 4 / 1989
Nationality: Egyptian
E-mail: Mostafa.a-othman@hotmail.com
Phone: 01069829944
Address: 261 Elmalek Faisal Street, Giza
Registration Date: 1 / 10 / 2012
Awarding Date: / /
Degree: Master of Science
Department: Petroleum and Metallurgy



Supervisors: Prof. Dr. Abdel-Hamid Ahmed Hussien
Prof. Dr. Elsayed Mahmoud Elbanna
Prof. Dr. Mohamed Abdel Wahab Waly

Examiners: Prof. Dr. Adel Abdel Moniem Saleh Nofal
Central Metallurgical R&D Institute (CMRDI)

Prof. Dr. Mohamed Mamdouh Ibrahim
Prof. Dr. Abdel-Hamid Ahmed Hussien
Prof. Dr. Elsayed Mahmoud Elbanna

Prof. Dr. Mohamed Abdel Wahab Waly
Central Metallurgical R&D Institute (CMRDI)

Title of Thesis:

EFFECT OF AUSTEMPERING PARAMETERS ON THE MICROSTRUCTURE, MECHANICAL PROPERTIES, AND MACHINABILITY OF AUSTEMPERED DUCTILE IRON

Key Words: Ductile iron, austempering heat treatment, microstructure, mechanical properties, machinability, retained austenite, strain hardening.

Summary:

When ADI was introduced to be used for engineering applications, there were many problems experienced in trying to machine it.

The first point of this research aims at Producing ADI samples with different microstructures and hardness levels. Heat treatment procedures will cover novel techniques (two-step austempering). This study will cover the effect of alloying element as well as influences the austempering temperature on the mechanical properties and wear behaviour of austempered ductile iron. The second point designed to measure the machinability of different ADI grades and determine the optimum machinability parameters such as cutting speed, feed rate, and depth of cut using taguchi method. Finally, point three designed to study the phase transformation during machining different grades of ADI at different machining trials. Also perform a quantitative analysis of the change in the volume fraction of retained austenite and martensite produce during different machining trials by using XRD and heat tinting techniques.

Acknowledgments

I would like to express my deep regards and sincere gratitude to Prof. Dr. Abdel-Hamid A. Hussein, Faculty of Engineering, Cairo University for his care, kind supervision, encouragement, constant efforts, and valuable stimulating guidance and fruitful discussion throughout this study.

I offer my profuse thanks with humble reverence to Prof. Adel Nofal, Foundry Technology Laboratory, Central Metallurgical Research and Development Institute (CMRDI), for his invaluable guidance and support. He was a beacon light, whose constant efforts and encouragement proved to be a parallel stimulus in completing this research successfully.

I would like to thank Prof. Dr. El-Sayed M. El-Banna, Faculty of Engineering, Cairo University for his supervision and support.

I am grateful to my supervisor Prof. Dr. Mohammed Waly, Head of the Foundry Technology Laboratory, Central Metallurgical Research and Development Institute (CMRDI), for his support and co-operation in the hours of need and for his expert.

I would like to thank Prof. Dr. Ahmed Ramadan, Faculty of science, Helwan University for his kindly assistance in X-Ray diffraction analysis.

Last but not least, special thanks are due to the staff of Foundry Technology Laboratory, CMRDI and particularly metallographic, melting, workshop staff for their sincere help.

Table of Contents

Abstract	X
Chapter 1: Introduction.....	1
Chapter 2: Literature Review.....	3
2.1. Introduction.....	4
2.2. Background and History.....	4
2.3. Market Development.....	4
2.4. Properties and Selection of ADI.....	5
2.5. Austempering Heat Treatment for ADI Production.....	6
2.5.1 The Austenitization Stage	7
2.5.2 The Quenching Stage.....	8
2.5.3 The Austempering Stage.....	9
2.6. Novel Austempering Cycles of Ductile Iron.....	10
2.6.1 Two-step Austempering Process.....	10
2.6.2 Intercritical Austenitizing Process.....	13
2.7. Microstructure of Austempered Ductile Iron.....	15
2.8. The Importance of Retained Austenite in ADI.....	16
2.9. Machinability Challenges of ADI.....	17
2.9.1 Machinable ADI (MADI TM).....	17
2.9.2 Integration of Machining in the ADI production cycle.....	19
2.9.3 Machining practice of ADI.....	20

Chapter 3: Experimental Work..... 22

3.1.	Research Objectives.....	23
3.2.	Preparation of Castings and alloying.....	23
3.3.	Heat Treatment.....	24
3.4.	Characterization of ADI.....	26
3.4.1.	Chemical Composition Analysis.....	26
3.4.2.	Metallographic analysis.....	26
3.4.3.	XRD analysis.....	28
3.4.4.	Hardness Test.....	28
3.4.5.	Tensile Test.....	28
3.5.	Machinability Measurements.....	29
3.5.1.	Taguchi Method.....	29
3.5.2.	Cutting Forces Measurement.....	31
3.5.3.	Surface Roughness Measurement.....	31

Chapter 4: Results and Discussion..... 32

4.1.	Characterization of ADI.....	33
4.1.1.	Metallographic Analysis.....	33
4.1.2.	XRD Analysis	36
4.1.3.	Mechanical Properties	36
4.2.	Machinability Testing.....	39
4.2.1.	Machinability measurements	40
4.2.2.	XRD Analysis of machinability samples	42
4.2.3.	Machinability measurements	45
4.3.	Taguchi Experiments.....	56
4.3.1.	Signal to noise ratio analysis of cutting force and surface roughness data	56
4.3.2.	Analysis of Variance (ANOVA) calculation	56
4.3.3.	Confirmation Tests.....	59

Chapter 5: Conclusion.....	60
References	61
Appendix A: Taguchi Experimental Results.....	65
Appendix B: Analysis of Variance (ANOVA) tables.....	73
Appendix C: XRD Analysis.....	75

List of Tables

Table 2.1: Mechanical properties of ADI according to ASTM 897M 90.....	6
Table 2.2: Influence of two- step austempering on hardness and tensile properties....	12
Table 2.3: Austenite Volume Fraction and Carbon Content, as Measured by Using X-Ray Methods, for 120-Minute Austempering Heat Treatments at Various Temperatures.....	17
Table 2.4: Points of Improvement Offered by ADI with Dual Mixed Structure.....	19
Table 2.5: Mechanical properties of the material tempered and non-tempered samples	19
Table 3.1: Different heat treatment cycles applied in this research.....	26
Table 3.2: Chemical composition of the two grades of ductile irons.....	26
Table 3.3: Process parameters and their values at different levels.....	29
Table 3.4: Experimental design using L ₁₆ orthogonal array.....	30
Table 4.1: The Brinell Hardness Test Results.....	38
Table 4.2: The chemical composition of ductile iron.....	40
Table 4.3: The Brinell Hardness Test Results for machinability samples.....	41
Table 4.4: The optimum cutting parameters of different ADI-grades for cutting force data.....	61
Table 4.5: the optimum cutting parameters of different ADI-grades for surface roughness data.....	61

List of Figures

Figure 2.1: ADI – European Market Distribution – Years 2012-2013.....	5
Figure 2.2: Comparison of the cost (a) and weight (b) per unit of yield strength of different materials.....	6
Figure 2.3: A schematic diagram of; (a) ADI heat treatment cycle, and (b) the various phases of the ADI observed during ADI heat treatment cycle.....	7
Figure 2.4: Effect of austenitizing temperature on the mechanical properties of Austempered Ductile Iron casting.....	8
Figure 2.5: Schematic diagram shows the effect of quench severity on the austempering reaction.....	9
Figure 2.6: Effect of austempering temperatures on (a) yield strength, (b) elongation in ADI castings.....	10
Figure 2.7: Schematic diagram showing the effect of austempering time on the amount and stability of austenite and the hardness of ADI.....	11
Figure 2.8: Schematic of the conceived two-step austempering process: (1) A-B: heat up to the austenitizing temperature; (2) B-C: hold at the austenitizing temperature (2 h); (3) C-D: quench to austenitizing temperature; (4) D-E: hold at the austenitizing temperature (for a few minutes until nucleation is completed); (5)E-F: raise temperature immediately to second austempering; (6) F-G: hold at the second austempering temperature (usually 2 h); (7) G-H: air cool to room temperature.....	12
Figure 2.9: Stress-Strain curves of different ductile irons.....	13
Figure 2.10: The microstructure of IADI is well-formed graphite nodules in a matrix of ferrite and isolated island of austenite.....	14
Figure 2.11: The intercritical austempering treatment process.....	14
Figure 2.12: Microstructure of ductile iron austempered at different temperature, a) 371 °C b) 288 °C and c) 260 °C then 343 °C.....	16
Figure 2.13: A graph showing the change in the austenite volume fraction with the austempering conditions.....	17
Figure 2.14: Options for integrating casting, machining and heat treatment of ADI components.....	20

Figure 3.1: a) A typical vortex unit is made up for major cast iron and steel components: (A) refractory, (B) additives hopper, (C) interchangeable calibrated orifice and (D) shut off slide, b) Vortex unit available at CMRDI.....	25
Figure 3.2: Dimensions of Y-blocks and Ingots used in this research.....	25
Figure 3.3: Heat tinted grade ADI-900-375 microstructure - Kovacs method.....	27
Figure 3.4: Heat tinting procedure.....	28
Figure 3.5: Experimental set up using KISTLER dynamometer 9255B.....	31
Figure 4.1: Microstructure of ADI samples; a) ductile iron, b) austempering at 275°C, c) austempering at 375 °C, d) double step austempering 275 °C+375 °C, f) IADI (820-300 °C).....	36
Figure 4.2: XRD analysis for the three different grades of ADI and IADI.....	37
Figure 4.3: The ultimate tensile strength for both unalloyed and alloyed ADI austempered by single and double step process.....	39
Figure 4.4: The yield strength for both unalloyed and alloyed ADI austempered by single and double step process.....	39
Figure 4.5: The ductility for both unalloyed and alloyed ADI austempered by single and double step process.....	40
Figure 4.6: Microstructure of machinability measurements samples; a) ductile iron, b) austempering at 275°C, c) austempering at 375°C, d) double step austempering 275 °C+375 °C, f) austenitized at 820 °C then austempering at 300 °C.....	43
Figure 4.7: XRD analysis for the four different grades of ADI used in the machinability measurements tests.....	45
Figure 4.8: The variation of cutting force with the depth of cut under different cutting speeds for the investigated AI- grades.....	50
Figure 4.9: Effect of cutting speed and depth of cut on the cutting force for different ADI-grades.....	53
Figure 4.10: Variation of both martensite content and cutting force with the cutting depth for a) ADI-275, b) ADI-375, and c) ADI-2step at cutting speed of 55 m/min.....	57
Figure 4.11: ADI specimens etched by heat tinting technique, showing the variation of martensite, austenite and ferrite content before machining operation.....	58

Figure 4.12: ADI specimens etched by heat tinting technique, showing the variation of martensite, austenite and ferrite content with the change of cutting depth.....	60
Figure 4.13: Variation of cutting force with depth of cut, the cutting speed, and for different ADI-alloys, a) ADI-275, b) ADI-375, c) ADI-2step, and d) IADI....	62
Figure 4.14: Variation of surface roughness with cutting depth, feed rate and cutting speed for the ADI-alloys. A) ADI-275, b) ADI-2step, and c) IADI.....	63

Nomenclature

γ_{HC}	Rich carbon austenite
V_{γ}	Volume fraction of austenite
OA	Orthogonal array
S/N	Signal to noise ratio
DOF	Degree of Freedom
ANOVA	Analysis of Variance
V_c	Cutting speed, m/min
f	Feed rate, mm/rev
a_c	Depth of cut, mm

Abstract

The austempering process of the ductile irons was able to generate materials with unique combination of strength, abrasion resistance, as well as toughness. These unique properties make ADI finding a new applications in many engineering fields like; automotive, agricultural, earthmoving, mining, construction as well as military applications.

When ADI was introduced to be used for engineering applications, there were many problems experienced in trying to machine it. The hardest grades of ADI give a hardness of ~50 HRC which would represent a serious problem for any high volume machining operation. The reduced hardness of the lower grades of ADI (300-350 BHN) does not mean better machinability. When subjecting those grades to considerable strains, associated with machining, the high content of retained austenite, which may reach up to 40-43% would transform by TRIP effect to martensite leading to drastic reduction in machinability compared to steels of equivalent hardness.

The first point of this research aims at Producing ADI samples with different microstructures and hardness levels. Various structures will be produced by changing the austempering temperature and using the alloying elements such as Cu and Mo. Heat treatment procedures will cover novel techniques (two-step austempering). This study will cover the effect of alloying element as well as influences the austempering temperature on the mechanical properties and wear behaviour of austempered ductile iron. The second point designed to measure the machinability of different ADI grades and determine the optimum machinability parameters such as cutting speed, feed rate, and depth of cut using taguchi method. Finally, point three designed to study the phase transformation during machining different grades of ADI at different machining trials. Also perform a quantitative analysis of the change in the volume fraction of retained austenite and martensite produce during different machining trials by using XRD and heat tinting techniques and we will try to link this quantitative analysis with the results obtained from machinability measurements.

The machinability experiments results show that depth of cut is the main machining parameters which control and determine the measured cutting force values. The highest cutting force values recorded at high depth of cut at 1.5:2 mm. The optimum machining parameters obtained at depth of cut (0.5:1 mm)

The XRD analysis shows that ADI -375 has a 32.5% retained austenite which transforms to martensite during machining, so this grade shows the same cutting force values of the harder grade of ADI (275). In two-step ADI grade, the cutting forces F_c values are continuously increased when depth of cut changed from 0.5 mm to 1.5 mm but a great decrease with 17.6 % is observed when depth of cut increased from 1.5 mm to 2 mm.

The machinability results proved that IADI has the lowest cutting force values which are decreased by ~ 30:35 % at lower depth (0.5-1 mm) of cut and decreased by ~ 15:20% at higher depth of cut (1.5-2 mm) than other ADI grades introduced in this research.

Chapter 1 :Introduction

In the last 20 years, the dream material, the austempered ductile iron (ADI) with its unique combination of strength, abrasion resistance, toughness, fatigue resistance and machinability has been finding new applications in automotive, agricultural, earthmoving, mining, construction as well as military applications.

Since the announcement of the first production of ADI in the last century, the research started in many industrialized countries, which provided sound products of this dream material in many region in the world during the 1990's and beyond. By the turn of the century, the ADI market had begun to rapidly accelerate from a modest beginning in the early 1970's to an estimated worldwide production level of 125,000 tons in 2005. This growth is expected to continue with the annual world production of ADI reaching 500,000 tons by the end of this year 2013.

The attractive properties of ADI return to its distinct and unique microstructure, which consists of acicular ferrite with enriched carbon austenite (ausferrite). The austempering transformation in ADI can be performed in two-stage reaction:

Stage I Reaction: $\gamma_c \rightarrow \alpha + \gamma_{HC}$ (toughening)

Stage II Reaction: $\gamma_{HC} \rightarrow \alpha + \varepsilon$ - carbides (embitterment)

When ADI was introduced to be used for engineering applications, there were many problems experienced in trying to machine it. The hardest grades of ADI give a hardness of ~50 HRC which would represent a serious problem for any high volume machining operation. The reduced hardness of the lower grades of ADI (300-350 BHN) does not mean better machinability. When subjecting those grades to considerable strains, associated with machining, the high content of retained austenite, which may reach up to 40-43% would transform by TRIP effect to martensite leading to drastic reduction in machinability compared to steels of equivalent hardness.

Therefore, this research objective has been divided into 3 points. The first point aims at Producing ADI samples with different microstructures and hardness levels. Various structures will be produced by changing the austempering temperature and using the alloying elements such as Cu and Mo. Heat treatment procedures will cover novel techniques (two-step austempering). This study will cover the effect of alloying element as well as influences the austempering temperature on the mechanical properties and wear behaviour of austempered ductile iron. The second point designed to measure the machinability of different ADI grades and determine the optimum machinability parameters such as cutting speed, feed rate, and depth of cut using taguchi method. Finally, point three designed to study the phase transformation during machining different grades of ADI at different machining trials. Also perform a quantitative analysis of the change in the volume fraction of retained austenite and martensite produce during different machining trials by using XRD and heat tinting techniques and we will try to link this quantitative analysis with the results obtained from machinability measurements.