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## Effects of refrigeration and cryogenic treatments on austempered ductile iron

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EFFECTS OF REFRIGERATION AND CRYOGENIC TREATMENTS ON  
AUSTEMPERED DUCTILE IRON

An Abstract of a Thesis  
Submitted  
In Partial Fulfillment  
Of the Requirements for the Degree  
Master of Arts

Gerard R. Thiel  
University of Northern Iowa  
August 1996

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## ABSTRACT

Low temperature treatment, as a method of improving mechanical properties of metals, has been studied and used for well over 60 years. Until recently these treatments have been limited to hardness and wear improvements of high alloy irons and steels. The purpose of this research is to determine the effects of refrigeration and cryogenic treatments on the mechanical properties, including hardness and tensile strength of austempered ductile iron.

This research included the production of a high strength ductile iron of low alloy content capable of being used in either the as cast, conventionally heat treated or austempered condition. Refrigeration and cryogenic treatments were applied to the material at different points within the production cycles to determine the most effective order of treatment. After production of the base material at the university, representative specimens were treated by a series of sub-zero temperatures and austempering cycles at commercial treatment facilities. These specimens were then returned to the university and were subjected to a series of mechanical tests that included tensile and hardness property testing.

After analysis of the data, it was determined that the greatest effects on the iron were in the increase in hardness and tensile strength of samples that had been cryogenically treated and after undergoing a commercial austempering cycle. Tensile and hardness properties of the material were increased approximately 16% over the non-treated samples. The study concluded that cryogenic treatment of austempered ductile

iron is possible, and that it may have benefits over conventional hardening methods by preserving the surface quality of machined components.

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A Thesis

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August 1996

This Study By: Gerard R. Thiel

Entitled: EFFECTS OF REFRIGERATION AND CRYOGENIC TREATMENTS ON  
AUSTEMPERED DUCTILE IRON

has been approved as meeting the thesis requirement for the Degree of Masters of Arts in  
Industrial Technology.

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## CHAPTER 1

### INTRODUCTION

More than 60 years ago, extensive research was conducted on austenitic iron alloys. These experiments showed major improvements in the mechanical properties when exposed to cryogenic temperatures after a conventional heat treatment (Kies & Schelleng, 1968). Since this time, there has been a continued debate over the usefulness of cryogenic treatments stemming from the lack of visible microscopic changes to the treated metal. Compiled data on various wear properties and case studies from actual industry applications show promising results of cryogenic treatments on ferrous and non-ferrous alloys. Because of this uncertainty about cold treatments, only limited research has been done on selected alloys. The proposed research is an effort to continue and expand the current knowledge base of the effect of cryogenic treatment on austempered ductile irons (ADI).

Research data of sub-zero treatments investigated in the early 1930s show great promise in improving mechanical properties when applied to materials currently used in industry. Cryogenic treatments have been used to improve physical attributes such as hardness, tensile strength and wear properties while maintaining corrosion resistance. While mechanical property improvements can be obtained with more conventional thermal heat treatments, they come with an associated increase in hardness and at the expense of machinability of the treated components. It has been shown that cryogenic

treatments and subsequent conventional heat treatments can improve the tensile properties and wear resistance of the material without an increase in hardness by the formation of submicroscopic carbides Baron (1982). Thermal heat treatments conventionally used to harden metals affect the corrosion resistance of the material and often degrade machined surfaces requiring post processing of the component parts.

ADI is an alloyed and heat treated ductile cast iron with a similar composition to that of conventional ductile iron. As in most cast irons, the material is made up of graphite in spherical shape in a variable matrix which controls its mechanical properties. Alloying elements such as nickel, copper and molybdenum are added to the ADI to enhance the matrix structure and improve the material's responsiveness to heat treat. The properties of the material are obtained by austempering (a specialized isothermal heat treatment) to impart a high degree of strength, toughness and wear resistance to the cast parts. As reported by Berg (1985), although commercialized in the 1970s, ADI has failed to make a significant impact in terms of usage. This material was originally developed to replace steel castings in many applications because of the lower production costs. Unfortunately, the mechanical properties of the material fall short when compared to steel exhibiting a 20% lower elastic modulus and in general lower impact strength (Harding, 1985).



### Statement of the Problem

Based on the previous discussion, the problem can be defined as the lack of published data on cryogenic treatment of Austempered Ductile Irons. Because of this lack of data and limited information concerning the effect of cryogenic treatment, it was felt necessary to investigate the effects of sub-zero temperature treatments on materials not previously investigated.

The objective of this research was to determine the influence of cryogenic treatment on the tensile strength and hardness of grade 1, austempered ductile iron. The study included the comparison of the tensile properties of austempered ductile iron cryogenically treated at two sub-zero temperatures both before and after austempering. To achieve the goals of the study, tensile specimens were produced from a ductile iron capable of being austempered to the ASTM grade 1 strength levels. The samples received combinations of cryogenic, conventional heat treatments and austempering treatments in an effort to determine treatment levels and treatment sequences that maximize mechanical properties. The results were analyzed, discussed, and compared in the light of previous studies presented and conclusions drawn.

## CHAPTER 2

### REVIEW OF THE LITERATURE

#### Cold, Cryogenic or Refrigeration Treatments

Classifications of sub-zero temperature treatments of metals have traditionally been linked with the treatment method used to lower the temperature of the metals to be treated. A typical home-type freezer can lower the temperature of the work to approximately 0°F(-18°C) and provide phase transformation from austenite to martensite. Dry ice with a surface temperature of -93°C (-109°F) can be placed on top of the work in a closed insulated container to provide cold treating, although the typical chamber temperature is only about -73°C (-75°F). Mechanical refrigeration units with circulating air are commercially available and can lower the temperature of the work to -87°C (-125°F). Treatments to temperatures at or below -84°C (-120°F) are commonly considered cold or refrigeration treatment. Liquid nitrogen, with a temperature of -195°C (-320°F) is also used to lower the metal's temperature further. This can be accomplished by submersing the work into the liquid nitrogen, a process commonly referred to as the wet method or shock cooling, or by cooling the parts slowly transferring heat from the parts to the liquid nitrogen without contact. The latter is the accepted method and has been more consistent in improving the properties of the treated metals. The consistency is a result of a computer-controlled process of the heat exchange from the liquid nitrogen to the work pieces providing for a slow cool down and heating. Treatment of materials at or

below  $-143^{\circ}\text{C}$  ( $-315^{\circ}\text{F}$ ) are generally considered Cryogenic. Although liquid helium is more expensive, it can replace liquid nitrogen to lower the temperature even further to  $-273^{\circ}\text{C}$  ( $-460^{\circ}\text{F}$ ). Treatment at  $-273^{\circ}\text{C}$  is considered deep cold (Calson, 1991).

According to the literature, cryogenic treatments act on ferrous metals by fully transforming any retained austenite in the structure. The phase change that takes place in ferrous metals as result of cold treatment is from austenite to martensite. Conventional heat treatments can only transform about 85% of the austenite to martensite. Austenite is a composition of the pure iron (Fe) and carbon and is referred to as gamma iron. Its face centered cubic crystal structure can dissolve up to 2.06% carbon at  $1132^{\circ}\text{C}$  ( $2096^{\circ}\text{F}$ ). Carbon stabilizes austenite and increases the range of its formation. Retained austenite in metals provides toughness and ductility while maintaining the ability to transform to martensite upon plastic deformation. This makes austenitic alloys wear resistant to most environments where surface deformation of the metal takes place.

Martensite is a metastable structure that has the same composition as the austenite from which it forms. Its body centered tetragonal (bct) crystal structure is a solution of carbon in iron (Fe). As a result of transformation without compositional change, it is formed without diffusion and at low temperatures. The most remarkable property of martensite is its potential of high hardness which depends on the carbon content and on eutectoid composition. Martensite has a Rockwell C hardness of 65, which may be described as “glass hard.” This hardness is an inherent property of martensite in part because of very severe distortions that accompany its formation. The distortions arise

because martensite has a larger specific volume than the austenite from which it forms (Brick, Gorden, & Phillips, 1965). Figure 1 Illustrates the Bain model of martensite transformation from face centered cubic to body centered tetragonal.

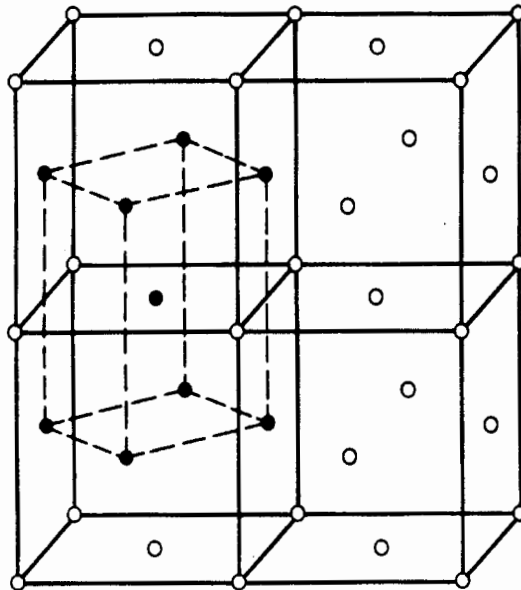


Figure 1. Bain model of transformation of martensite from austenite (Brick et al., 1965).

The resultant structures after cryogenic treatment are fully martensitic and exhibit greater wear resistance and tensile strength. The cold treatment of metals may be beneficial, in the case of metastable martensite and; increase strength; and provide greater dimensional or microstructural stability, improved wear resistance, and relief of residual

stresses. During cold treating, transformation of the retained austenite to martensite will occur when a minimum of  $-120^{\circ}\text{F}$  ( $-84^{\circ}\text{C}$ ) is obtained. Additional time or chilling will not cause reversal or detrimental effects (Calson, 1991 ).

Although chemical composition, phases and resultant structures control the mechanical properties of the material, they may not occur readily from the manufacture of metals. Often metals will require heat treatment to impart structures containing specific phases that control desired mechanical properties. Soaking time at the austenitizing temperature and the cooling rate at which the cooling takes place are both critical in determining the resultant structure of the metal. Typically this phase transformation takes place at well above room temperature because of cooling from above the  $A_{c3}$  (temperature where austenite is stable) through a temperature zone where martensite is formed ( $M_s$ -  $M_f$ , martensite start through martensite finish). The resultant structure is one of primarily martensite but retaining some amount of the austenite that is stable at room temperature. By causing complete transformation from austenite, in-service transformation is eliminated, which may cause cracking and dimensional changes that can cause failure (Calson, 1991). A beneficial aspect of retained austenite is that it is thought to prevent crack propagation by either changing the growth direction of an advancing crack or absorbing great amounts of energy, (Meng, Tagashira, Azuma, & Sohma, 1994). This retained austenite is also beneficial to toughness and ductility of the material but degrades the hardness, wear resistance and tensile strength.

### Historical Background of Sub-zero Temperature Treatments

As far back as 1932, Wasserman (cited in Kies & Schelleng, 1968) conducted experiments at sub-zero temperatures to improve the properties of ferrous alloys by cold treating and re-austenitizing the material at a low temperature. It was reported that iron-nickel alloys could be strengthened by transforming the retained austenite to martensite with the use of cryogenic treatment and then re-austenitizing the material at low temperature. In 1962 Krauss and Cohen quantified property improvements and reported that the strength of an iron-nickel alloy could be increased as much as 2.5 times the original tensile strength by a similar refrigeration / re-austenization cycle.

Further research conducted by Gorbach and Malyshev in 1964 (cited in Kies & Schelleng, 1968 ) showed that the strengthening in high carbon austenite is dependent upon the amount of martensite formed. To that point in time only one researcher, Detrich (cited in Kies & Schelleng, 1968), had studied the effects of refrigeration treatments on a ductile grade of austenitic cast iron. The primary results were concerned with the magnetic properties as a result of phase changes in the material and not with the strengthening of the alloy.

Kies and Schelleng (1968) published their findings relating to the strengthening of Ni-Resists due to refrigeration and re-austenization treatment. These studies addressed several types of austenitic cast irons including Ni-Resist and Ductile Ni-Resist alloys with nickel contents ranging from 15 to 21%, Chromium 0 - 2%, Manganese 0.3 - 1.0%, and Silicon 1.7 - 3.7%. Treatment temperatures of  $-93^{\circ}\text{C}$  ( $-110^{\circ}\text{F}$ ) and  $-210^{\circ}\text{C}$  ( $-320^{\circ}\text{F}$ )

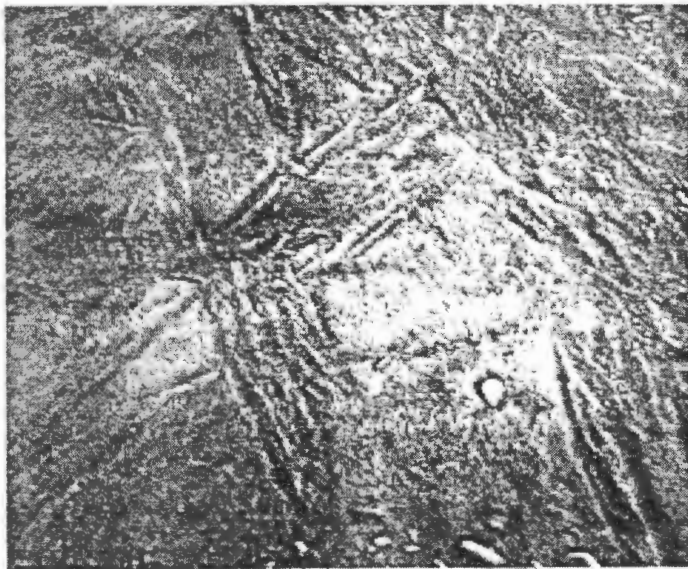
were used. The results concluded that significant increases in yield strength may be achieved by a refrigeration and re-austenization process.

Grant and Morrison (1972) discussed the refrigeration of grade S-Ni Cr 20 2 austenitic iron at the liquid nitrogen temperature  $-196^{\circ}\text{C}$  ( $-312^{\circ}\text{F}$ ) to form martensite, and then the re-austenitization at about  $650\text{-}760^{\circ}\text{C}$  ( $1200\text{-}1400^{\circ}\text{F}$ ). This resulted in increasing the tensile strength from 386 MPa (56,000 psi) to more than 496 MPa (72,000 psi) and proof strength from 193 MPa (28,000 psi) to more than 331 MPa (48,000 psi). Substantial increases in the mechanical properties of both the flake and spheroidal graphite austenitic irons by the less severe temperatures of  $-78^{\circ}\text{C}$  ( $-108^{\circ}\text{F}$ ) can be obtained by adjusting the composition, though permanent property improvements are limited to the increase in martensite content.

### Mechanics of Cryogenic Treatments

Kies and Schelleng (1968) had observed that austenite was transformed to martensite upon refrigeration. This martensite consisted of fine plates of alternating ferrite and iron carbide ( $\text{Fe}_3\text{C}$ ), and resulted in an increase in hardness upon refrigeration. After reheating and re-austenitization of the alloys that contained 1% chromium, fine carbide particles were observed which were absent in the chromium free irons. These fine carbide particles occupied the locations where the martensite plates formerly existed and were hypothesized to contribute to the strengthening of the alloy. Detrich (1964) had observed the same effect in a high-manganese chromium free alloy. The resultant

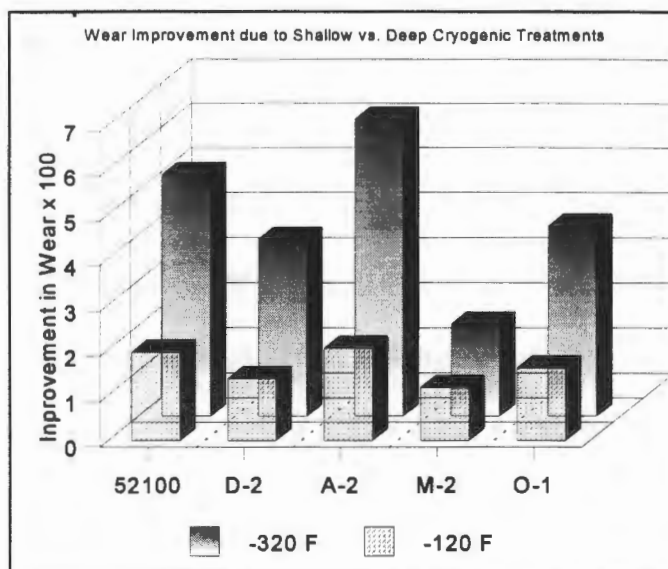
structures were entirely austenitic which accounts for similar hardness values as in the conventionally treated unrefrigerated alloys. Recent research work conducted by Meng et al. (1994) identifies the carbides as Eta-carbide precipitates ( $\eta$ ), which are only formed when the treatment temperatures are below  $-93^{\circ}\text{C}$  ( $-110^{\circ}\text{F}$ ). Cryogenic treatments were also effective at reducing the amount of commonly formed detrimental  $\epsilon$ -carbides and developing a finer martensitic structure as shown in Figure 2.



**Figure 2.** Carbide precipitate in prior martensite areas 2% Nital Etch, 10,000X (Kies & Schelleng, 1968).

While cold or refrigeration treatments improve wear resistance up to 100%, results of cryogenic treatments have shown improvements in excess of 500% (Baron, 1974). Results of refrigeration and cryogenic treatments for various alloys are shown in Figure 3.





**Figure 3.** Wear improvements at cold and cryogenic temperatures (Baron, 1974).

A factor in improving the wear resistance through sub-zero treatments is the elimination of retained austenite and the formation of a homogenous martensitic structure. It has also been suggested that the precipitation of the fine  $\eta$ -carbides rather than removal of austenite from the matrix is responsible for the improvement of wear resistance, (Meng et al., 1994). This formation of  $\eta$ -carbides occur with little change in the hardness value of the material.

Consistent throughout the literature is the principle that materials treated by refrigeration or cryogenics must be thermally treated before any sub-zero treatment is used. Ni-Resists require an annealing treatment before refrigeration, if the full potential

of the strengthening treatment is to be realized. The alloys must be annealed at 1700°F to achieve the maximum benefit of the strengthening treatment. Marshall (1987) believed that the inconsistency in the pre-heat treatment may be a reason for the lack of consistency of property improvements in previous studies. Annealing the Ni-Resist alloys serves to deplete the austenite matrix of carbon by raising the temperature of the metal into the austenitizing temperature and holding for sufficient time to allow for diffusion of the carbon. This depletion of carbon raises the  $M_s$  temperature of the alloy causing greater quantities of martensite to form at a given refrigeration temperature yielding higher hardness, yield strength and tensile strength. Addition of silicon reduces the solubility of carbon in the austenite that in turn, increase how much martensite is formed. Although increasing manganese content lowers the  $M_s$  temperature, it also retards softening upon reheating by retarding the transformation of austenite. Manganese was found beneficial in raising the yield strengths although the hardnesses of the refrigerated samples were lower. Manganese was also beneficial to ductility (Kies & Schelleng, 1968).

In 1968, Trucks (cited in Sweeney, 1988) reported that cryogenic treatment removes the kinetic energy of atoms. Normal attraction between atoms makes them attract, but their energy is quelled by low temperature cooling. Thus final treatment below -300°F in a dry atmosphere transforms retained austenite into the harder, more desirable, martensite. During this transformation, smaller carbide particles are released and distributed evenly through the mass of the material. These smaller spherical particles

co-exist in addition to larger carbon carbides present before the cryogenic treatment.

These smaller carbon carbide particles help to support the martensite matrix. In an application such as cutting tools, this effect reduces the heat build up on the cutting edge, and consequently increases the wear resistance and “red” hardness of the tool.

### Benefits of Hardening by Refrigeration and Cryogenic Treatment

The conventional practice of hardening ferrous materials involves (a) the heating to austenitizing temperatures and (b) the cooling at a controlled rate to develop specific phases in the materials structure. Under normal conditions, temperature gradients that are formed on the cooling of the material produce non-uniform dimensional and volumetric changes. When metals are rapidly cooled from high temperatures, compressive stresses develop in lower volume areas that cool first and tensile stresses develop in areas of greater volume areas that are the last to cool. Even in castings or machined parts of uniform thickness, the surface cools first and the core last. In such cases stresses develop because of the phase (volume) change between those layers that transform first and the center that transforms last. When both volume and phase changes occur in pieces of uneven cross section, normal contractions due to cooling are opposed by transformation expansion. The increase in volume of the outer martensite is somewhat counteracted by the initial contraction due to chilling. The residual stresses will remain until a means of relief is applied. This residual type of stress is frequently observed during the quenching

of hardenable alloys. The surface is transformed to martensite before the interior is (Calson, 1991).

### Austempering

Research shows that there are three key points in the austempering of ductile iron. These points are associated with the temperature and the holding time at temperature that the material experiences throughout the process, as shown in figure 4.

The first phase of heat treatment is the austenization of the material which is done at temperatures of approximately 1650°F (900°C) for a period of approximately two hours. The time and temperature are dependent on the thickness of the part to be treated and the chemical analysis of the material. As the austenitizing temperature is increased, the solubility of carbon in austenite is increased thereby raising the  $M_s$  temperature and increasing the amount of martensite that is formed on cooling. Thus, a high austenitizing temperature will yield a high carbon content in the resulting austenite. Ideally the austempering temperature should be at such a temperature to lower the  $M_s$  to a point below room temperature to prevent the formation of martensite. This time and temperature should allow for the generation of a uniform matrix carbon composition. At this point the major constituents are austenite and carbide ( $Fe_3C$ ).

The second phase is the quenching of the material to a point below the point where pearlite will form but above the point where martensite will form and holding to allow sufficient time for phase change to take place. This is accomplished by quenching

the work into a molten salt bath and holding at this temperature. The material is held at this temperature for a sufficient period in order for bainite or more correctly ausferrite to form.

The third phase of the heat treatment of austempered ductile iron is the cooling from the austenitizing temperature to room temperature. This phase is only important if a significant amount of austenite transforms to martensite (Rouns & Rundman, 1987).

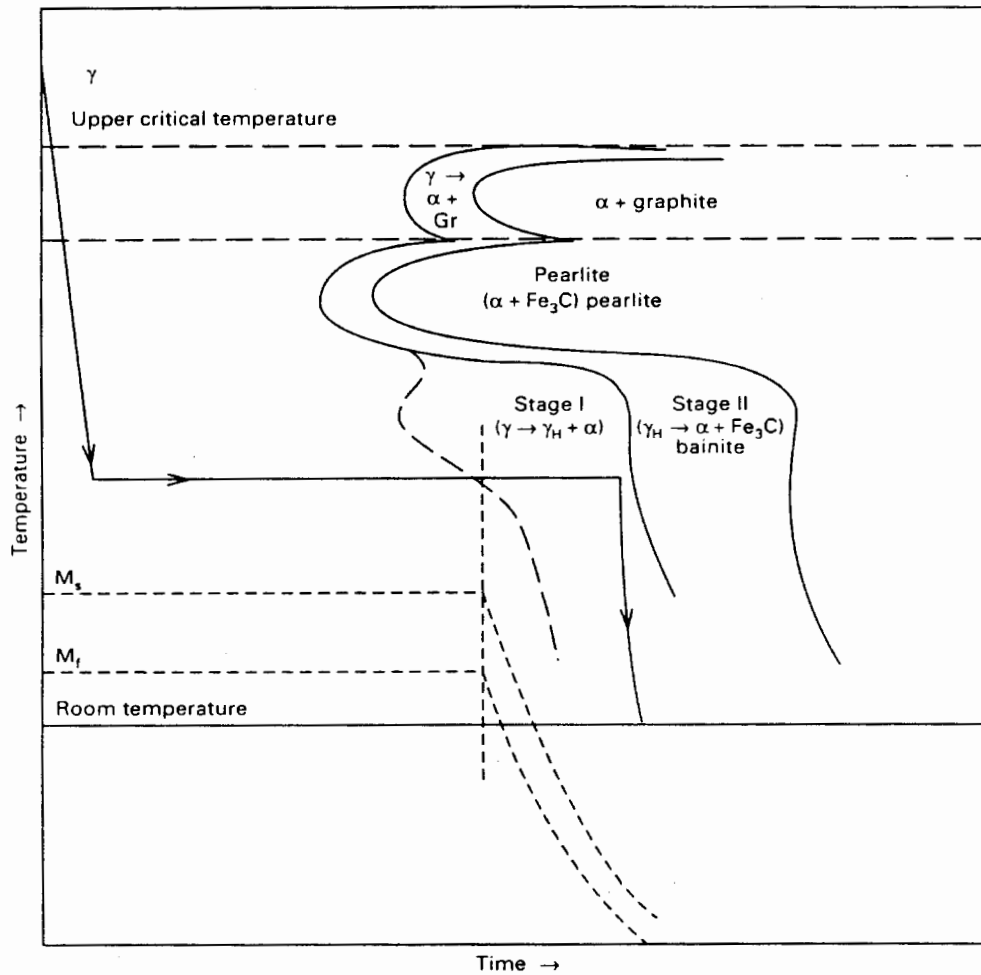


Figure 4. TT diagram of a processing sequence for austempering. American Society for Metals, Chandler, H., (1995) Heat Treating Ductile Iron.

## CHAPTER 3

### METHODOLOGY AND DISCUSSION

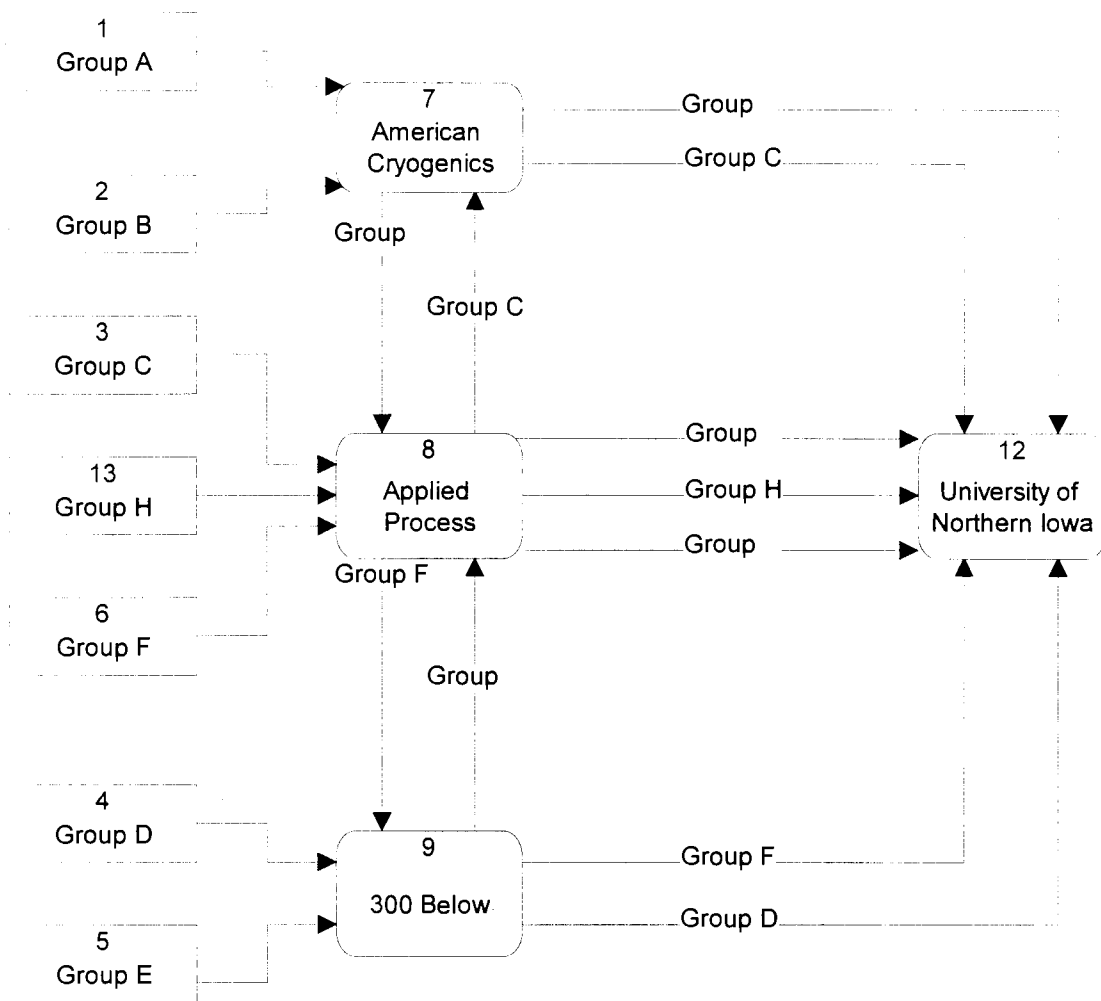
#### Introduction

In the present study, basic metallographic evaluation tools were used that would in broader scopes be inadequate. Because of the limitations in metallographic evaluation equipment, results of the research were limited to the dependent variable of mechanical property changes only. It was also not the intent of the research to explain the physical chemistry or mechanism of property improvement, but rather the ability of the independent variable of cryogenic treatment to modify the properties of austempered ductile iron in a beneficial manner.

Certified testing sources outside the university were used for both the processing and evaluation of the test samples. Chemical analysis verification for the purpose of determining specific austempering temperatures of the test heats were conducted under ISO 9002 certification at the John Deere Waterloo Foundry. The calculation of the austempering processing temperatures were completed with the use of a proprietary software program developed by Applied Process Incorporated. The process flow for the test samples is outlined in Figure 5.

#### Production of the Test Material

All of the samples were produced from a ductile iron containing approximately 1.5% Ni. And .30% Mo. This chemistry was used by Janowak and Gundlach, (1983) in a



**Figure 5.** Process flow of test samples

study to develop a ductile iron for commercial austempering. All samples were poured according to the American Society for Testing Materials (ASTM) 1" Y-block standard and were allowed to cool to under 162°C (350°F) in the molds before removal. All sample groups were austenitized for two hours at 927°C (1700°F) and air cooled to room



temperature to obtain a matrix primarily of pearlite, ferrite and small amounts of retained austenite, in order to maximize the effects of the cryogenic treatment.

### Charge Materials

Charge materials for the experimental heats consisted of commercially available high quality materials including Sorel pig iron, low carbon steel punchings, 75% FeSi, MgFeSi Molybdenum oxide briquettes and nickel shot as listed in Table 1. All efforts were made to keep tramp elements (elements not accounted for in the original experiments) to a minimum. All metallic materials were clean and free of rust or other contaminants to reduce their possible detrimental effects.

All of the materials listed were donated by local industry or their respective suppliers. Charge materials were placed in the furnace in such a manner to maximize the charge density and to allow for thermal expansion to prevent bridging. The order of placement of the charge materials in the furnace was such that melting losses were minimized.

### Melting Equipment

The experimental heats were melted at the University of Northern Iowa in a 300 lbs. capacity box furnace with a 180 KW, Power supply. This unit has the capability of melting and superheating 300 pounds of ferrous metal in approximately 60 minutes. The furnace uses a magnesia-alumina crucible as a working lining with a dry vibratable magnesia refractory to hold the crucible and provide a safety lining. The crucible is further held in place by a top cap consisting of 75% alumina plastic refractory.

Table 1

Charge Materials used in the Experimental Heats

Description of charge material	Weight used lbs.	Percent of charge
Sorel F-1 pig iron	55	21.5
Low carbon steel punchings	100	39.0
Carbon raiser	4.25	1.6
Molybdenum oxide briquettes	1.3	.5
Ferro silicon 75%	2.76	.1
Nickel	3.75	1.5
Ductile Iron returns	89	35.0

Melting Procedure

After charging, the furnace was powered to 10% of the rated electrical capacity for 10 minutes to prevent thermal shock to the furnace lining. Power to the furnace was then raised to 95% of rated electrical capacity until sufficient liquid formed and space was available for additional charge material. Chemical analysis of the base iron was determined after meltdown and superheat to 1440°C (2650°F) by use of an Electronite Data Cast 2000 thermal analysis unit.

### Magnesium Treatment

After the base iron was raised to a temperature of 1495°C (2750°F) and the correct base iron chemistry verified, the metal was tapped into a flow-through treatment chamber for magnesium treatment. Immediately preceding tapping of the iron, a 3.25% magnesium ferro silicon in the quantity of 2% of the charge weight was placed into the flow-through chamber along with 75% ferro silicon for post inoculation.

### Austempering and Cryogenic Treatments

All of the samples were austempered at Applied Process Inc., Detroit MI., a commercial austempering facility. All of the samples were austenitized for 100 minutes at 878°C (1638°F) in a carburizing atmosphere and quenched in a molten salt bath at a temperature of 362°C (710°F) and held at this temperature for a period of 123 minutes. The castings were removed from the salt bath and washed with water to remove any adhering salt from the austempering process and cooling the samples to room temperature.

### Treatment Groups

In order to isolate the effects of the cryogenic treatment, the samples were processed in Seven groups. All of the samples in the seven groups received a normalizing heat treatment at 917°C (1700°F) for a period of two hours and then were cooled to room temperature in still air.

Group G--Normalize only. Group G castings were prepared as mentioned above and served as the control group for the study.

Group H--Austempering only, no cryogenic treatment. Samples were austenitized as shown in figure 6 for 100 minutes at 878°C (1638°F) in a carburizing atmosphere, quenched in a molten salt bath at a temperature of 362°C (710°F) and held at this temperature for a period of 123 minutes. The castings were removed from the salt bath and washed with water to remove any adhering salt from the austempering process and then cooled to room temperature. These castings did not receive any form of cryogenic treatment..

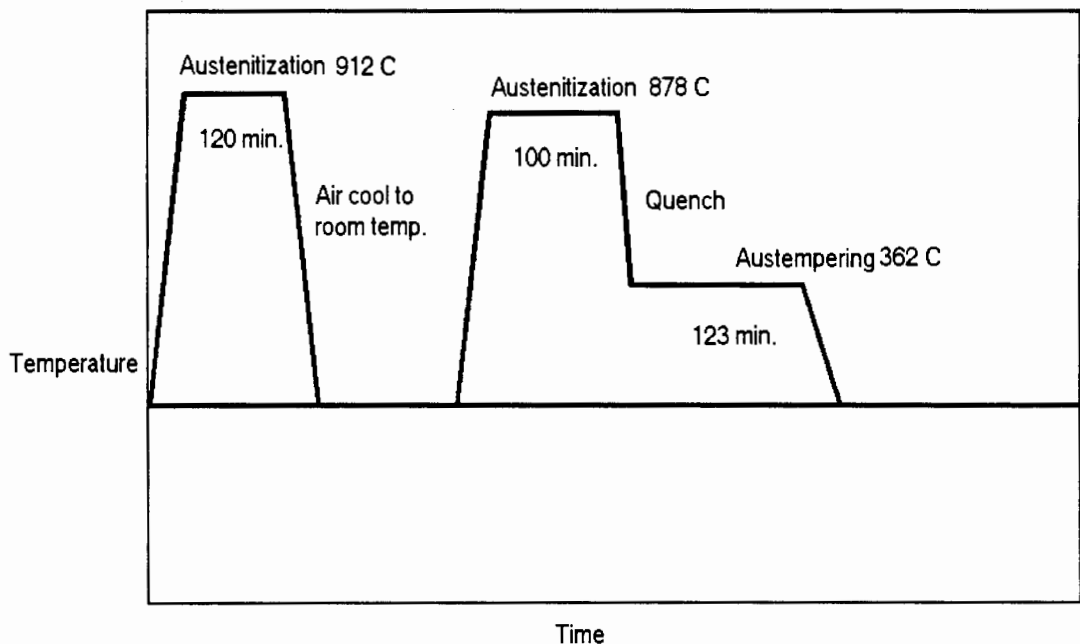


Figure 6. Group H Treatment cycle.

Groups A and D--Cryogenic Treatment without Austempering. The Group A samples were refrigerated by cooling as outlined in figure 7 at a rate of  $40^{\circ}\text{C}$  per hour to a temperature of  $-93^{\circ}\text{C}$  ( $-110^{\circ}\text{F}$ ) and then holding at that temperature for a period of two hours. The samples were then raised to room temperature from the treatment temperature at a rate of  $40^{\circ}\text{C}$  per hour. Group D samples were cryogenically treated by cooling at a rate of  $40^{\circ}\text{C}$  per hour to a temperature of  $-210^{\circ}\text{C}$  ( $-320^{\circ}\text{F}$ ) and held for a period of 20 hours. The samples were heated to room temperature at a rate of  $40^{\circ}\text{C}$  per hour. These castings did not receive any thermal treatment after exposure to sub-zero temperatures.

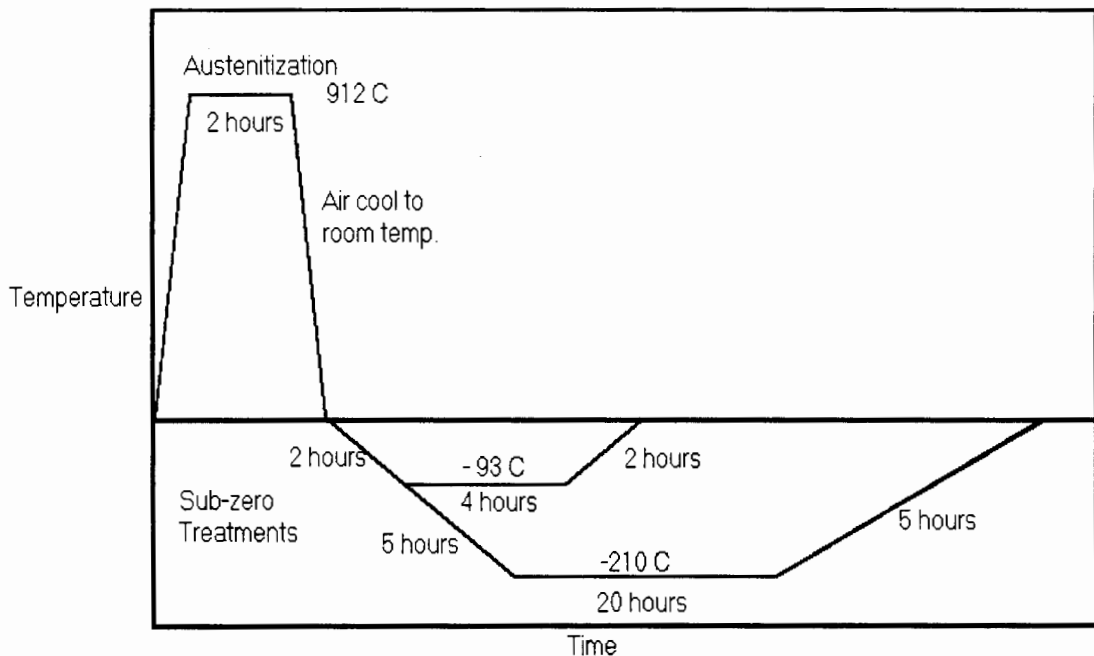
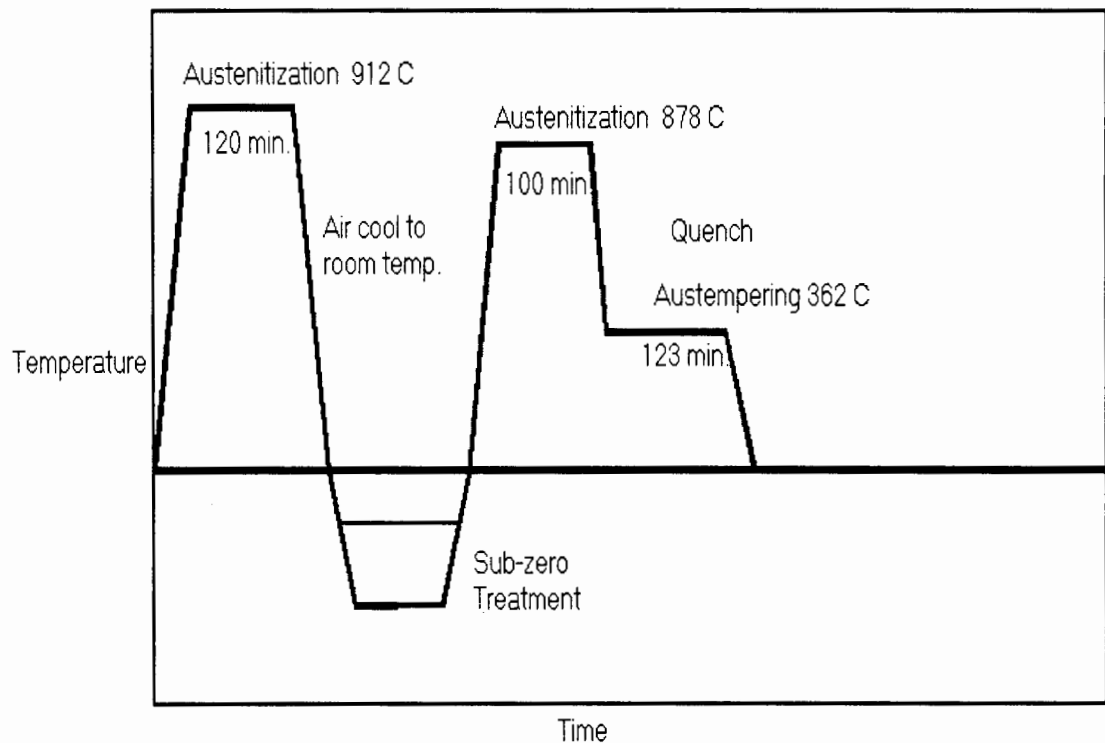


Figure 7. Schematic diagram of cryogenic treatment.

Groups B and E--Cryogenic treatment before austempering. The Group B samples were refrigerated by cooling at a rate of 40°C per hour to a temperature of -93°C (-110°F) and holding for a period of two hours. The samples were then raised to room temperature from the treatment temperature at a rate of 40°C per hour. Group E samples were cryogenically treated at -210°C by cooling at a rate of 40°C per hour and holding for a period of 20 hours. The temperature of group E samples was then raised to room temperature at a rate of 40°C per hour. Both groups were austenitized as shown in Figure 8 for 100 minutes at 878°C (1638°F) in a carburizing atmosphere and quenched in a molten salt bath at a temperature of 362°C (710°F) and held at this temperature for a period of 123 minutes. The castings were removed from the salt bath and washed with water to remove any adhering salt from the austempering process and then cooled to room temperature.

Groups C and F--Cryogenic treatment after austempering. Groups C and F were austenitized per the cycle shown in Figure 9 for 100 minutes at 878°C (1638°F) in a carburizing atmosphere and quenched in a molten salt bath at a temperature of 362°C (710°F) and held at this temperature for a period of 123 minutes. The castings were removed from the salt bath and washed with water to remove any adhering salt from the austempering process, cooling the samples to room temperature. The Group C samples were refrigerated by cooling at a rate of 40°C per hour to a temperature of -93°C (-110°F) and holding for a period of two hours. The samples were then raised to room temperature from the treatment temperature at a rate of 40°C per hour. Group F samples

were cryogenically treated at  $-210^{\circ}\text{C}$  by cooling at a rate of  $40^{\circ}\text{C}$  per hour and holding for a period of 20 hours. The temperature was then raised to room temperature at a rate of  $40^{\circ}\text{C}$  per hour.



**Figure 8.** Schematic diagram of cryogenic treatment before austempering.

### Determination of Mechanical Properties

Mechanical properties were determined with the use of a Satec tensile testing machine located at the John Deere Waterloo foundry. Mechanical property analysis was completed under the guidelines of ASTM A 370.

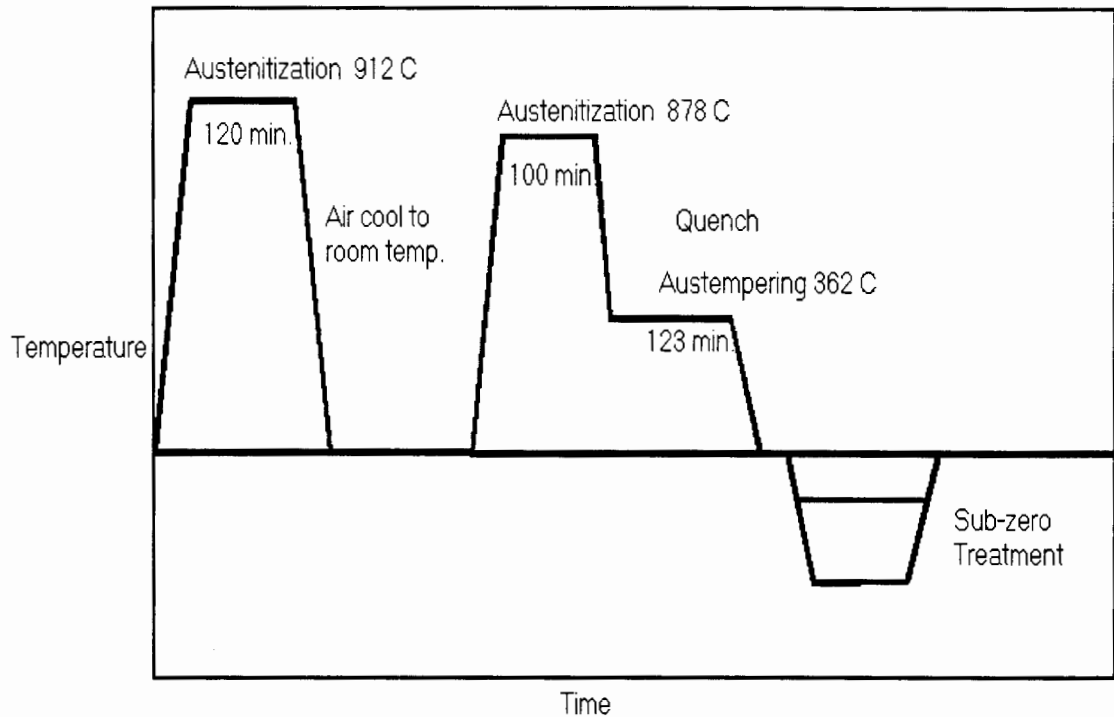


Figure 9. Schematic diagram of cryogenic treatment after austempering.

### Sample Preparation

The castings were sectioned as outlined in figure 10 to provide 1"x 1"x 8" rectangular specimens. Two specimens were removed from each ASTM Y-block casting. Specimens a and b were used in the tensile testing of the samples. A section from each test bar will be used in the microscopic evaluation of the materials structure after treatments. All thermal and cryogenic treatments were conducted before the samples were machined for tensile testing. Machining of the tensile specimens was conducted at



the American Pattern Company, located in Cedar Falls, Iowa. Gages used in the measurement of the tensile specimens were calibrated per ISO 9000.

### Tensile and Yield Determination

All tests were completed in accordance with ASTM A897 Standard Specification for Austempered Ductile Iron Castings. And ASTM A 370 Methods and Definitions for Mechanical Testing of Steel Products. The mechanical tests were completed with a Satec 60,000 pound capacity tensile test machine and Tinius Olsen Brinell hardness tester. Tensile strength, yield strength and elongation were recorded for each of the samples.

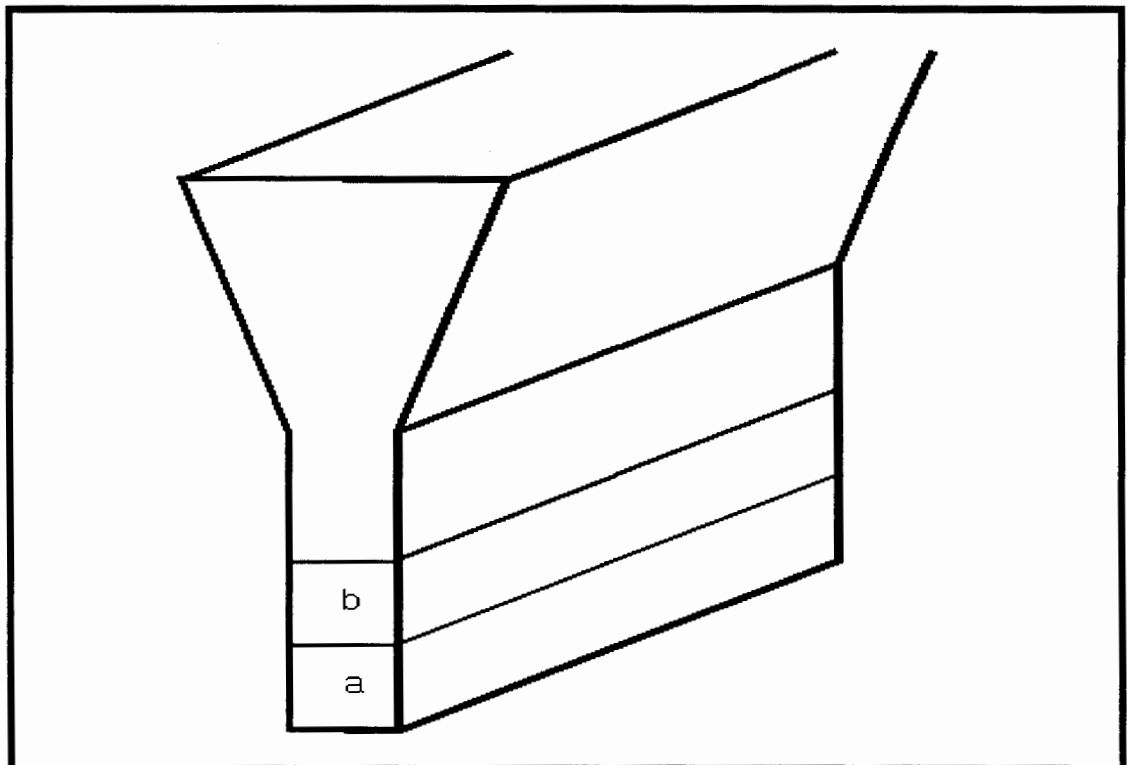


Figure 10. Sectioning of test specimens.

Statistical Evaluation of the Data

The research data collected during the study was analyzed using a oneway Analysis of Variance (ANOVA) at the 0.05 level of significance. A Sheffee' post hoc test was used after a significant F was found in the oneway ANOVA, to determine all possible significant differences between the groups. The type and number of tests are outlined in Table 2.

Table 2

Group Array of Tests for Determination of Effects

Group	No Austempering Treatment	Sub-Zero Treat Before Austempering	Sub-zero Treat After Austempering
No Sub-zero Treatment	Group G	None	None
Refrigeration Treat at -93°C	Group A	Group B	Group C
Cryogenically Treat at -210°C	Group D	Group E	Group F

Note. Group H was the control Group and was austempered without cryogenic treatment. Group I was tested as cast, without any treatment, for reference purposes only.

## CHAPTER 4

### RESULTS AND DISCUSSION

#### Base Material for Tests

Chemical analysis of the test heat was obtained using an Angstrom optical emission spectrograph and a Leco carbon sulphur determinator located at the John Deere Waterloo foundry. The foundry metallurgical laboratory was also used for the tensile and hardness testing of the samples. Metallographic analysis of the samples was completed using a Nikon Unigraph metallograph with image analysis system to determine nodule count, nodularity, pearlite and ferrite content. Higher magnification metallographic work at 1000X was completed at the John Deere Product Evaluation Center. Results of the chemical analysis are listed in table 3. Complete results of the mechanical tests are listed in appendix A. Statistical analysis of the mechanical property data was completed using SPSS for windows version 6.1.2. and is listed in appendix B.

Molybdenum percentages were slightly lower than the target value of .30 %. This low value resulted from oxidation of the Molybdenum oxide briquettes and as a result of the floatation of the alloy on the surface of the melt partially trapping the alloy in the slag layer of the molten bath. This percentage of Molybdenum in combination with the residual amounts of other carbide forming elements was felt adequate to develop similar results to Kies and Schelleng (1968). Nickel content, determined to be 1.34% was lower than used in Kies and Schelleng's research. All other elements were within industry standards for Austempered Ductile Iron (Rouns & Rundman, 1987).

Table 3

Chemistry of Material Used for Testing.

C	Si	Mn	Ni	Cr	Mo	Cu	P	S	Mg
3.76	2.65	.373	1.34	.04	.228	.16	.016	.006	.039

Structure and Properties of the Base Material

The as cast samples exhibited a mean hardness of 235 BHN as shown in Table 4. The nodularity rating of the sample as reported by image analysis was determined to be in excess of 200 nodules per square centimeter. Matrix structure of the as cast samples shown in figure 11, was 65% pearlite as reported by image analysis. Figure 12 illustrates the pearlite content of the as cast samples. Mean tensile strength of the as cast samples was 709.9 MPa which corresponded to over 100 ksi.

Effects of Normalizing Treatment

The normalizing of the samples raised the mean hardness from 235 BHN in the as cast samples to 240 BHN for the normalized samples. Mean tensile was increased from 709.9 MPa to 713.7 MPa as shown in table 4. This mechanical property increase coincided with an slight increase of pearlite in the normalized samples as shown in Figure 12. The pearlite contained in all of the samples existed in two distinct areas. The majority

of the pearlite was very fine and could only be resolved at a magnification of 1000X. Within these areas coarser pearlite was resolvable at 400X as shown in Figure 13. The mechanical properties of the samples meet the requirements of the ASTM A 536 grade 80--55--06 are only slightly below grade 100--70--03 as a result of the tensile failure at the gage marks of the specimens.



Figure 11. Structure of as cast samples 100X 4% Nital.

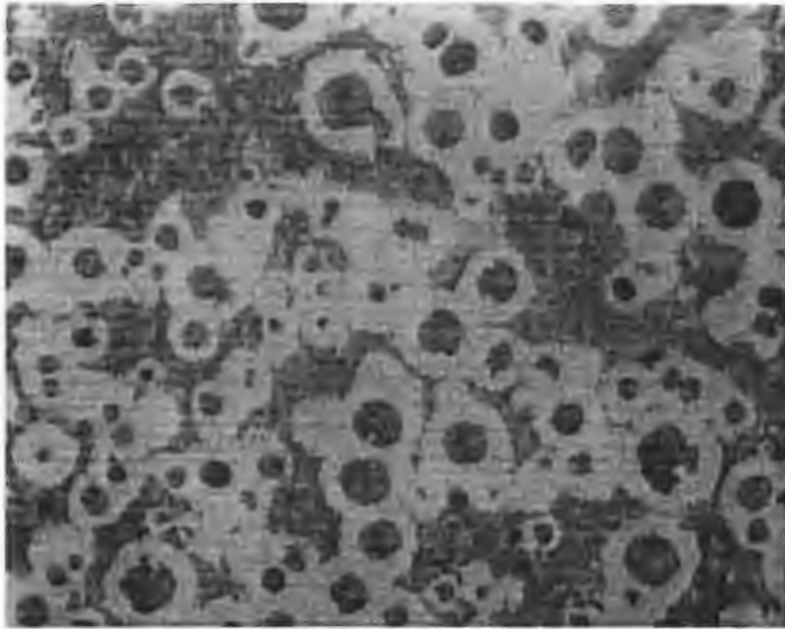


Figure 12. Structure of normalized samples 100X 4% Nital.



Figure 13. Pearlite resolvable at 400X and 1000X in the cryogenically treated samples 400X 4% Nital.

Table 4

Mean Values for the Mechanical Properties of the Test Samples

Group	Tensile (MPa)	Yield (MPa)	Elongation	BHN
I (AC)	709.9	465.83	7.50	235
H (NA)	949.5	630.7	6.61	285
G (N)	713.7	471.6 *	6.13 **	240
A (NR)	765.7	511.6	4.49 **	270
D (NC)	821.9	531.9	5.82	302
B (NRA)	958.1	622.7	7.12	297
E (NCA)	970.4	620.2	7.40	298
C (NAR)	1006.2	674.7	7.31	302
F (NAC)	1136.7	801.4	2.66	345

Note. \* Yield was not recorded for one of three samples. \*\* Fracture occurred at gage marks.

AC -- As Cast, N --Normalize, A--Austempered, R--Refrigeration, C--Cryogenic treatment.

Effects of Refrigeration after Normalizing

Refrigeration of the normalized samples yielded a slight increase in tensile strength of the hardness values of the samples, raising the tensile to 765.7 MPa and BHN to a mean value of 270 as shown in Table 4. This change was not statistically significant for either the tensile or BHN values of the samples as shown in appendix B.

Microstructural changes due to the refrigeration treatment were not evident, although the

samples did contain a larger percentage of pearlite in the structure as shown in Figure 14. This increase in pearlite is not thought to be a result of the treatment but rather due to the normal variation in the cooling rate of the original normalized samples accounting for the increase in both tensile and BHN hardness. Martensite was not observed in any of the normalized and refrigerated samples.

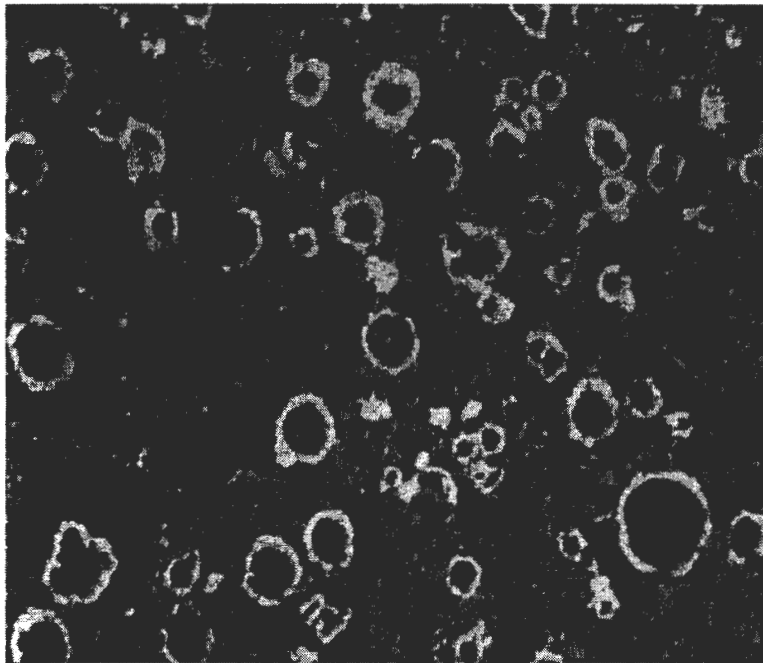


Figure 14. Structure of refrigerated sample 100X 4% Nital.

#### Effects of Cryogenic Treatment after Normalizing

Tensile and BHN hardness values were increased in the cryogenically treated samples as shown in table 4 from 713.7 MPa to 765.7 MPa for tensile and 240 BHN to



270 BHN respectively. Both the tensile and BHN increases were determined significant at the .05 level of significance. Examination of the microstructure revealed very fine martensite in small areas thought to be resulting from transformation of any retained austenite from the normalizing treatment as seen in Figure 15. Etching of the samples with a solution of CuCl revealed the possibility of retained austenite in the samples as shown in Figure 16. It is noteworthy that this phase was observed to some extent in all of the samples.



Figure 15. Fine martensite in normalized and cryogenically treated samples 1000X 4% Nital.

### Effects of Refrigeration Treatment Preceding Austempering

Although there was an increase in tensile strength and hardness of the samples that received this treatment compared to those receiving the normalizing treatment, this increase can be associated with the austempering of the samples and not with the prior refrigeration treatment. Mean tensile and hardness values increased from the normalized sample value of 713.7 MPa to 958.1 MPa and from 240 BHN to 297 BHN respectively. The increase from samples that were normalized and austempered without sub-zero temperature treatment was much less and was found to be statistically insignificant as seen in Appendix B. The microstructure of the samples as seen in Figure 17 revealed a matrix consisting primarily of ausferrite and graphite nodules.

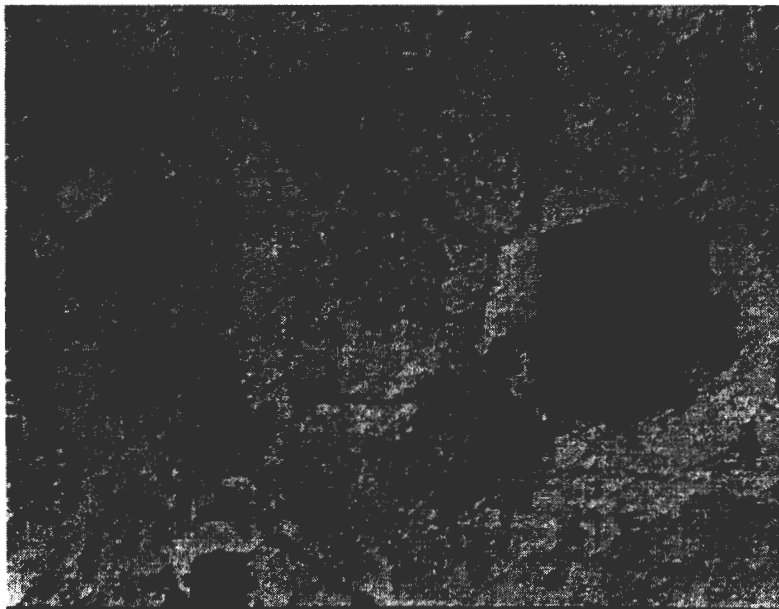


Figure 16. Light areas indicating retained austenite in normalized & cryogenically treated sample 400X etched CuCl.

### Effects of Cryogenic Treatment Preceding Austempering

Samples that were treated cryogenically before austempering showed similar results to the refrigerated and austempered samples with increases in both tensile strength and hardness from the normalized samples and slight increases from the normalized and austempered results. The increase in tensile strength and hardness was not statistically significant when comparing the normalized and austempered group of samples with the cryogenic treatment and subsequent austempering group as seen in Appendix B. The microstructure of the samples as seen in Figure 18 consists of graphite nodules in a matrix of ausferrite.

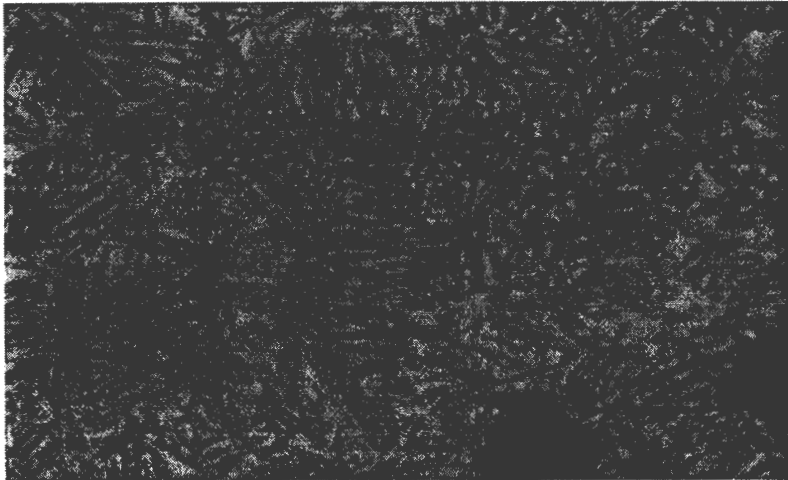


Figure 17. Microstructure of refrigerated and austempered sample illustrating ausferrite matrix 400X 4% Nital.

### Effects of Refrigeration Treatment following Austempering

Samples receiving a refrigeration treatment following austempering show a slight increase in tensile strength and hardness over the samples that were normalized and austempered as seen in Appendix A. Statistical analysis of the data determined that the observed increase was not statistically significant to show an effect of refrigeration treatment on austempered the structure as shown in Appendix B. The microstructure of these samples consisted of graphite nodules in a matrix of ausferrite as seen in Figure 19.

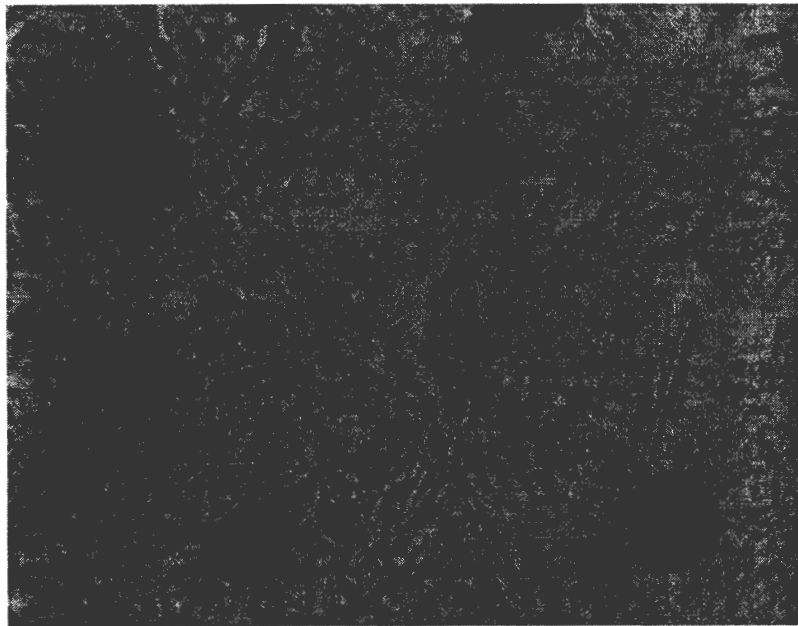


Figure 18. Microstructure of cryogenically treated and austempered sample 400X, 4% Nital.

### Cryogenic Treatment following Austempering

Samples that were cryogenically treated after austempering show the largest increase in tensile strength of all the treatment groups. Tensile strength increased from 949.5 MPa in the normalized and austempered samples to 1136.67 MPa when cryogenically treated. Hardness values increased from 285 BHN in the normalized and austempered samples to 345 BHN when cryogenically treated. Both the tensile and hardness increases were determined to be statistically significant. Comparison of the microstructure of the austempered cryogenically treated sample with the normalized austempered sample shows that ausferrite is present in both structures but differs in that the size of the ausferrite in that the cryogenically treated sample is much finer. This fine ausferrite is shown in Figure 20.

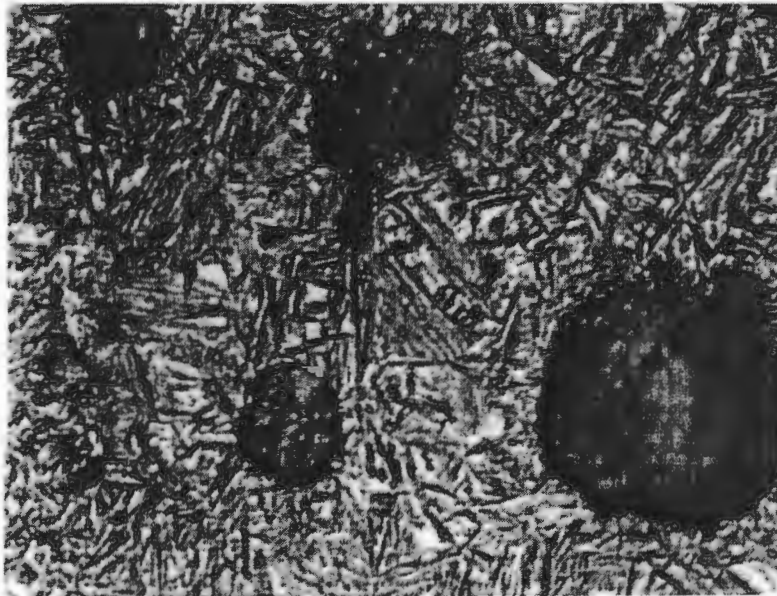


Figure 19. Microstructure of refrigeration treatment following austempering 400X 4% Nital.

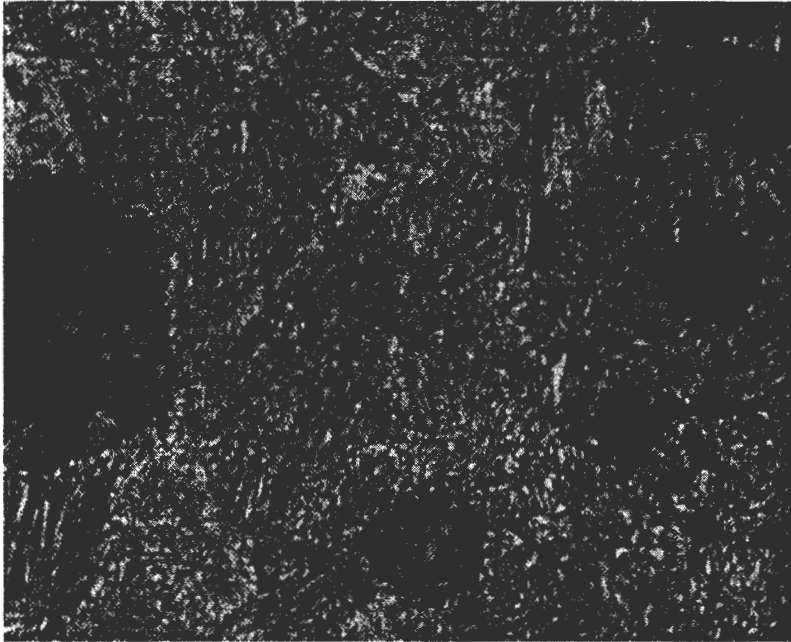
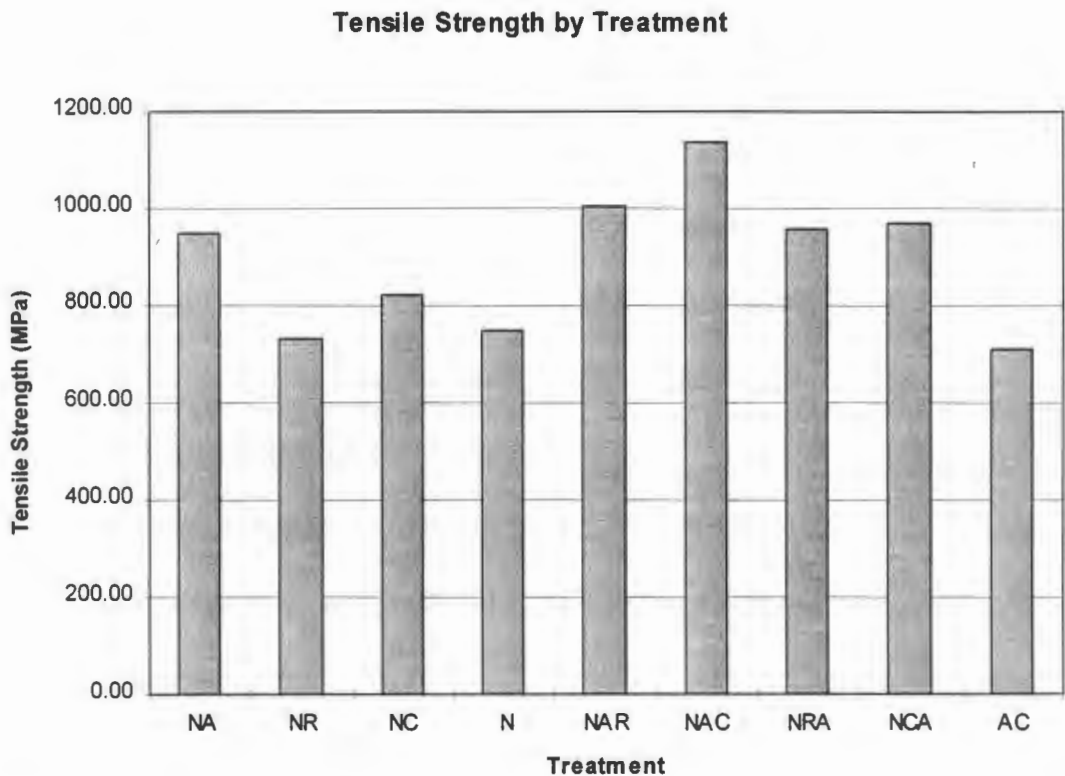


Figure 20. Microstructure of austempered and cryogenically treated sample. 400X, 4% Nital.

The research demonstrated that the greatest effect of the treatments studied was the increase of mechanical properties obtained when previously austempered ductile iron was subjected to cryogenic temperatures. This is detailed in Figure 21 which compares the treatment groups and their mean tensile strengths. This would seem to agree with the published literature in that the greatest effect of sub-zero treatments is to lower the temperature of the material to below the  $M_s$  region to form martensite. The resultant martensite appears to be much finer than that obtained from more conventional quench

and temper treatments. Of all the samples studied, the austempered ductile iron groups contained the largest percentage of retained austenite. This leads the researcher to believe that cast irons with minimum amounts of alloying elements, that promote the presence of retained austenite could also be modified using refrigeration or cryogenic treatment. Resources were not available to determine if Eta-carbides as described by Meng et al. (1994) could be reproduced in this alloy.



**Figure 21.** Tensile strength as a function of treatment.

## CHAPTER 5

### SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

In this research, the effects of two sub-zero temperature treatments have been studied when applied to a cast ductile iron which has the necessary alloying elements and chemical composition to benefit from the treatment. Although fully austenitic irons and high alloy steels have been investigated as to the effects of both refrigeration and cryogenic treatments, it is the authors understanding that few if any research has been conducted on the alloy family of austempered ductile irons. The literature shows that alloys that contain a percentage of retained austenite in their structure can have their physical properties modified by sub-zero temperature treatment. This effect is accomplished by lowering the temperature of the material to below the  $M_s$  temperature of that specific alloy to transform retained austenite in the structure to martensite. This transformation results in increased tensile, yield and hardness values for the alloy without the degradation of the surface finish of the component (Carlson, 1995). This preservation of the surface finish could allow for finishing operations including complex machining to be accomplished in a softer, more easily machineable state and at a later time be hardened to resist wear without degradation of this finish and need of subsequent machining operations.

Austempered ductile iron when subjected to cryogenic temperatures showed a mean increase of 187 MPa (27 ksi) tensile strength, 179 MPa (26 ksi) yield strength and an increase in hardness of 40 BHN over the untreated samples. Samples that received only a normalizing treatment prior to sub-zero temperature treatment appeared to exhibit



hardness increases unproportionate to the increase in tensile and yield strength as shown in figure 22 and 23.

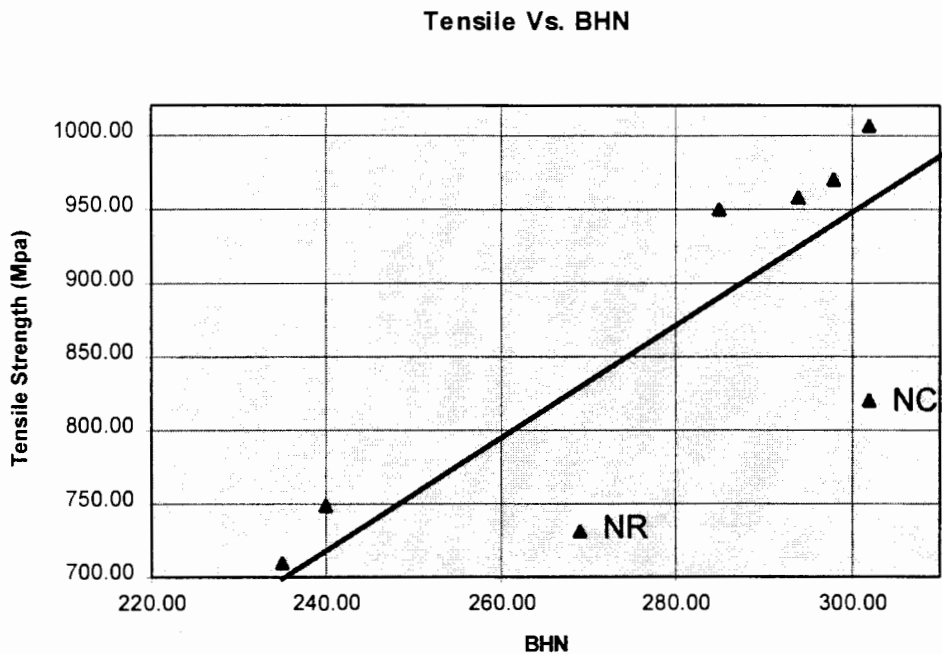


Figure 22. Tensile strength and respective hardness for test samples.

### Recommendations for Future Research

Because of the constraints of resources and equipment placed on the study it would be recommended that further research be completed before this information is utilized in an industrial application to produce cast components. Future research should be conducted with larger sample sizes in each of the treatment groups. It would also be recommended that future studies explore the variation of elements used to form retained austenite in the materials. The current study used only nickel, by far the most common

austenite stabilizer, but possibilities exist for the use of copper in amounts in excess of those used to promote pearlite. The possibility exists that copper used in conjunction with nickel could be a cost effective replacement for nickel alone (Viau, Gagne, & Thibau, 1977). The most valuable work on the ADI family may well be the study of sub-zero treatments on unalloyed austempered ductile iron.

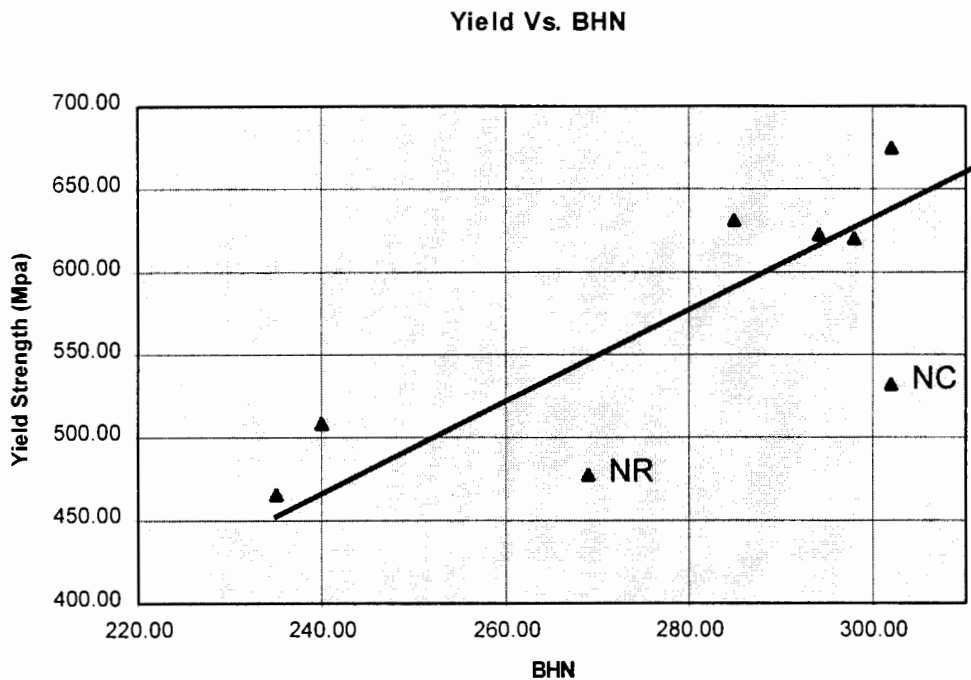


Figure 23. Yield strength and respective hardness for test samples.

Further research should be aided by the use of X-ray diffraction to determine the amounts of retained austenite in the samples before sub-zero temperature treatment and after treatment to determine the amount of retained austenite transformed to martensite.

X-ray diffraction in conjunction with scanning electron microscopy could determine if Eta-carbides could be produced in the treated samples. The production of these carbides may be facilitated by the cycling of refrigeration or cryogenic treatments with re-austenitization. Further studies should determine the effect of sub-zero temperature treatments on the wear resistance and impact strength of the materials. As a result of the fine martensitic structures obtained with sub-zero temperature treatments, impact properties at high hardness may be improved.

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## APPENDIX A

Mechanical Property Data

Group	Serial No.	Treatment	Tensile (MPa)	Yield (MPa)	%Elongation	BHN
H	1	NA	950.10	642.20	6.85	285
H	2	NA	955.30	619.80	7.11	285
H	3	NA	943.10	630.00	5.87	285
mean value			949.50	630.67	6.61	285
A	4	NR	791.90	525.60	3.39	262**
A	5	NR	770.50	506.50	6.14	285
A	6	NR	734.70	501.70	3.35	262**
mean value			765.70	511.27	4.29	270
D	7	NC	775.10	502.30	6.57	302
D	8	NC	876.90	560.50	5.41	302
D	9	NC	813.80	532.80	5.47	302
mean value			821.93	531.87	5.82	302
G	10	N	717.90	496.00	4.29	229**
G	11	N	749.30	none	6.71	262
G	12	N	674.00	447.20	7.40	229
mean value			713.73	471.60	6.13	240

Group	Serial No.	Treatment	Tensile (MPa)	Yield (MPa)	%Elongation	BHN
B	13	NRA	971.30	619.50	7.80	293
B	14	NRA	964.50	621.70	7.76	297
B	15	NRA	938.40	626.80	5.79	292
mean value			958.07	622.67	7.12	294
E	16	NCA	996.70	625.70	9.33	300
E	17	NCA	959.80	607.00	6.00	302
E	18	NCA	954.80	627.80	6.87	292
mean value			970.43	620.17	7.40	298
C	19	NAR	982.60	678.60	6.18	302
C	20	NAR	1023.00	673.50	7.83	302
C	21	NAR	1013.00	671.90	7.93	302
mean value			1006.20	674.67	7.31	302
F	22	NAC	1169.00	798.80	3.56	352
F	23	NAC	1116.00	796.70	2.34	341
F	24	NAC	1125.00	808.70	2.07	341
mean value			1136.67	801.40	2.66	345
I	25	AC	714.60	469.90	7.17	229
I	26	AC	699.90	462.80	7.62	229
I	27	AC	715.20	464.80	7.70	248
mean value			709.90	465.83	7.50	235

Note. \*\* Fracture occurred at gage marks. AC -- As Cast, N --Normalize, A--Austemper, R--Refrigeration, C--Cryogenic treatment.

## APPENDIX B

Statistical Property Results

Variable BHN  
By Variable Treatment

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	8	27487.1852	3435.8981	37.6499	.0000
Within Groups	18	1642.6667	91.2593		
Total	26	29129.8519			

Group	Count	Mean	Standard Deviation	Error	95 Pct Conf Int for Mean
Grp 1	3	269.6667	13.2791	7.6667	236.6793 TO 302.6540
Grp 2	3	296.6667	3.5119	2.0276	287.9426 TO 305.3908
Grp 3	3	302.0000	.0000	.0000	302.0000 TO 302.0000
Grp 4	3	302.0000	9.0000	5.1962	279.6425 TO 324.3575
Grp 5	3	298.0000	5.2915	3.0551	284.8550 TO 311.1450
Grp 6	3	344.6667	6.3509	3.6667	328.8901 TO 360.4432
Grp 7	3	240.0000	19.0526	11.0000	192.6703 TO 287.3297
Grp 8	3	285.0000	.0000	.0000	285.0000 TO 285.0000
Grp 9	3	235.3333	10.9697	6.3333	208.0829 TO 262.5838
Total	27	285.9259	33.4721	6.4417	272.6848 TO 299.1670

GROUP      MINIMUM      MAXIMUM

Grp 1	262.0000	285.0000
Grp 2	293.0000	300.0000
Grp 3	302.0000	302.0000
Grp 4	293.0000	311.0000
Grp 5	292.0000	302.0000
Grp 6	341.0000	352.0000
Grp 7	229.0000	262.0000
Grp 8	285.0000	285.0000
Grp 9	229.0000	248.0000
TOTAL	229.0000	352.0000



Variable BHN  
 By Variable Treatment.

Multiple Range Tests: Scheffe test with significance level .05

The difference between two means is significant if  
 $MEAN(J) - MEAN(I) \geq 6.7550 * RANGE * \sqrt{1/N(I) + 1/N(J)}$   
 with the following value(s) for RANGE: 6.34

(\*) Indicates significant differences which are shown in the lower triangle

Mean	Treatment	
		9 7 1 8 2 5 3 4 6
235.3333	Grp 9	
240.0000	Grp 7	
269.6667	Grp 1	
285.0000	Grp 8	* *
296.6667	Grp 2	* *
298.0000	Grp 5	* *
302.0000	Grp 3	* *
302.0000	Grp 4	* *
344.6667	Grp 6	* * * * * * * *

Variable TENSILE  
By Variable Treatment

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	8	517574.4119	64696.8015	82.1121	.0000
Within Groups	18	14182.3533	787.9085		
Total	26	531756.7652			

Group	Count	Mean	Standard Deviation	Error	95 Pct Conf Int for Mean
Grp 1	3	765.7000	28.9005	16.6857	693.9063 TO 837.4937
Grp 2	3	958.0667	17.3679	10.0274	914.9220 TO 1001.2114
Grp 3	3	1006.2000	21.0409	12.1480	953.9309 TO 1058.4691
Grp 4	3	821.9333	51.3850	29.6672	694.2844 TO 949.5823
Grp 5	3	970.4333	22.8846	13.2124	913.5843 TO 1027.2824
Grp 6	3	1136.6667	28.3608	16.3741	1066.2138 TO 1207.1195
Grp 7	3	713.7333	37.8225	21.8368	619.7759 TO 807.6907
Grp 8	3	949.5000	6.1221	3.5346	934.2917 TO 964.7083
Grp 9	3	709.9000	8.6654	5.0030	688.3736 TO 731.4264
Total	27	892.4593	143.0111	27.5225	835.8859 TO 949.0326

GROUP MINIMUM MAXIMUM

Grp 1	734.7000	791.9000
Grp 2	938.4000	971.3000
Grp 3	982.6000	1023.0000
Grp 4	775.1000	876.9000
Grp 5	954.8000	996.7000
Grp 6	1116.0000	1169.0000
Grp 7	674.0000	749.3000
Grp 8	943.1000	955.3000
Grp 9	699.9000	715.2000
TOTAL	674.0000	1169.0000



Variable YEILD  
By Variable TREATMEN Treatment

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	8	267141.9472	33392.7434	154.2010	.0000
Within Groups	17	3681.4067	216.5533		
Total	25	270823.3538			

Group	Count	Mean	Standard Deviation	Error	95 Pct Conf Int for Mean
Grp 1	3	511.2667	12.6429	7.2994	479.8596 TO 542.6738
Grp 2	3	622.6667	3.7448	2.1620	613.3640 TO 631.9693
Grp 3	3	674.6667	3.4990	2.0202	665.9745 TO 683.3589
Grp 4	3	531.8667	29.1112	16.8074	459.5496 TO 604.1837
Grp 5	3	620.1667	11.4509	6.6112	591.7207 TO 648.6126
Grp 6	3	801.4000	6.4086	3.7000	785.4800 TO 817.3200
Grp 7	2	471.6000	34.5068	24.4000	161.5687 TO 781.6313
Grp 8	3	624.6667	5.1160	2.9537	611.9577 TO 637.3756
Grp 9	3	465.8333	3.6611	2.1137	456.7387 TO 474.9280
Total	26	596.1846	104.0814	20.4120	554.1452 TO 638.2240

GROUP MINIMUM MAXIMUM

Grp 1	501.7000	525.6000
Grp 2	619.5000	626.8000
Grp 3	671.9000	678.6000
Grp 4	502.3000	560.5000
Grp 5	607.0000	627.8000
Grp 6	796.7000	808.7000
Grp 7	447.2000	496.0000
Grp 8	619.8000	630.0000
Grp 9	462.8000	469.9000
TOTAL	447.2000	808.7000



Variable ELONG  
By Variable Treatment

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	8	64.8243	8.1030	6.0467	.0008
Within Groups	18	24.1213	1.3401		
Total	26	88.9456			

Group	Count	Mean	Standard Deviation	Error	95 Pct Conf Int for Mean
Grp 1	3	4.2933	1.5994	.9234	.3202 TO 8.2665
Grp 2	3	7.1167	1.1491	.6634	4.2621 TO 9.9712
Grp 3	3	7.3133	.9828	.5674	4.8720 TO 9.7547
Grp 4	3	5.8167	.6531	.3771	4.1943 TO 7.4391
Grp 5	3	7.4000	1.7271	.9971	3.1096 TO 11.6904
Grp 6	3	2.6567	.7939	.4583	.6846 TO 4.6288
Grp 7	3	6.1333	1.6332	.9429	2.0761 TO 10.1905
Grp 8	3	6.6100	.6539	.3775	4.9856 TO 8.2344
Grp 9	3	7.4967	.2857	.1650	6.7869 TO 8.2064
Total	27	6.0930	1.8496	.3560	5.3613 TO 6.8246

GROUP	MINIMUM	MAXIMUM
Grp 1	3.3500	6.1400
Grp 2	5.7900	7.8000
Grp 3	6.1800	7.9300
Grp 4	5.4100	6.5700
Grp 5	6.0000	9.3300
Grp 6	2.0700	3.5600
Grp 7	4.2900	7.4000
Grp 8	5.8700	7.1100
Grp 9	7.1700	7.7000
TOTAL	2.0700	9.3300

Variable ELONG  
 By Variable TREATMEN Treatment

Multiple Range Tests: Scheffe test with significance level .05

The difference between two means is significant if  
 $MEAN(J) - MEAN(I) \geq .8186 * RANGE * \sqrt{1/N(I) + 1/N(J)}$   
 with the following value(s) for RANGE: 6.34

(\*) Indicates significant differences which are shown in the lower triangle

Mean	Treatment	
		6 1 4 7 8 2 3 5 9
2.6567	Grp 6	
4.2933	Grp 1	
5.8167	Grp 4	
6.1333	Grp 7	
6.6100	Grp 8	
7.1167	Grp 2	*
7.3133	Grp 3	*
7.4000	Grp 5	*
7.4967	Grp 9	*