

THE EFFECT OF SN CONTENT AND ISOTHERMAL TRANSFORMATION
TEMPERATURE ON THE MECHANICAL PROPERTIES OF
AUSTEMPERED DUCTILE CAST IRON.

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ABSTRACT

THE EFFECT OF SN CONTENT AND ISOTHERMAL TRANSFORMATION TEMPERATURE ON THE MECHANICAL PROPERTIES OF AUSTEMPERED DUCTILE CAST IRON.

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In this study the effects of Sn content and isothermal transformation temperature on the ultimate tensile strength (UTS), elongation and hardness of austempered ductile cast iron (ADI) was investigated. To determine the possible effect of Sn on these properties the Sn content of standard GGG30, GGG40 and GGG50 materials were taken as reference, whose chemical compositions vary from 0,016 to 0,050% in terms of Sn. However the Sn content was increased to a maximum of 0,26% for investigating the effect of Sn on mechanical properties. The test specimens were obtained from standard 1-in Y-blocks and austenitized at 925°C for 1 hour and austempered at 350°C, 375°C, 400°C and 420°C for 60 minutes in a salt bath.

Microstructural examination of the specimens were also done. To determine the amount of retained austenite of the specimens X-Ray analyses were used.

As a result of the study The UTS increases up to 0,1% Sn and then levels off. A similar behavior is observed in the variation of hardness. Percent elongation decreases and falls to a minimum around 0,1% Sn, from this point on, a slight increase is observed. Strength and hardness increase while percent elongation decreases with decreasing temperatures.

Keywords: Ductile Iron, Austempering, Sn content

ÖZ

ÖSTEMPERLEME İŞLEMİNDE KALAY MİKTARI VE İZOTERMAL DEĞİŞİM SICAKLIĞININ KÜRESEL GRAFİTLİ DÖKME DEMİRLERİN MEKANİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİLERİ

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Bu çalışmada kalay miktarı ve izotermal değişim sıcaklığının östemperlenmiş küresel grafitli dökme demirlerin çekme mukavemeti, uzama ve sertlik değerleri üzerindeki etkileri araştırılmıştır. Kalay miktarındaki değişimin olası etkisini belirlemek için standart GGG30, GGG40 ve GGG50 malzemeleri referans olarak alınmıştır.

Bu malzemelerdeki kalay miktarı %0,016 ve %0,050 arasında değişmektedir. Bu çalışmada ise kalay miktarı, mekanik özellikler üzerindeki olası etkileri incelemek üzere %0,26'ya kadar arttırılmıştır.

Test numuneleri standart 1 inch (2,54 cm)lik Y bloklar kullanılarak elde edilmiştir ve 925°C'da 1 saat östenitlendikten sonra 350°C, 375°C, 400°C ve 420°C'deki tuz banyolarında 60 dakika östemperlenmiştir

Parçaların mikro yapıları incelenmiştir. Parçalardaki kalan östenit miktarını belirlemek üzere X-Işını analizi yapılmıştır.

Çalışmanın sonucunda numunelerin çekme mukavemetinin %0,1 Sn miktarına kadar arttığı ,bu değerin üzerindeki seviyelerde sabitlendiği belirlenmiştir. Benzer bir değişim sertlik değerlerinde de gözlenmiştir. Yüzde uzama değerlerinin % 0,1 Sn 'da minimuma indiği , bu değerden sonra da çok az yükseldiği belirlenmiştir. Artan izotermal değişim sıcaklığıyla çekme mukavemeti ve sertlik değerleri azalırken uzamanın arttığı belirlenmiştir.

Anahtar Kelimeler: Küresel Grafitli Dökme Demir, Östemperleme, Kalay Miktarı

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CHAPTER 1

INTRODUCTION

Austempered Ductile Cast Iron (ADI) is a material that offers the best combination of low cost, design flexibility, good machinability, high strength to weight ratio, good toughness, wear and fatigue resistance. After casting, Ductile Iron (DI) receives the austempering heat treatment, which brings mechanical properties that are superior to all cast irons, and many cast steels.

The production of ADI (austempering heat treatment) is neither new nor novel, and has been applied for several decades to ductile cast iron and wrought steels. The history of the ADI begins in 1952 at International Harvester Company succeeding in producing high strength ductile cast iron with bainitic matrix while investigating isothermal heat treatments of tank-track castings. The importance of the ADI increased by the improvements in 1970's.

The new and superior properties of ADI result from the effect of austempering heat treatment on carbon and silicon rich Ductile Iron matrix.

The preponderance of information on the austempering of steel, and the superficial similarities between the austempering heat treatments applied to

steels and ADI, have resulted in the understanding of the microstructure formed which gives ADI its superior mechanical properties. ADI is often referred to as “Bainitic Ductile Iron”, because heat treated ones contains bainite, like in austempered steel, is a mixture of acicular ferrite and carbide in the needle like form.

In many cases composition of ADI castings differs little from conventional Ductile Iron casting. When selecting the composition for both ADI and conventional Ductile Iron, consideration should be given first to limiting elements which adversely effect the casting quality through the production of non-spheroidal graphite, the formation of carbides and inclusions, and promoting the shrinkage. For this reason it’s known from the previous research that the percentages of carbon, silicon and other major and minor alloying elements that control the mechanical properties are important.

This study evaluates the effect of a minor alloying element Sn and the Isothermal Transformation Temperature in Austempering heat treatment on the tensile strength, elongation and hardness of ADI.

Tin(Sn) is used in the range of 0,025% to 0,1% in the casting process as a perlitic reaction stimulator. In raw castings Sn content within these limits can slightly increase the strength of the regions that would otherwise contain free ferrite. Excess of this level can lead to embrittlement in raw castings [1]. The Isothermal Transformation Temperature is one of the most important properties that affect all the mechanical properties of ADI.

The aim of the study is to determine the possible changes in the mechanical properties with the Sn content and the isothermal transformation temperature.

CHAPTER 2

LITERATURE REVIEW

2.1 Heat Treatment

The ADI heat treatment cycle consists of three main stages.

- 1) Austenitizing
- 2) Quenching
- 3) Isothermal Transformation

Schematic illustration of these steps can be seen in figure2.1.

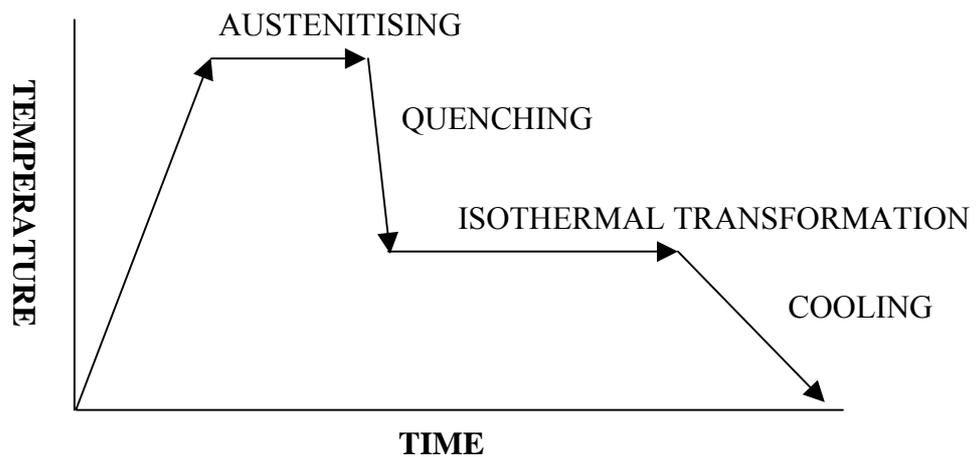


Figure 2.1. schematic illustration of all stages of Austempering Heat Treatment.

The final properties of the ADI are determined by all of these stages. The most important stage among them is the isothermal transformation.

2.1.1 Austenitizing

Austenitizing temperature and time are two main factors that affect the final properties of ADI.

The austenitizing temperature controls the carbon content of the austenite, which in turn affects the structure and properties of the austempered casting. The austenitizing temperatures above 925°C increase the carbon content of the austenite that increases the hardenability, while lowers the ductility through the formation of bainite after isothermal transformation stage. Reducing the austenitizing temperature produces ADI with the best properties, but in this case silicon content, which exerts a strong influence on this critical temperature should be controlled carefully [7]. Austenitizing temperatures in the range of 900°C-925°C seem to be good for all GGG40 standard castings. The effect of silicon content on the austenitizing temperature can be well understood by Figure 2 and 3.

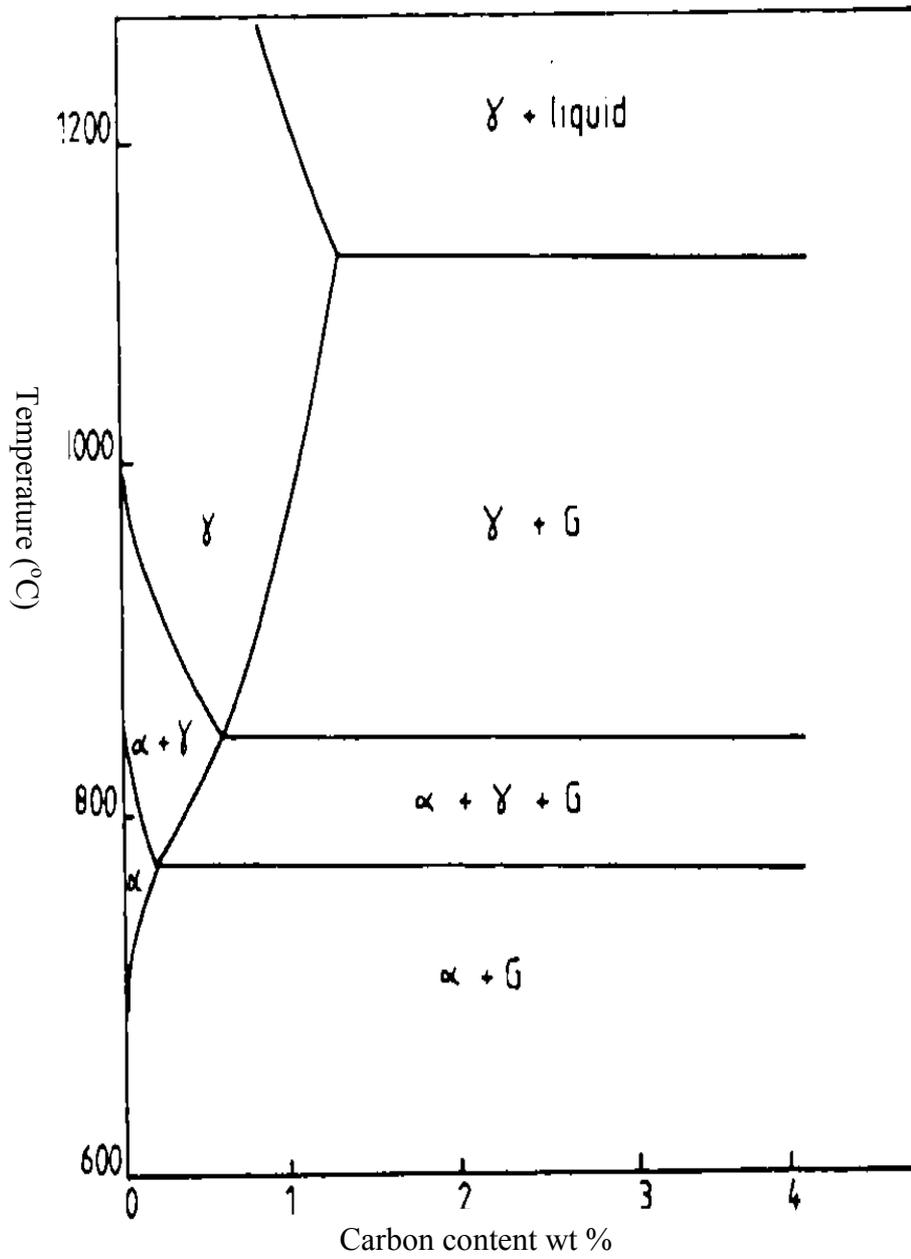


Figure 2.2. A section of Fe-C-Si phase diagram (Si 1%).[2]

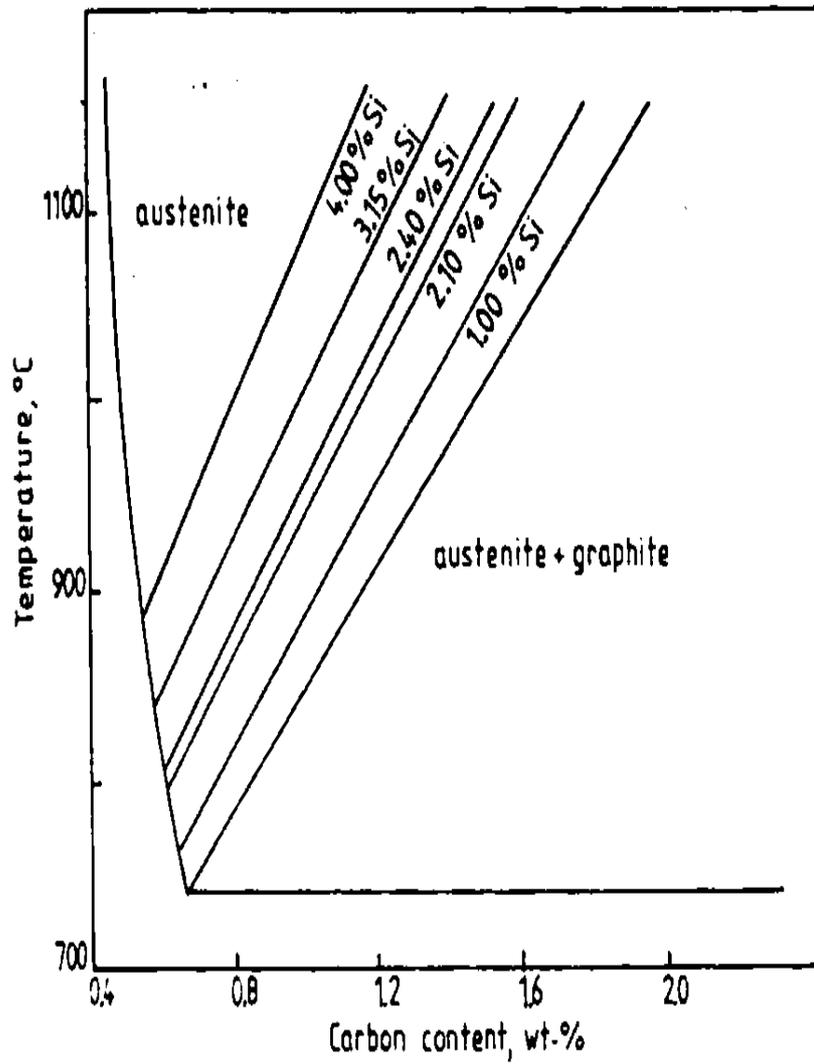


Figure 2.3. The change at the borders of the Fe-C-Si Phase diagram with the change of the Si content.[7]

As it can be seen from the figures austenitization temperature directly effected by the change of Si content.

Figure 2.4 shows the effect of austenitizing temperature on mechanical properties.

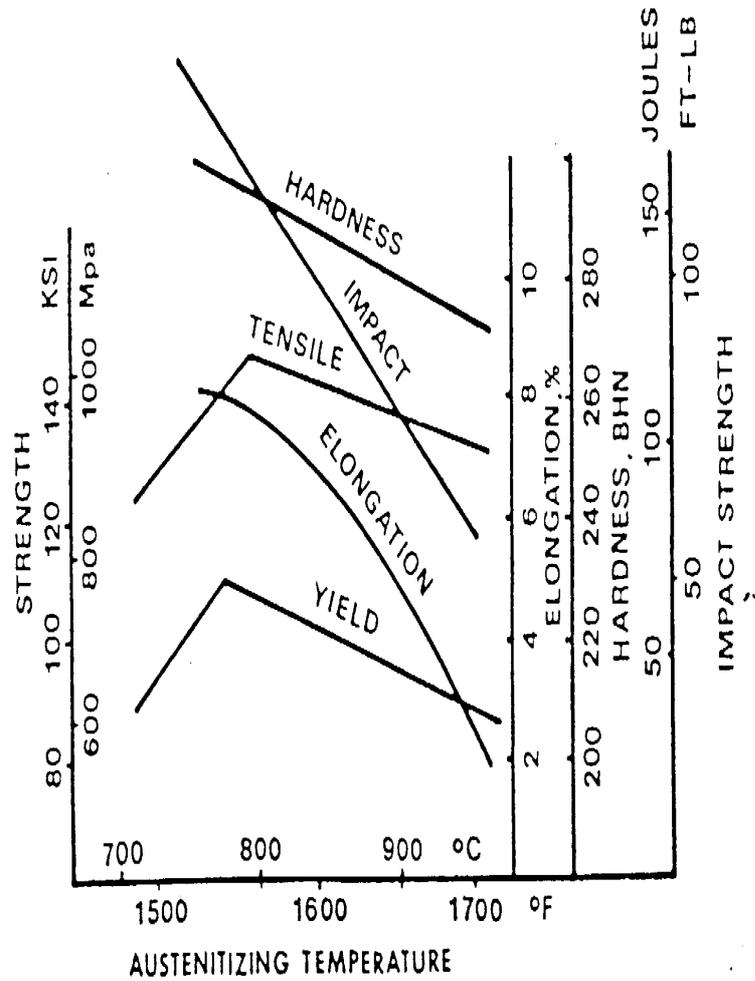


Figure 2.4. The effect of austenitizing temperature on the mechanical properties of austempered ductile cast iron. [3]

Austenitizing time should be enough to ensure that casting has been completely converted to carbon saturated austenite. At high austenitizing temperatures, a fully austenitic matrix may be obtained in 1 hour. On the other hand lower temperatures ensure higher quality through the reduction of the microsegregation of alloying elements so austenitizing time up to 3 hours may be required. Like the austenitizing temperature austenitizing time has some critical effects on the final mechanical properties as shown in figure 2.5

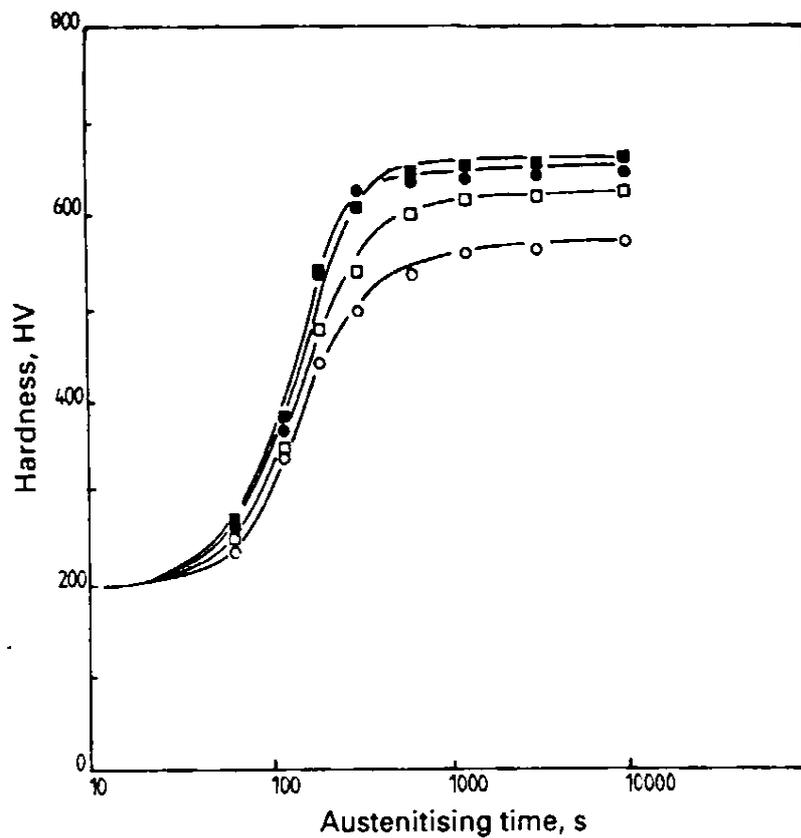


Figure 2.5 The effect of austenitizing time on hardness of ADI.[3]

2.1.2 Quenching

Quenching is the second stage of the Austempering Heat Treatment. In this stage, the most important factor that affects the final mechanical properties of ADI is the cooling rate of the austenitized casting. The importance of the cooling rate can be seen from the TTT diagram (figure 2.6) that shows the regions of transformation according to the microstructures.

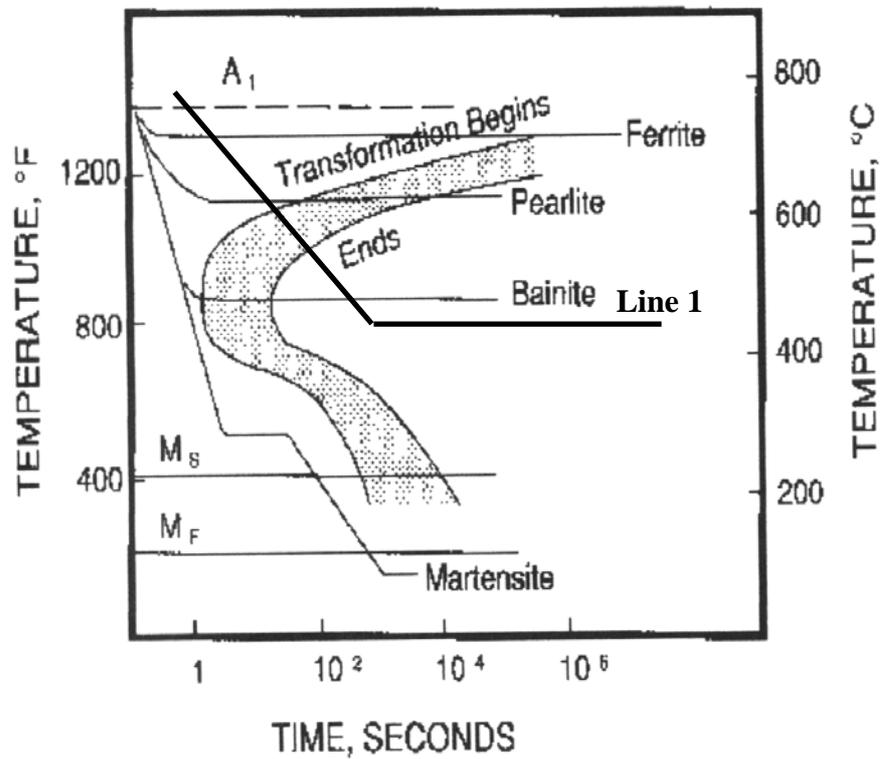


Figure 2.6 Typical TTT Diagram for a low silicon ductile cast iron.

The line 1 on the figure shows the path of an unsuccessful bainitic transformation. Because of the low cooling rate the transformation path crosses the pearlite region, which results in reduction of mechanical properties of ADI.

The amount of alloying elements is also important for quenching stage. Addition of alloying elements like Cu and Mo shift the C curves to left on TTT diagram and this motion stimulates the perlitic reaction. Therefore to avoid the formation of perlitic microstructure the cooling rate must be increased.

2.1.3 Isothermal Transformation

Isothermal Transformation is the main stage at which the bainitic transformation occurs. The time and temperature of Isothermal Transformation are the factors that affect the amount of bainitic transformation that occurs in the matrix. The two step reaction that takes place at this stage has an important effect on mechanical properties.

As a first step, the austenite(γ) decomposes into acicular ferrite(α) and high carbon austenite. The morphology and relative amount of bainitic ferrite formed during this first reaction are largely determined by the isothermal transformation temperature and time. [4-5].

The products of this reaction will be much finer, if isothermal transformation takes place at lower temperatures (<350°C).[6]



During the second step, the carbon-enriched austenite further decomposes into bainitic ferrite and carbides.



These carbides increase the hardness, while decrease the elongation and tensile strength of the specimens, so it is not preferred.

Isothermal transformation that leads to bainitic microstructure at the matrix can be explained in three different steps as follows.

- 1- Kinetic Definition For Bainitic Transformation.
- 2- Microstructural Definition.
- 3- Crystallographic Definition.

2.2 Definitions For Isothermal Transformation

2.2.1 Kinetic Definition For Bainitic Transformation

The investigations on the Kinetics of the bainitic transformation began by the Study of Davenport and Bain. In the beginning it was believed that the transformation of bainite was similar to martensite, which is known to be diffusionless This approach to the bainitic transformation is documented by Zener. However results obtained from the measurements of over all reaction rate, showed that the bainitic transformation is relatively slower when it is compared with the martensitic transformation. Further analyses revealed that the isothermal bainitic transformation is a nucleation and growth type process and also sometimes shows shear type transformation features. There are three steps of bainitic transformation; Nucleation of bainitic ferrite, growth and re-nucleation.[11]

The nucleation of the bainite starts at Bainite Start Temperature(B_s) that can be calculated by the use of the empirical formula (formula 1) stated by Steve and Haynes in 1956.[17]

$$B_s(^{\circ}\text{C}) = 830 - 270W\%_{\text{C}} - 90W\%_{\text{Mn}} - 37W\%_{\text{Ni}} - 70W\%_{\text{Cr}} - 83W\%_{\text{Mo}} \dots \text{formula 1}$$

B_s ($^{\circ}\text{C}$) : Bainitic transformation start temperature.

The nucleation starts when the energy obtained from temperature gradient exceeds the required activation energy for nucleation, G^* as it is shown in the studies of Bhadesia [18], the activation energy is related to the driving force of nucleation. He states that the nucleation process for bainite and Withmanstater ferrite is, like martensite, displacive in character but unlike martensitic nucleation, which is diffusionless, there is a redistribution of carbon during the formation of nucleus.[13] Another difference between martensitic nucleation and bainitic nucleation is rate of reaction. The nucleation rate of martensite is close to speed of sound while nucleation rate of bainite is lower than the speed of sound in iron (1Km/sec).

The growth is the step in which bainite leafs form. Type of the growth process is not completely clear today. However, it is known that depending on the temperature, amount of excess carbon at the nucleated sites is reduced with reaction. Ko and Cottrell stated that a coherent growth can only be at bainitic transformation temperature when the strain occurred due to the density change is relieved. This relieve of stress can only be obtained by reduction of the carbon content in bainite. Such kind of a reduction can occur either by diffusion from bainite or precipitation in bainite. Proceeding the growth stage, a new nucleation form at the tip of the growth body and the process continues as shown in figure 2.7.

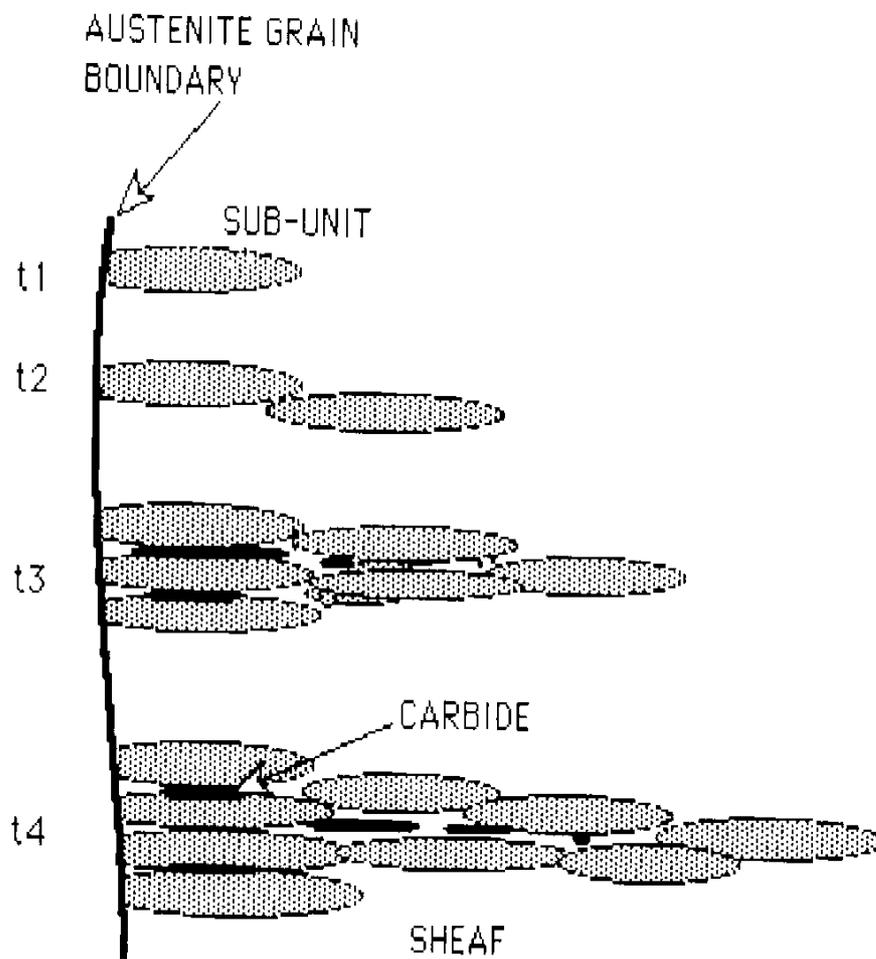


Figure 2.7 schematic illustration of the bainite formation stages. [11]

Time is another important concern for bainitic transformation and figure 2.8 and 2.9 illustrates the change of the length of bainite leaves and the amount of bainitic transformation with time.

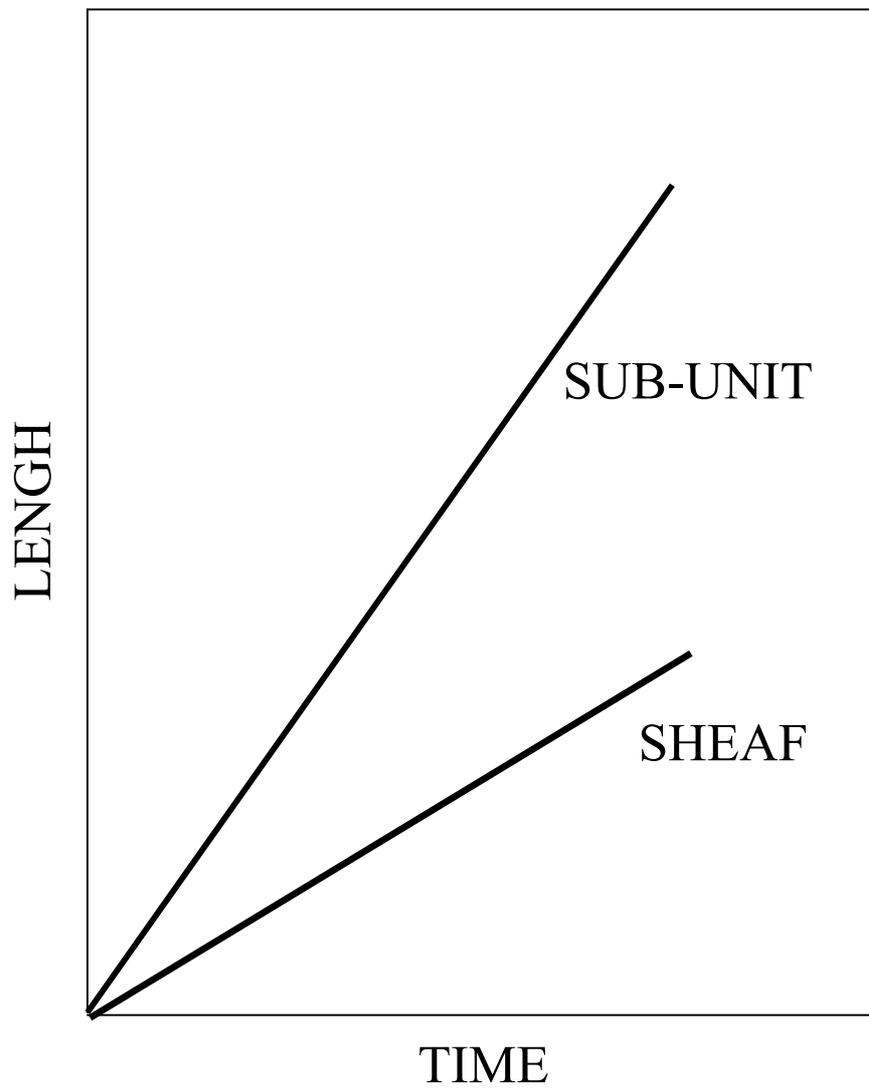


Figure 2.8 The change of the length of the sub-units and sheaf with time.[3]

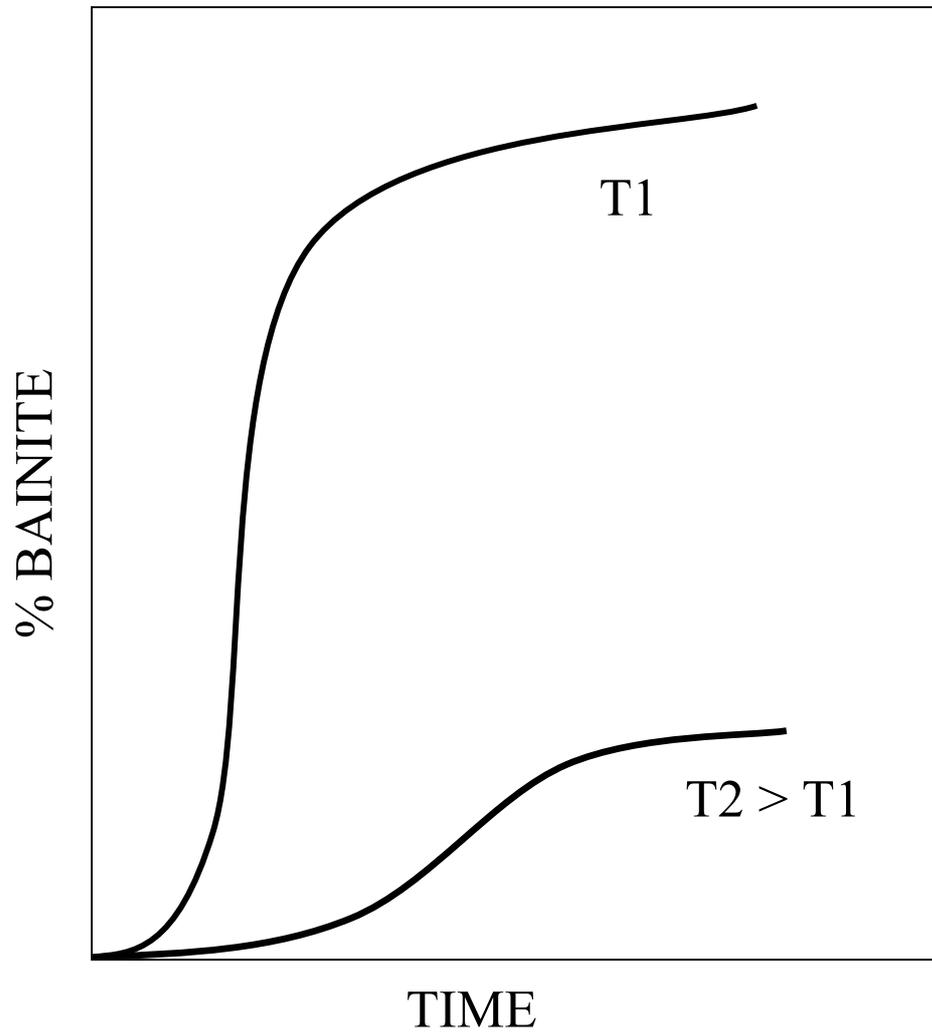


Figure 2.9 The change of the amount of bainitic transformation with time at different temperatures.[11]

To understand the nature of nucleation and these data are obtained by the frequent use of the special techniques like Dilatometry, Magnetometry and Resistometry. These methods provide the required data to understand the nature of nucleation and growth process. In addition to this by the use of the same methods the time intervals for the steps of bainitic transformation is obtained. This obtained data are used to construct the Transformation Diagrams for the practical use. There are two types of transformation diagrams.

- 1) Time Temperature Transformation Diagrams.(TTT)
- 2) Continuous Cooling Transformation Diagrams.(CCT)

These diagrams are used to determine the optimum time and temperature for the bainitic transformation that reveals required mechanical properties for design. The examples of TTT and CCT diagrams are given in the figures 2.10 and 2.11.

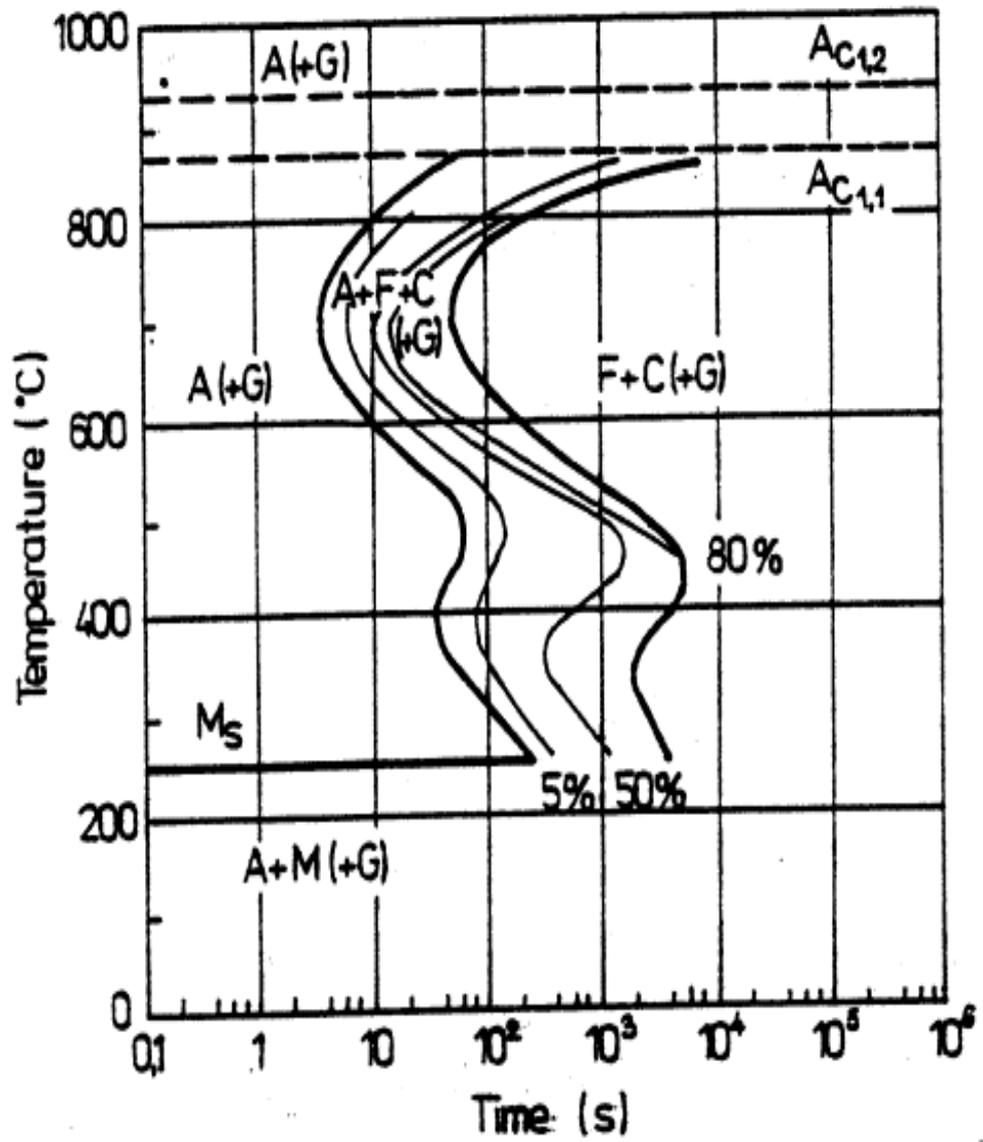


Figure 2.10. The TTT Diagram of un-alloyed ductile iron containing 0.27%Mn and 1.72% Si.

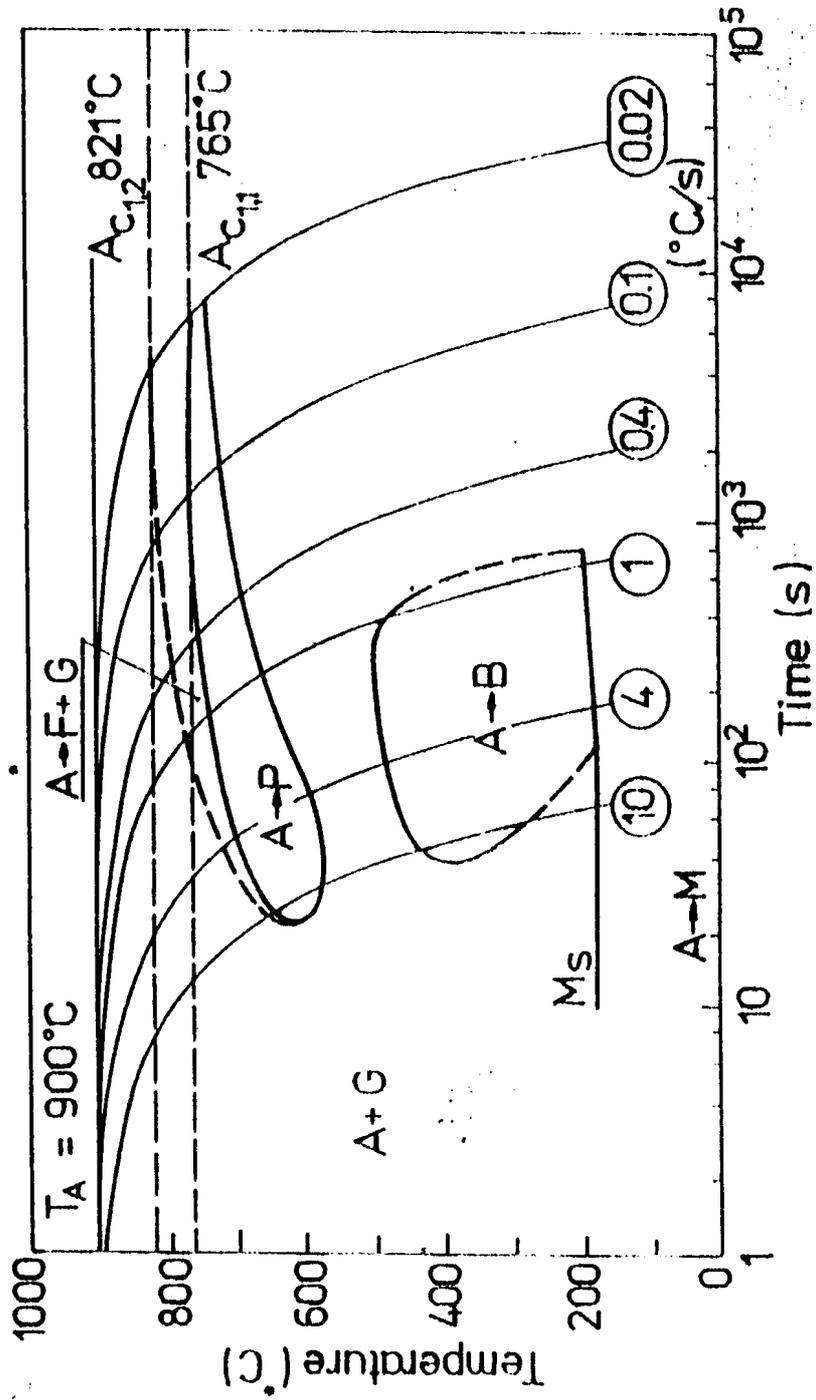


Figure 2.11 Continuous cooling transformation diagrams for unalloyed ductile iron (3.59% C, 2.50%Si,0.65%Mn, 0.051% P). [3]

2.2.2 Microstructural Definition

The bainitic microstructure that is generally seen in castings and steels, which consists of a non-lamellar mixture of ferrite and carbides, can be classified into two branches, Upper and Lower Bainite.

2.2.2.1 Upper Bainite

At austenitization temperatures above 350°C α grains diffuse to the γ matrix. This diffusion causes the motion of α grain boundaries to the γ matrix by the formation of high carbon γ areas. The shapes of these grain boundaries are either lath or needle like, which means one dimension is longer than the other two. Laths are connected either closely or separated by a thin retained austenite layer. The dimension of these individual laths are assumed to be 0,1 μm , while their shape is closely related with the rate of bainitic transformation.[14]

As it is explained, bainitic transformation is a diffusion-controlled process, in which the driving force is the difference between the austenitization and isothermal transformation temperatures. Needles and laths of bainitic ferrite can be found in initial stages of structure. Increase in the time of isothermal transformation leads to branching of the laths.

Generally branching occurs in advanced transformation stage of upper bainite. This causes a decrease in the amount of retained austenite between the branches of bainite. The branched particles can change the growth direction of some laths in the sheaf. Also, they can bring a non-uniform thickening of individually growing laths. Besides branching, nucleation of new particles on the existing laths of bainitic ferrite can result

same conclusions. The growth of the laths causes an increase at the amount of high carbon austenite by the emission of carbon from laths.

2.2.2.2 Lower Bainite

At temperatures below 350 °C bainite forms as single plates with a thickness between 0.5 to 0.1 μm.[3] The sheaves consist of high amount of parallel plates lying close to each other or separated by carbon enriched austenite. Some plates overlap each other in the direction of lengthening like the ones of the upper bainite. The majority of the structure forms from sheaves after about 10 min., which is relatively a short time.[11] The lower bainitic structure consists of a large number of narrow plates. If the sheaves consist of substructural ferrite units, their lengths become shorter than the overall length of the sheaf. The following items make the substructure of the sheaf more complex;

- The direction of lengthening of some plates
- The growing together of two or more plates
- The one-sided thickening of some of the plates

As in the case of upper bainite, these short lateral particles can also be observed in the sheaf, mutually separated by retained austenite.

The holding time and temperature also effects the complexity of the sheaves. Since the reaction is relatively faster than the ones at the upper bainite, even holding several minutes at a temperature of 300 °C causes an increase in the complexity of the substructure of the sheaves. The austenitic matrix, separate the bainitic sheaves of plates like narrow strips both along the length and across the width of the sheaf. As the holding time increases a similar structure can also be seen without destruction.

The determination of austenite between the bainitic plates is important since it resembles the success of the bainitic transformation. Increasing of the holding time does not change the thickness of the bainitic plates but the carbon content of the ferrite increases. The increase in the holding time gives a chance to the diffusion reactions to occur. As a result of these diffusion reactions some cementite precipitates form in the plates of bainite. The amount of austenite and cementite cannot be determined only by the use of micro-structural analysis. Instead X-ray techniques must be used. The differences between two microstructures are illustrated at the figure 2.12.

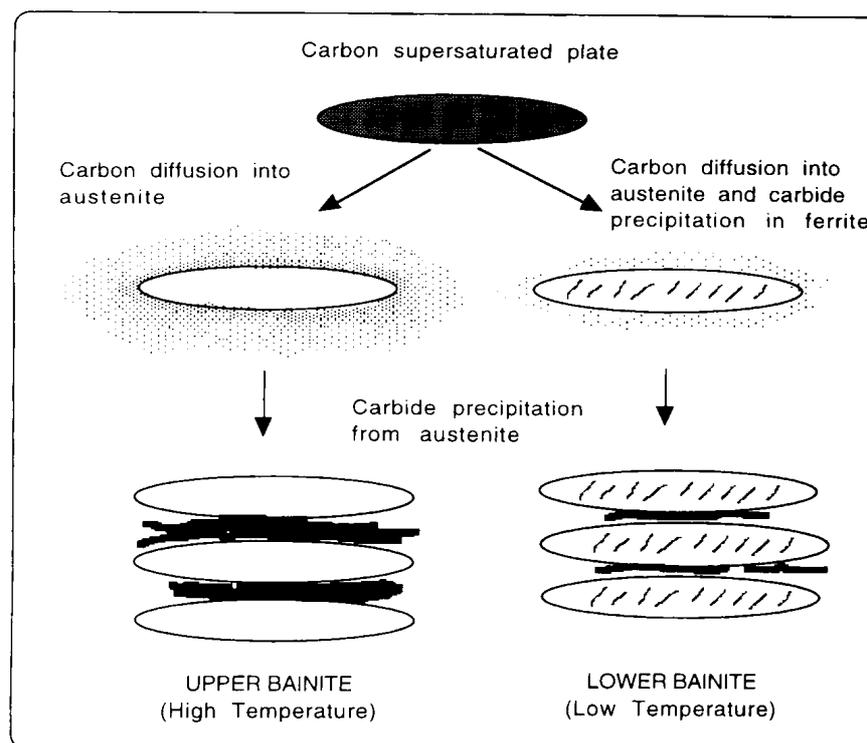


Figure 2.12. Schematic representation to the Upper and Lower Bainitic transformations. [11]

2.2.2.3 The Differences Between Bainite In Steels And Bainite In Ductile Iron

The bainitic microstructure of ADI shows some deviations from that of steels, because of the differences in the pre-heat treatment microstructure and the chemical composition.

The most important difference in between the microstructure of the ductile iron and steels is the spherical shaped graphites. These graphites determine the carbon content of the matrix by diffusing at the austenitization stage. Austenitization temperature becomes more important in ductile irons since it determines the rate of diffusion. Austenite from which the ferrite forms becomes enriched in carbon and further stabilized, the austenite in this matrix will decompose into ferrite plus carbide or, ultimately, ferrite plus graphite. The time that austenite is completely decomposed will vary depending on the alloy content of the matrix. It is known that most of the alloying elements like Mo, Cr, etc. will prolong this time.[10,8]

Ductile cast irons differ from most of the steels by the high concentration of C and Si. The presence of Si is necessary for the formation of graphite and also is responsible in some way for the stability of ferrite-austenite matrix as it evolves during austempering. The importance and affect of Si on austenitization will be discussed later.

2.2.3 Crystallographic Definition

It is believed that the mechanical properties of the ADI are directly related with the crystallographic texture formed during the transformation from austenite. Bain Strain is a critical key to understand the texture formed. Bain strain is the pure part of the lattice deformation, which for displacive transformation in steels converts austenite into ferrite or martensite [11]. The same stress also can be seen in the transformation of irons. Bain Strain deforms the matrix of the iron or steel by rotation in such a way that no plane or direction is rotated more than 11° . [11] That limiting angle defines the Bain Region. [16]

The change of orientation between bainitic ferrite and austenite is described with two classic relationships, “Kurdjumov-Sachs”(KS) and “Nishiyama-Wesserman”(NW). The difference in between these two orientation relationships is only 5.26° , which is in the Bain Region. [11]

King and Bell found that bainitic ferrite always exhibits an orientation which is close to KS or NW and well within the Bain Region. The figure 2.13 shows the Sterographic representation of KS and NW.

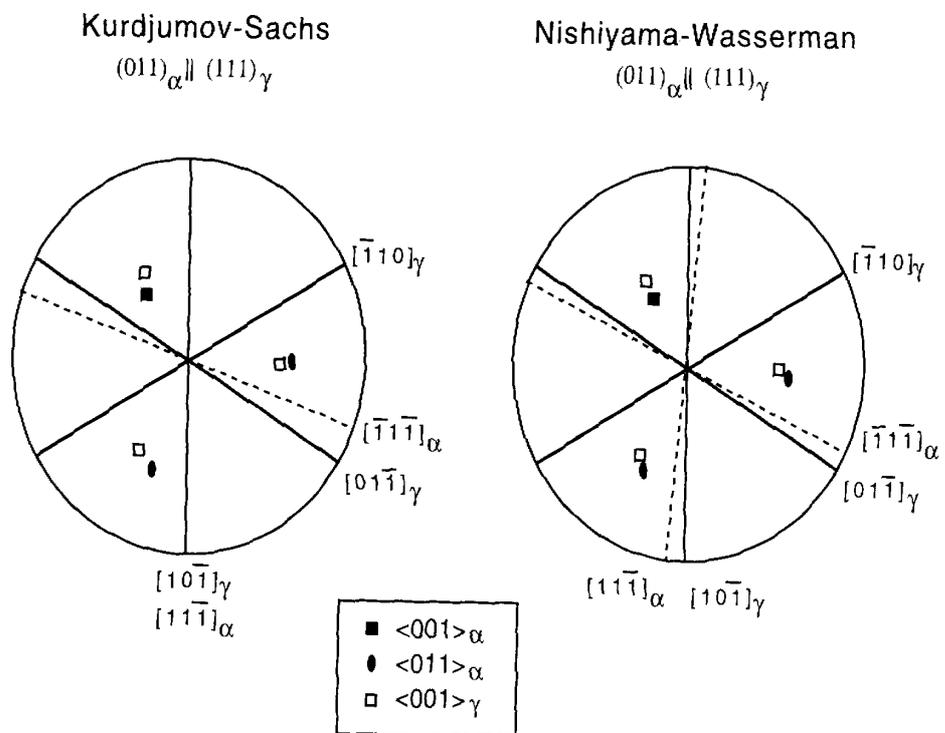


Figure 2.13. Stereographic representation of the KS and NW orientation relationship. [11]

2.3 Effect of Tin In Castings

The purpose of this part is to show the effects of Sn content on the mechanical properties the nodular cast iron.

Experimental work has proved that the addition of about 0.1 percent tin to flake graphite or nodular cast iron is extremely potent in promoting the formation of a fully pearlitic structure in the matrix.[12] This improves the wear resistance, reduces the dependence of hardness on section size and enhances machinability, thus reducing machine tool wear. The practice of making tin additions to cast iron is now widely accepted in industry throughout the world as a simple process which can be employed for the production of high quality castings in both mechanized and non-mechanized iron foundries.

Tin is added as pellets or stick to molten iron flowing from the cupola launder, or to the ladle of iron as it is filled. Since tin melts at 232°C it is instantly melted and dissolved in the molten cast iron which has a high liquid and solid solubility[1]. On the other hand, tin has a high boiling point, so that virtually no tin is lost by vaporization when it is added to the iron, even at these high temperatures of around 1400 °C.[12]

Unlike other pearlite promoters such as chromium, there is no tendency to form hard carbide particles or to increase the depth of chill, so that machining properties are unimpaired. The normal mode of use, therefore, is to determine the iron composition, which provides a fully pearlitic structure in the thinnest section to be machined.

Application of tin is essential for castings made in shell moulds where the nature induces fine graphite and associated surface layers of ferrite. The latter are virtually eliminated by prior alloying of the iron with tin.

Another property of tin is the stabilization of perlitic structure of iron at high temperatures and counteracts the natural tendency of iron to soften in prolonged high temperature applications. This softening comes from the decomposition to a ferritic structure and there is an associated dimensional growth of the castings, which is often unacceptable; this is reduced by the presence of tin. The pearlite stabilization is also of importance in maintaining a uniform hardness irrespective of the time that castings remain in the mould.

Since tin is not lost on remelting, scrap being recycled provides part of the necessary tin and the balance is added to casting. The actual percentage added might therefore only be, 0.06 per cent, but of course the tonnage of cast iron produced is very high. It may also serve as a convenient way of reusing tin bearing steel scrap.

Recent examples of the use of tin in cast iron include motorcar cylinder blocks, crankshafts, brake drums, transmission components, hydraulic lifting equipment and many other industrial applications.

2.4 Retained Austenite

There is difference between austempering methods mainly applied on steels and ductile cast irons. The treatment for steels aims to create a complete bainitic microstructure if possible. On the other hand the austempering of ductile cast iron aims in attaining the retained austenite content desired.

The presence of the retained austenite in steels especially in great amounts, is generally considered undesirable due to the fact that the martensitic transformation start (M_s) temperature of the retained austenite is above the room temperature.[13] For this reason, after the heat treatment process or while the working temperature decreases, the retained austenite tends to turn into martensite. Hard, brittle and non-tempered martensite forms in the regions where retained austenite is present. Because of this undesired phase transformation tensile stresses, which can in turn cause the rupture of the sample, develops immediately after heat treatment, during machining or at work.

Quenching the ductile iron in a salt bath after austenitizing brings a microstructure that consists of austenite and bainite. If the transformation period is short, the content of retained austenite will be low. Retained austenite is unstable and it can easily transform to martensite in time. The experiments have proved that a long transformation period can also reduce retained austenite content.

To increase the bainite content of the matrix, the temperature at which the transformation of bainite takes place is essential. The diffusion of carbon affects the amount of the austenite so at low transformation temperature the austenite content remains lower. At higher temperatures the austenite content also remains low because of the upper bainite and eventual pearlite.

In practice, the typical temperatures range for transformation is between 300 and 400 °C. Alloying and austenitizing can also have some effects on the amount of the retained austenite.

2.5 Effect of Composition on ADI

In many cases, the composition of ADI differs little from that of a conventional Ductile cast Iron. For both conventional Ductile Iron and ADI, during the determination of composition, and hence the raw materials, consideration should be given first to limiting elements which adversely affect casting quality through the production of non-spheroidal graphite, the formation of carbides and inclusions, and the promotion of shrinkage. Another point is the control of carbon, silicon and the major alloying elements that control the mechanical properties and microstructure. The effects of alloying elements are given in the following sections.

For light section castings (up to 10mm) a very rapid quench is usually sufficient to avoid the formation of ferrite and pearlite in even an unalloyed ductile cast iron. For castings of heavier section size selective alloying is required.

2.5.1 Effect of Carbon Content on ADI

Increasing the range of carbon content 3 to 4% enhance the tensile strength but has negligible affect on elongation and hardness.[3]

Carbon should be controlled within the range of 3.6-3.8% except when deviations are required to provide defect free casting.

2.5.2 Effect of Silicon Content on ADI

Silicon is one of the most important elements in ADI because it promotes graphite formation, decreases the solubility of carbon in austenite, increases the eutectoid temperature and inhibits the formation of bainitic carbide. Increase in the silicon content promotes the impact strength of ADI and lowers the brittle to ductile transition temperature. Silicon should be controlled closely with in the range 2.4-2.8%. [7]

2.5.3 Effect of Manganese Content on ADI

Manganese can be both beneficial and a harmful. It strongly increases hardenability. But during solidification, it segregates to cell boundaries where it forms carbides and retards the austempering reaction. As a result, for castings with either low nodule counts or section sizes greater than 19mm, manganese segregation at cell boundaries can be sufficiently high to produce shrinkage, carbides and unstable austenite. These microstructural defects and inhomogeneities decrease machinability and reduce mechanical properties. To improve properties and reduce the sensitivity of ADI to section size and nodule count, it is advisable to restrict the manganese level in ADI to less than 0.3%. [3]

2.5.4 Effect of Copper Content on ADI

Up to 0.85% copper may be added to ADI to increase the hardenability. It has no significant effect on tensile properties but increases ductility at isothermal transformation temperatures below 350°C.[10]

2.5.5 Effect of Nickel Content on ADI

Nickel may be used to increase hardenability of ADI up to 2%. For isothermal transformation temperatures below 350°C, nickel reduces tensile strength slightly but increases ductility. [10,3]

2.5.6 Effect of Molybdenum Content on ADI

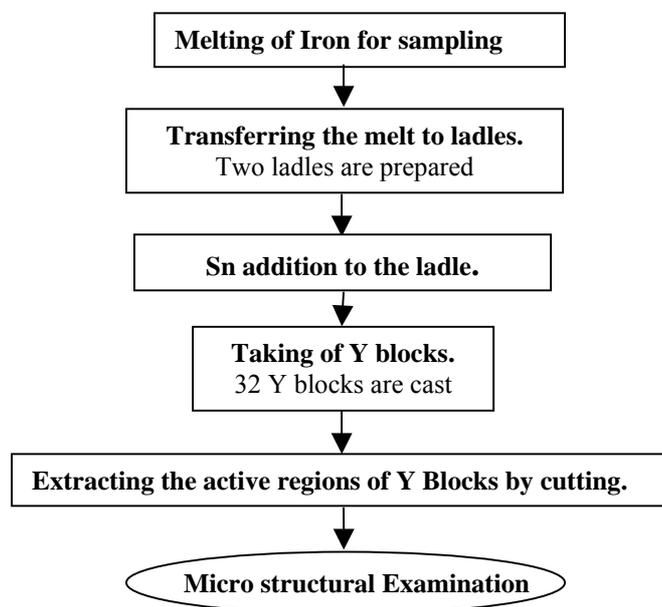
Molybdenum is the most potent hardenability agent in ADI and may be required in heavy section castings to prevent the formation of pearlite. However, both tensile strength and ductility decrease as molybdenum content is increased. This deterioration in mechanical properties probably caused by segregation of molybdenum to cell boundaries and the formation of carbides. The level of this element should be restricted by 0.2% in heavy section castings.[8]

CHAPTER 3

EXPERIMENTAL PROCEDURE

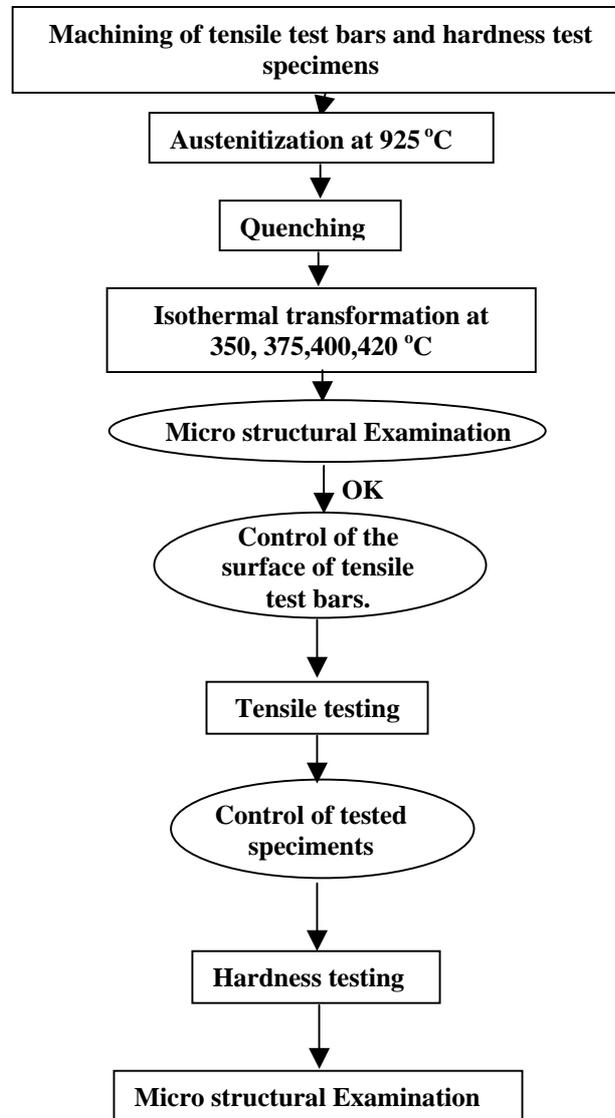
The following chart shows the experimental steps used in this thesis.

Chart 1 Experimental steps used in this thesis.



The steps of Chart 1 are continuing in page 32.

The steps of Chart.1



3.1 Material

Since the purpose of this study was to determine the effect of Sn content on the mechanical properties of the ductile iron. 8 specimens with different Sn contents were prepared. The chemical analyses of these specimens are given at the Table 3.1 and 3.2. All of the specimens were prepared at Erkunt Sanayi AŞ. The specimens were taken from standard 1in Y blocks and machined in the form of tensile test bars. Due to the increase in hardness after austempering, the tensile test bars were machined before heat treatment.

The dimensions of the standard Y blocks are given in figure 3.2. There are 3 main reasons for the use of the Y blocks incase of standard tensile test bars that are used for gray iron castings. These reasons are;

- 1) To prevent the formation of casting defects like shrinkage.
- 2) To take the slag from upper part of the casting.
- 3) To enable the full spherodization and complete formation of perlitic microstructure.

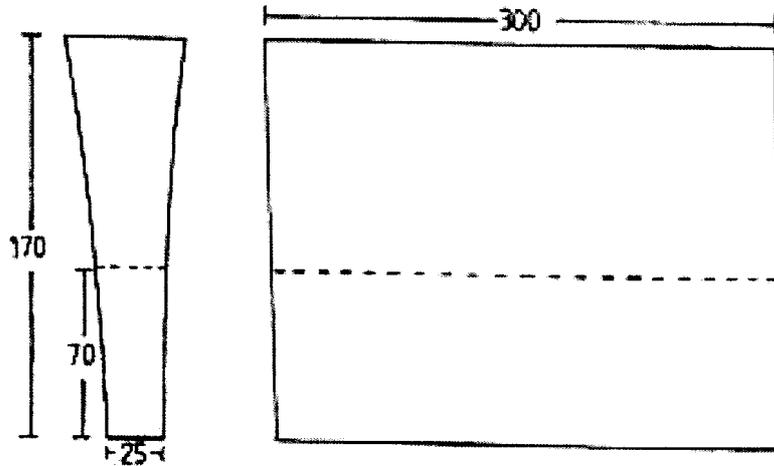


Figure 3.1 The dimensions of Y block.(all the dimensions are given in mm)

Sampling was done from two different ladles taken from the same furnace. The chemical analysis of the samples could be kept constant by taking ladles from the same furnace. Sn alloying of iron was done at the ladle. There were two main reasons for the use of two different ladles.

- To prevent the casting defects.
- To prevent the decomposition of Mg inoculant.

To prevent the casting defects sampling stopped when the temperature of melt decreased below 1360°C. The temperature was checked by the use of thermocouple. The decomposition of the Mg inoculant endangers the spherodisation . Studies showed that this decomposition starts after 3 or 5 minutes. Since the Sn addition is done in the ladle, this time interval only permits to prepare 4 different Sn contents for sampling.

Table 3.1 The composition of specimens.

Element	1.specimen	2.specimen	3.specimen	4.specimen
Si	2,52	2,52	2,52	2,52
Mn	0,162	0,162	0,162	0,162
P	0,028	0,028	0,028	0,028
S	0,013	0,013	0,013	0,013
Cr	0,011	0,011	0,011	0,011
Ni	0,024	0,024	0,024	0,024
Mo	0,002	0,002	0,002	0,002
Mg	0,0043	0,0043	0,0043	0,0043
Cu	0,215	0,215	0,215	0,215
Ti	0,022	0,022	0,022	0,022
C	3,63	3,63	3,63	3,63
Sn	0,016	0,027	0,041	0,055

Table 3.2 The composition of specimens.

Element	5.specimen	6.specimen	7.specimen	8.specimen
Si	2,55	2,55	2,55	2,55
Mn	0,162	0,162	0,162	0,162
P	0,028	0,028	0,028	0,028
S	0,014	0,014	0,014	0,014
Cr	0,012	0,012	0,012	0,012
Ni	0,022	0,022	0,022	0,022
Mo	0,002	0,002	0,002	0,002
Mg	0,0042	0,0042	0,0042	0,0042
Cu	0,211	0,211	0,211	0,211
Ti	0,022	0,022	0,022	0,022
C	3,61	3,61	3,61	3,61
Sn	0,1	0,16	0,21	0,26

3.2 Heat Treatment

For the determination of the ideal austenitization temperature a literature work was conducted. By the use of the graphs prepared from the results of pervious studies with the specimens that has similar chemical compositions, tensile strength and hardness values were obtained. The elimination of highest values revealed 925 °C as the austenitization temperature and 60 minutes as the austenitization time at which the best mechanical properties obtained. The austenitization of the specimens were done at 925 °C for 60 minutes.

After the determination of the austenitization temperature and time specimens with 8 different Sn content were quenched in the salt bath prepared with AS 135 salt . The quenching temperatures were 350 °C, 375 °C,400 °C,425 °C. At least 2 specimen from each Sn content were subjected to austempering heat treatment.

3.3 Mechanical Testing

Two mechanical tests were done on the samples according to the scope of the study.

- Tensile Testing
- Hardness Testing

3.3.1 Tensile Testing

The tensile testing specimens were obtained from the standard 1in Y-blocks and the specimen were machined to standard tensile strength test bars before the heat treatment. The dimensions of the specimens used are given in figure 3.2.

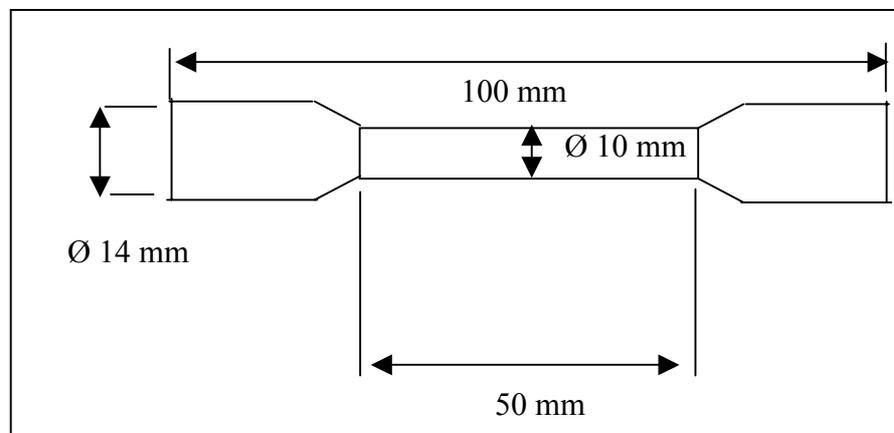


Figure 3.2 The dimension of the tensile test specimen

The tensile tests were done in Erkunt Sanayi AŞ using a 250 KN. Schenck Tensile Testing Machine. The ultimate tensile strength (UTS) and the amount of elongation were determined. Two specimens from each group were tested. All the specimens were checked visually for any casting defects.

For the determination of the amount elongation 10 mm. portion of the samples were marked before testing. After the test the portion was

re-measured and from the obtained data the percent elongation was determined. The UTS values were read from the computer of the tensile test machine.

3.3.2 Hardness Testing

Hardness samples were prepared from standard Y blocks. The hardness samples were at least 30 mm in diameter. Samples were isothermally transformed in the same bath with the tensile test bars to keep the same experimental environment. The samples prepared were subjected to Brinell hardness test by the use of the 2,5mm diameter ball indenter and a load of 185,5 kg.

6 hardness values were obtained from each sample and their average was taken. Some samples also crosschecked with the hardness values obtained from the tensile test bars

3.4 Metallographic Examination

All of the samples were subjected to Metallographic examination before the heat treatment process. The samples were taken from the active zone of the Y-blocks (From the same region also the tensile test bars were prepared). The samples were grinded with a group of emery papers changing from #220 to # 1200. After the grinding process the polishing process was done by the use of the rotary disk with the Al_2O_3 addition respectively. The etching operation of the samples is done with the %2 nital. After the preparation of the samples the spherodisation of the samples is checked by the use of Olympos microscope.

After the heat treatment process samples both from the tensile strength bars and the hardness test specimens were checked by the same process. The bainitic structure of the samples were checked with microscope.

3.5 X-Ray Diffraction Analysis

The X-ray diffraction process is done with use of the Philips diffractometer. The diffractometer is operating at 30kV and 10mA. The diffraction process is done from 20° to 120° . The scanning process is done with a speed of $1^{\circ}/\text{min}$.

Eight samples are examined with the X-ray diffraction to determine the amount of stabilized austenite according to the austempering conditions. The variation of the amount of stabilized austenite with the austempering condition is checked by the use of the results. The samples are prepared from the tensile strength test bars. During the preparation of the samples the cutter is continually cooled. The basic principle of this process is the difference in the crystal structures of the austenite and bainitic ferrite phases. As is known the ferrite phase has a Body Centered crystal structure (BCC) and the austenite phase has a Face Centered crystal structure (FCC) [15,16]. The FCC crystal structure reveals a peak of (110) while the BCC structure reveals a peak of (111). This difference in the orientation of the atoms affects the integrated intensities of the peaks at the diffractogramme. The calculation of the integrated intensities is done by the use of the milimetric paper. The closes peaks are chosen for the process. The formulas used in the calculation are listed below in an order and with a summery of the process.

The intensity of the Bragg reflection is calculated by the use of the fallowing equation:

$$I = KR/2\mu$$

In this formula K is a constant, μ is the linear absorption coefficient and R is represented by the following formula.

$$R = (1/v^2)[|F|^2 p(1 + \cos^2 2\theta) / (\sin 2\theta \cos \theta)](2e^{-2M})$$

In this formula

v : the lattice volume

F : the structure factor

P: Multiplicity factor

$2e^{-2M}$: Temperature factor.

For the austenite phase the equation can be modified in the following form.

$$I_\gamma = KR_\gamma X_\gamma / 2\mu_m$$

In this formula X_γ is the volume fraction of the austenite phase and μ_m is the linear absorption coefficient of the host material. Ferrite phase has the similar equation.

$$I_\alpha = KR_\alpha X_\alpha / 2\mu_m$$

By solving two equations together reveals the following equation.

$$I_\gamma / I_\alpha = R_\gamma X_\gamma / R_\alpha X_\alpha$$

By neglecting the graphite content in the structure and assuming that the matrix only consists of austenite and ferrite. The following equation can be obtained.

$$X_{\alpha} + X_{\gamma} = 1$$

This equation is the final equation used in all calculations.

CHAPTER 4

EXPERIMENTAL RESULTS

4.1 Mechanical Testing

4.1.1 Tensile Testing

Tensile tests were conducted on the austempered specimens. At least two specimens from each Sn content were tested. The tested bars were visually inspected in case of any casting defects. If the casting defects were observed the test would be repeated. Ultimate tensile strengths (UTS) and elongation values of the specimens were recorded. Recorded values are tabulated in table 4.1 and 4.2. The graphical representation of the UTS values versus Sn content is done in the figure 4.1. UTS values versus isothermal transformation temperature are plotted in the figures 4.2 to 4.3.

Figure 4.4 illustrates the change of percent elongation with respect to the Sn content of the specimens. The variation of percent elongation with the isothermal transformation temperature is shown in graphs from 4.5 to 4.6.

Table 4.1 The change in UTS values according to Isothermal Transformation Temperature and Sn % (MPa)

Sn content (%)	Isothermal Transformation Temperature(°C)			
	350	375	400	420
	ULTIMATE TENSILE STRENGTH (Mpa)			
0,016	921	847	812	772
0,027	952	882	872	797
0,041	748	924	901	823
0,055	981	943	911	856
0,1	1119	1115	1059	1004
0,16	1101	1025	1012	992
0,21	1048	1013	1007	983
0,26	1009	1005	991	958

Table 4.2 The change in elongation values according to Isothermal Transformation Temperature and Sn % (%)

Sn content (%)	Isothermal Transformation Temperature(°C)			
	350	375	400	420
	PERCENT ELONGATION			
0,016	5.3	6.1	7.4	8.4
0,027	5.1	5.6	6.8	8.5
0,041	4.8	5.4	6.8	8.1
0,055	4.9	5.3	6.5	7.4
0,1	4.2	5	6.3	7.4
0,16	5.0	6.0	7.1	8.6
0,21	4.9	6.1	7.5	8.7
0,26	5.1	6.2	8	8.9

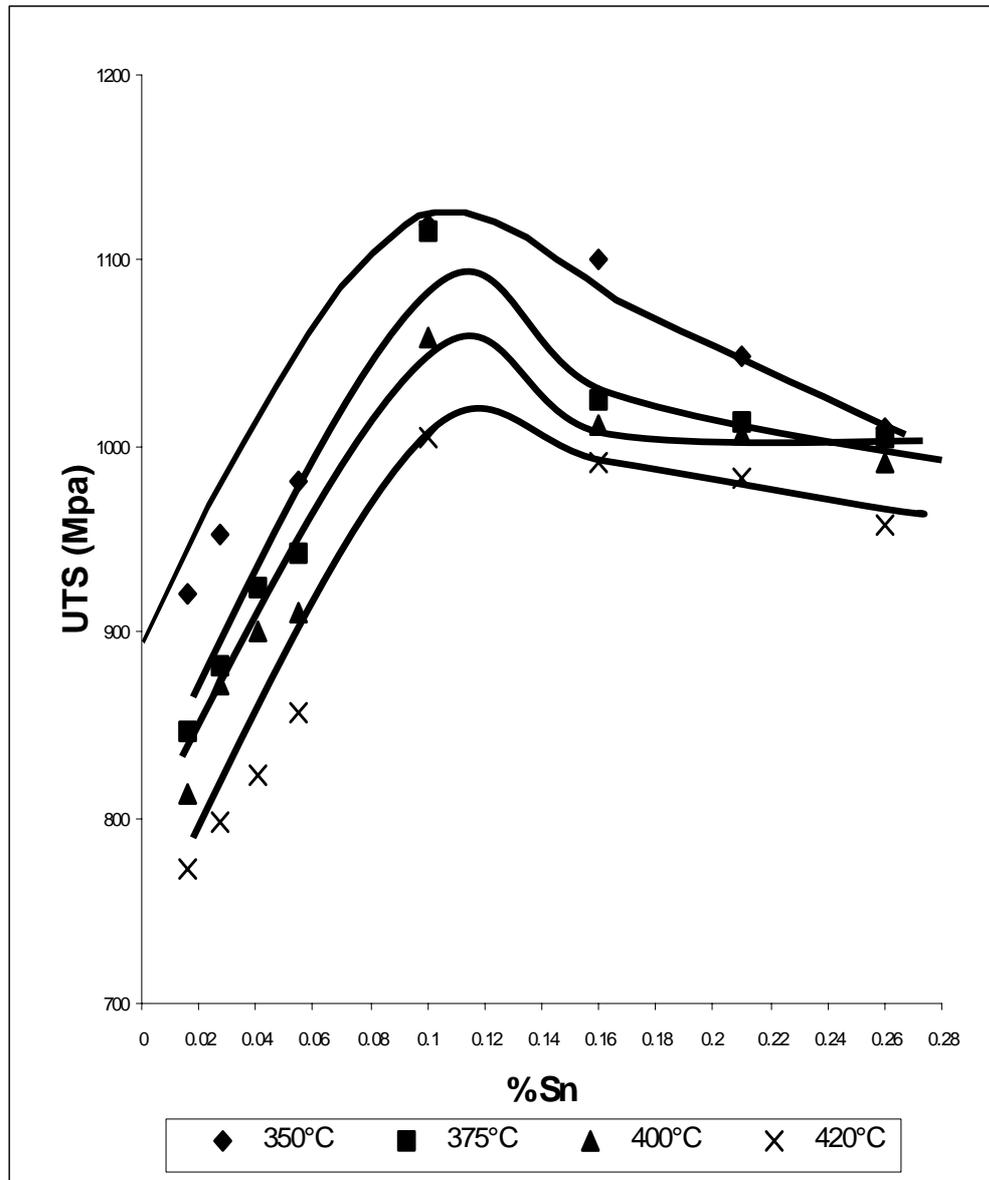


Figure 4.1 Variation of UTS with Sn% for Isothermal Transformation Temperatures .350°C, 375 °C, 400 °C and 420 °C.

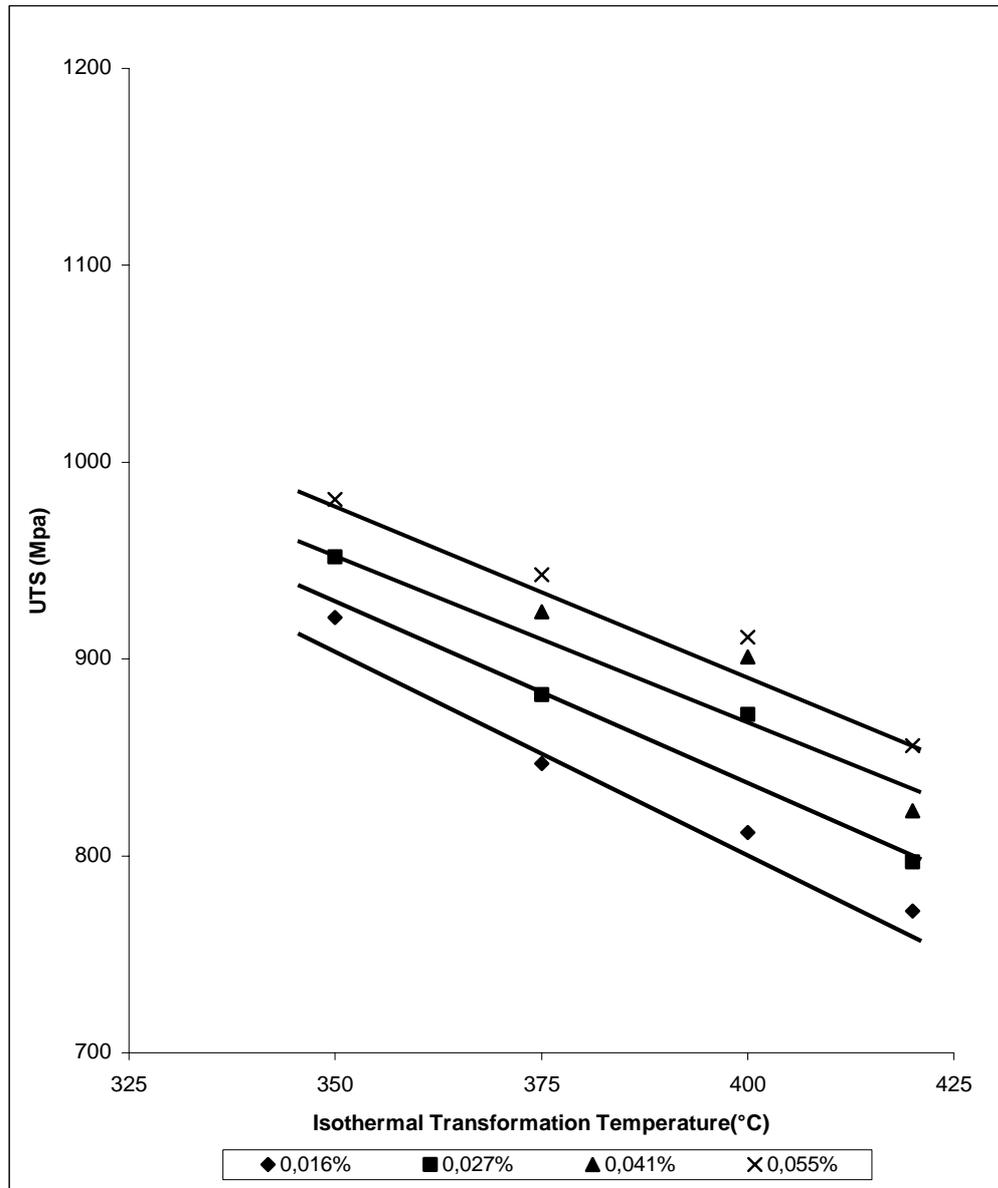


Figure 4.2 Variation of UTS with Isothermal Transformation Temperature for 0,016%Sn, 0,027%Sn, 0,041%Sn and 0,055%Sn.

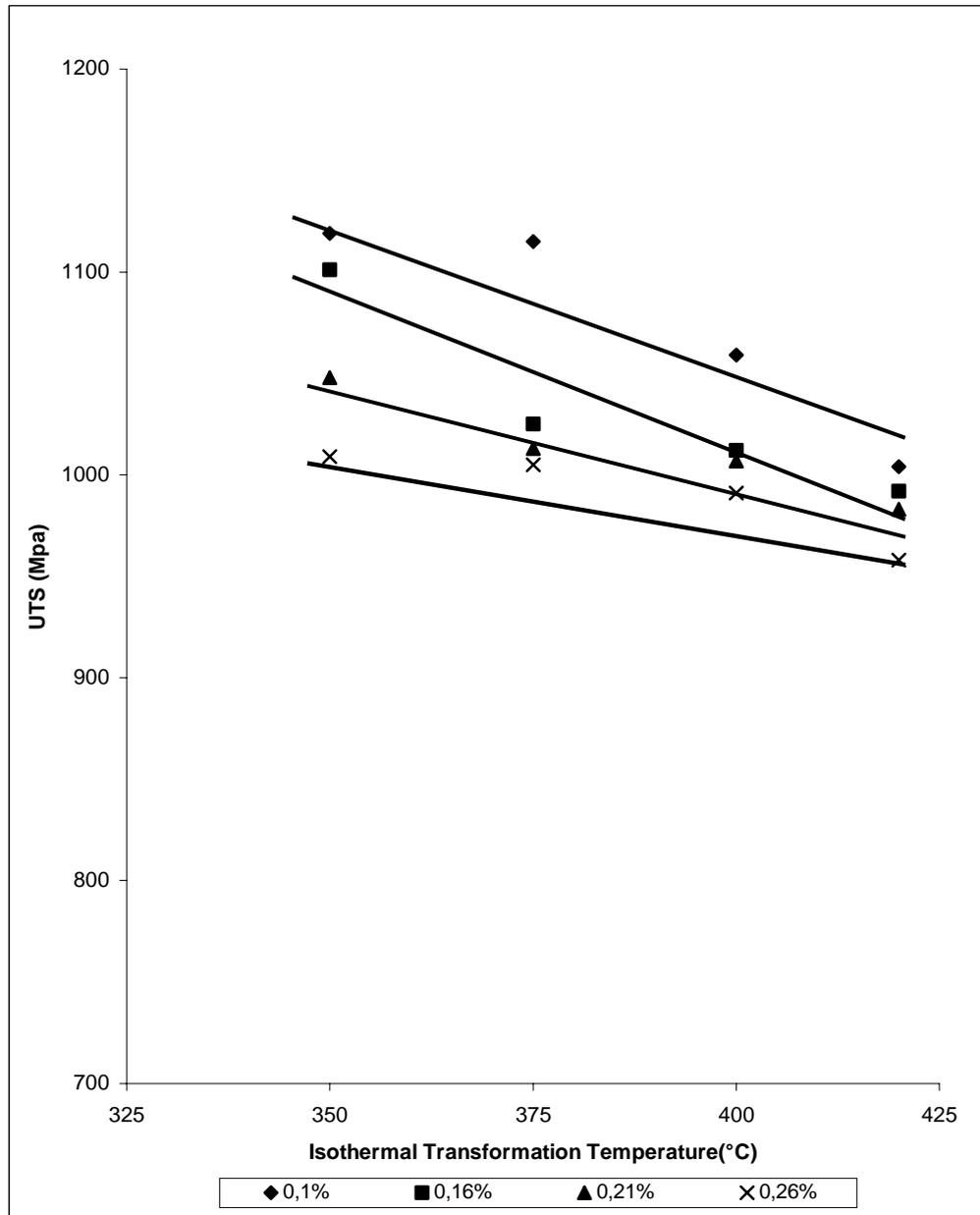


Figure 4.3 Variation of UTS with Isothermal Transformation Temperature for 0,1%, 0,16, 0,21%Sn, 0,26%Sn .

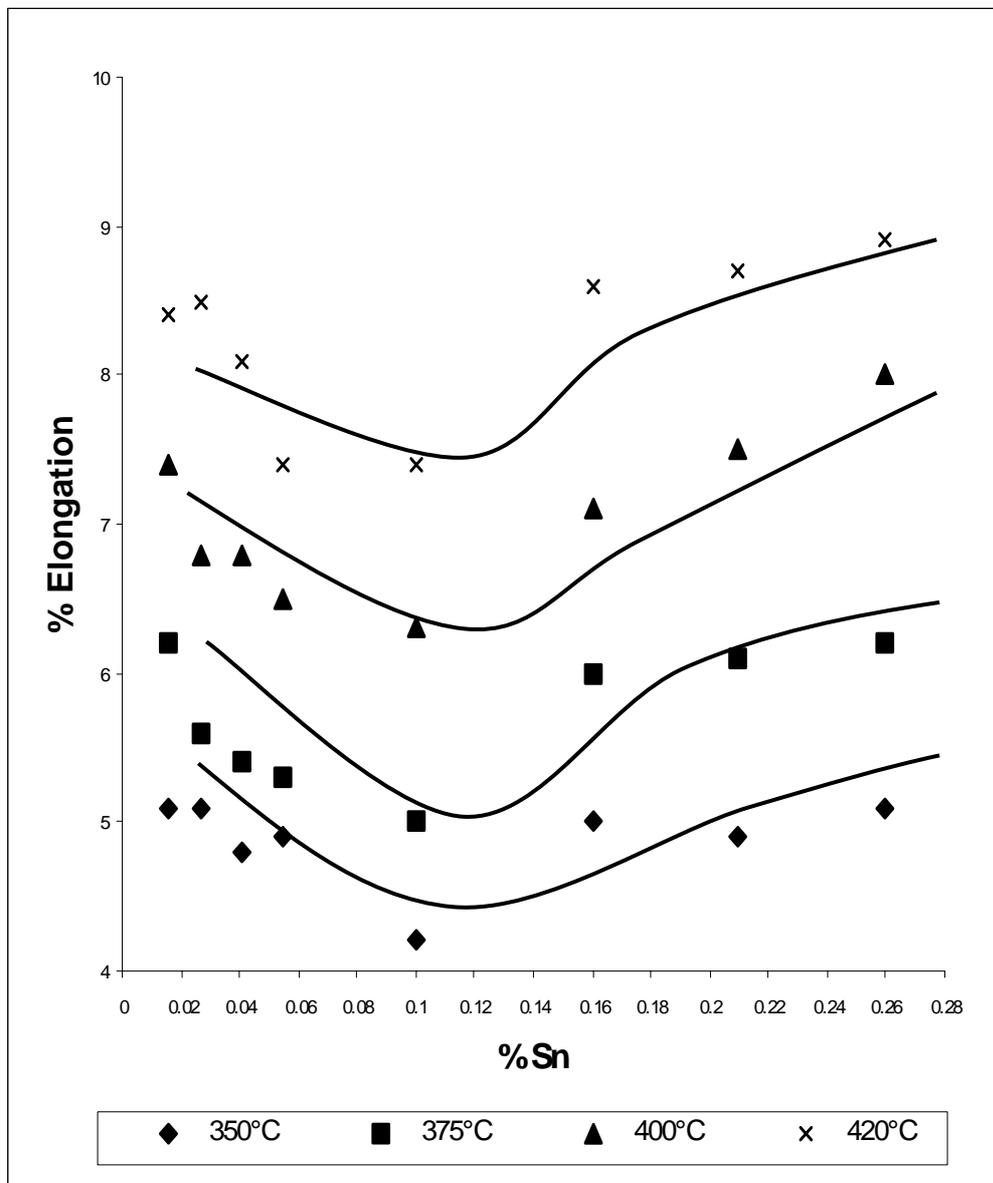


Figure 4.4 Variation of elongation values with Sn% for Isothermal Transformation Temperature 350°C, 375°C, 400°C and 420°C

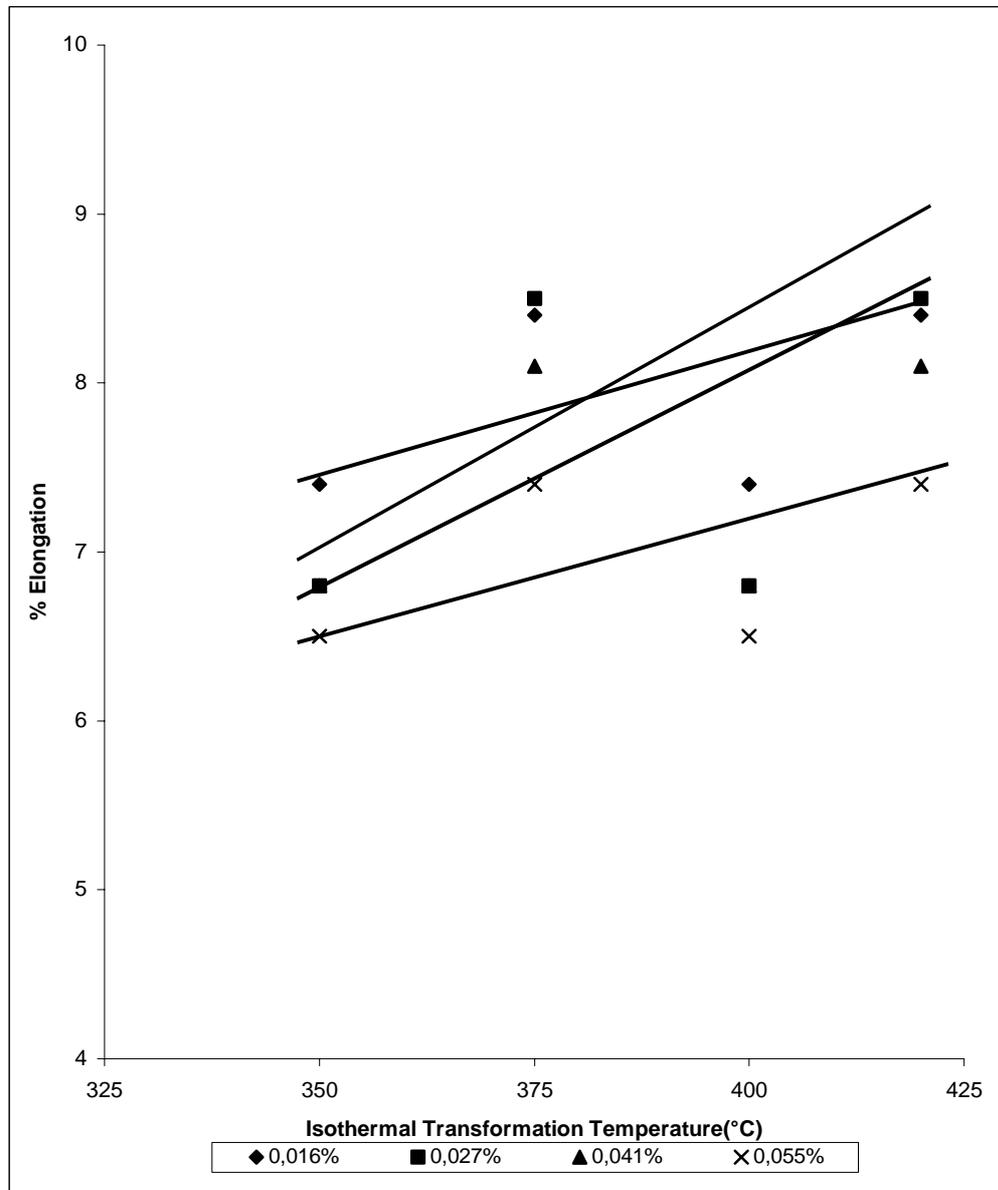


Figure 4.5 Variation of elongation values with Isothermal Transformation Temperature for 0,016 %Sn, 0,027%Sn, 0,041 %Sn and 0,055%Sn.

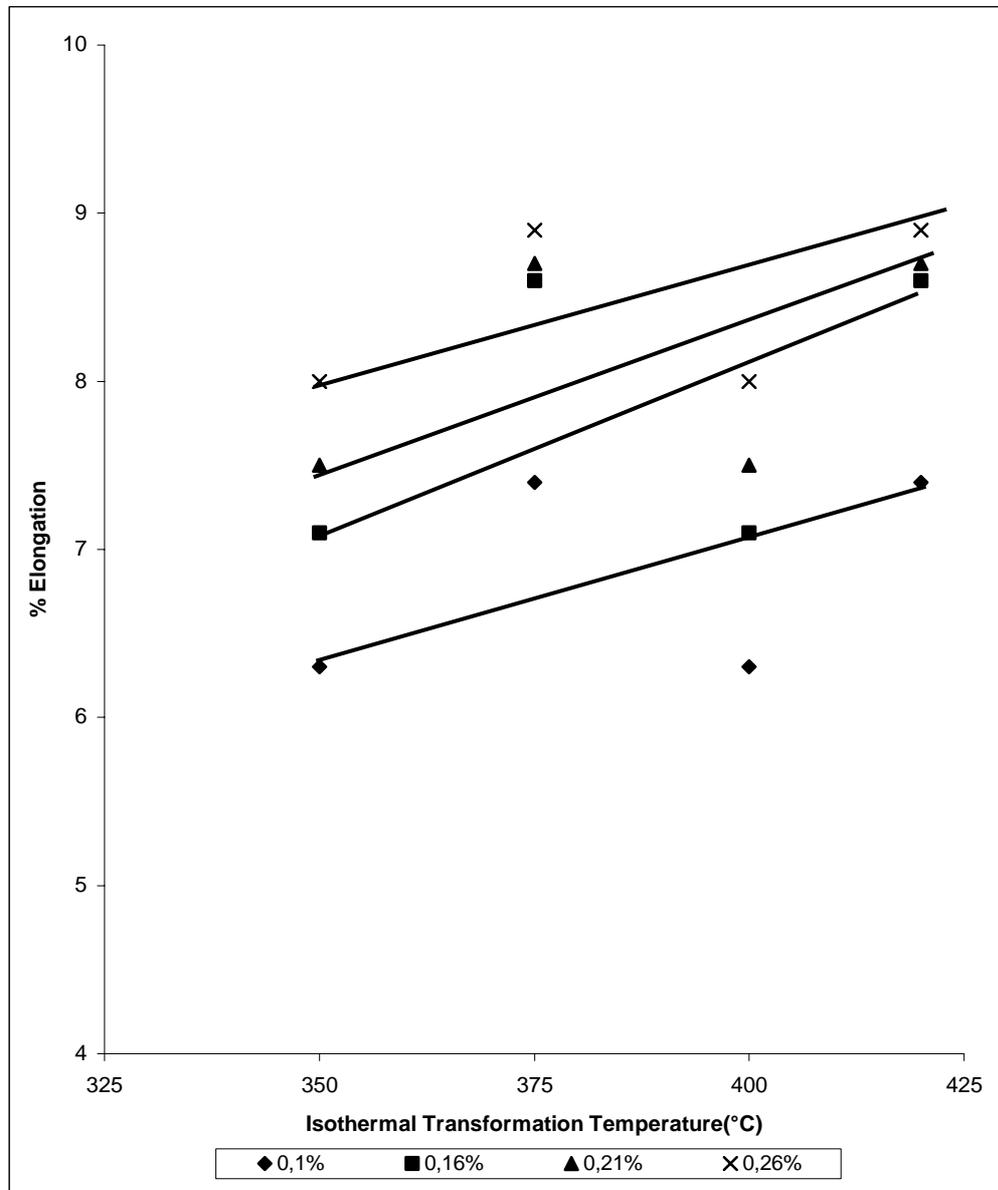


Figure 4.6 Variation of elongation values with Isothermal Transformation Temperatures for 0,1%Sn, 0,16%Sn, 0,21 %Sn and 0,26%Sn.

4.1.2 Hardness Test

Samples were prepared for the hardness test from each Sn content and isothermal transformation temperature. The samples were subjected to the Brinell Hardness test. Average 6 indentations were taken from each sample. In some cases to verify the results, some indentations were taken from the tensile test samples after tensile testing. The results are tabulated at in 4.3. For the ease of comparison the results are also graphically illustrated. Figure 4.7 illustrates the change in hardness values with Sn%. The change of the hardness values with isothermal transformation temperature is shown in figures from 4.8 to 4.11.

Table 4.3 The change in hardness values according to Isothermal Transformation Temperature and Sn % (Brinell Hardness)

Sn content (%)	Isothermal Transformation Temperature(°C)			
	350	375	400	420
	BRINELL HARDNESS			
0,016	323	331	319	302
0,027	325	321	322	306
0,041	331	329	351	307
0,055	345	331	327	311

Table 4.3 continues in page 53.

Continuous of Table 4.3.

0,1	371	364	328	314
0,16	363	368	317	312
0,21	354	341	327	310
0,26	341	320	310	305

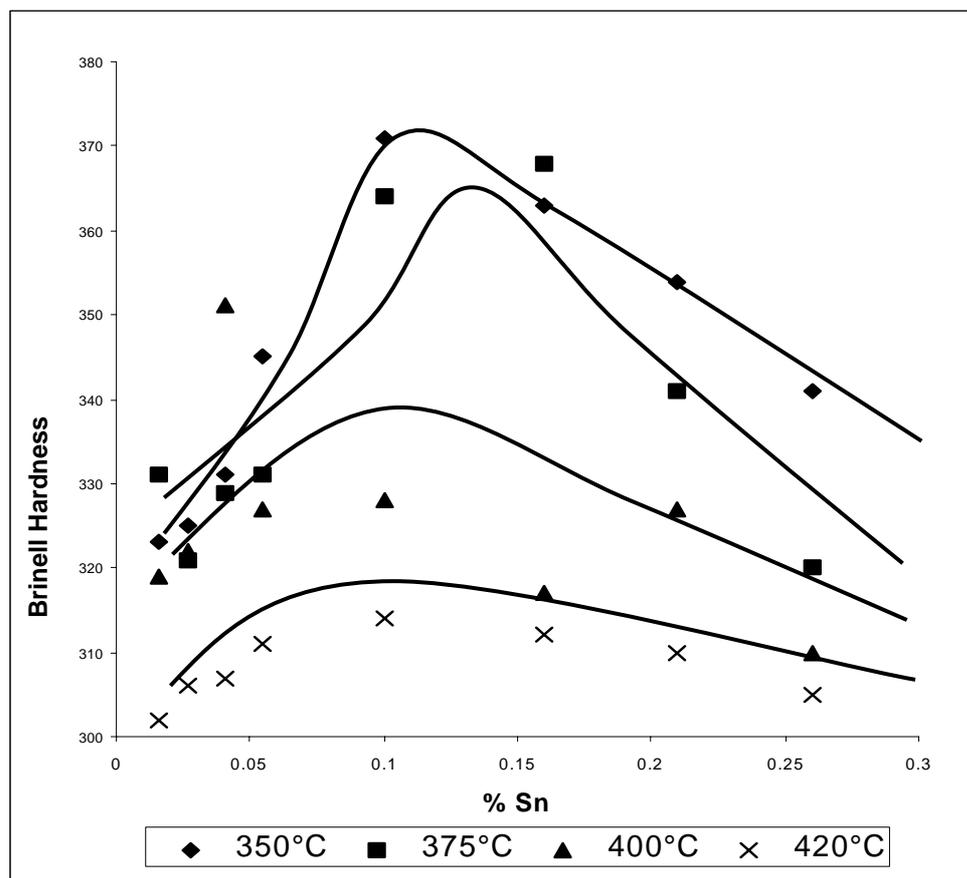


Figure 4.7 Variation of hardness values with Sn% for Isothermal Transformation Temperature . 350°C, 375°C 400°C and 420°C

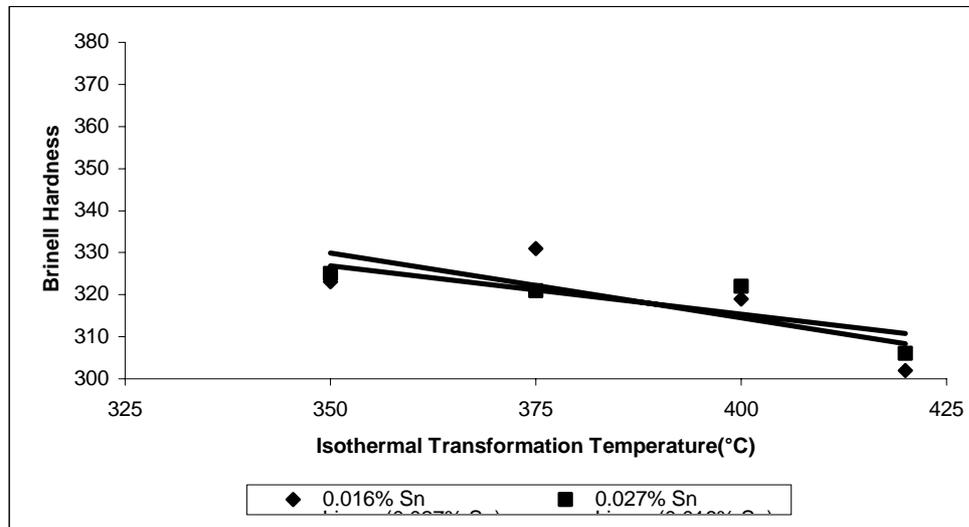


Figure 4.8 Variation of hardness values with Isothermal Transformation Temperature for 0,016 %Sn and 0,027 %Sn.

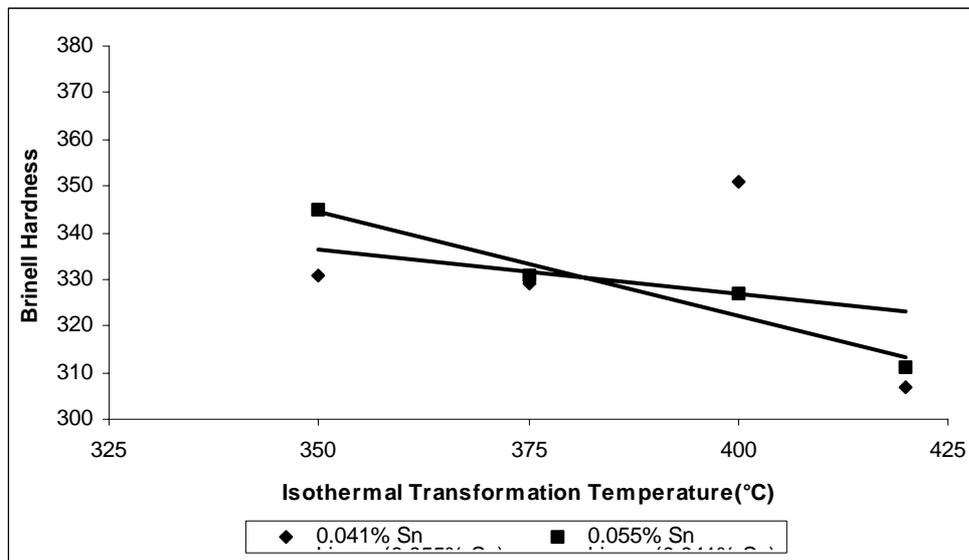


Figure 4.9 Variation of hardness values with Isothermal Transformation Temperature for 0,041 %Sn and 0,055 %

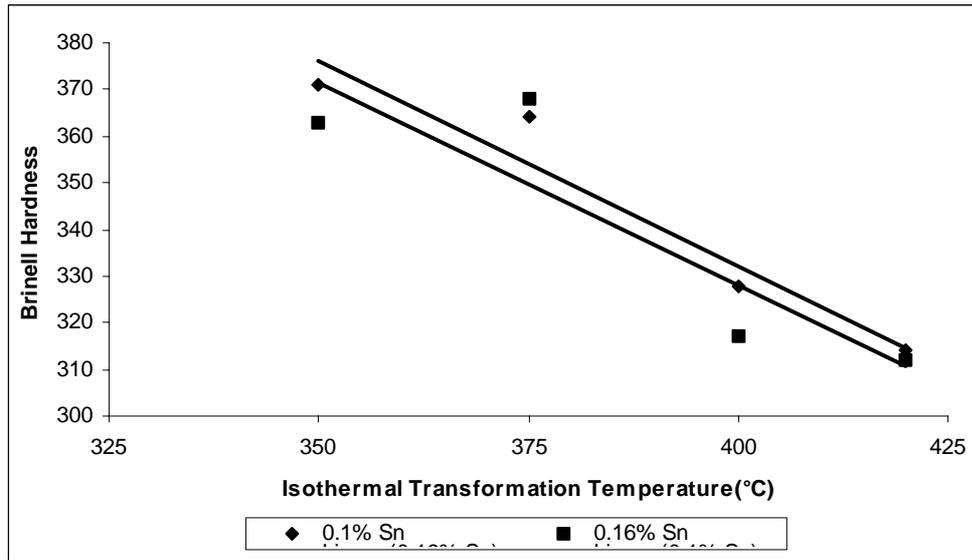


Figure 4.10 Variation of hardness values with Isothermal Transformation Temperature for 0,1 %Sn and 0,16 %

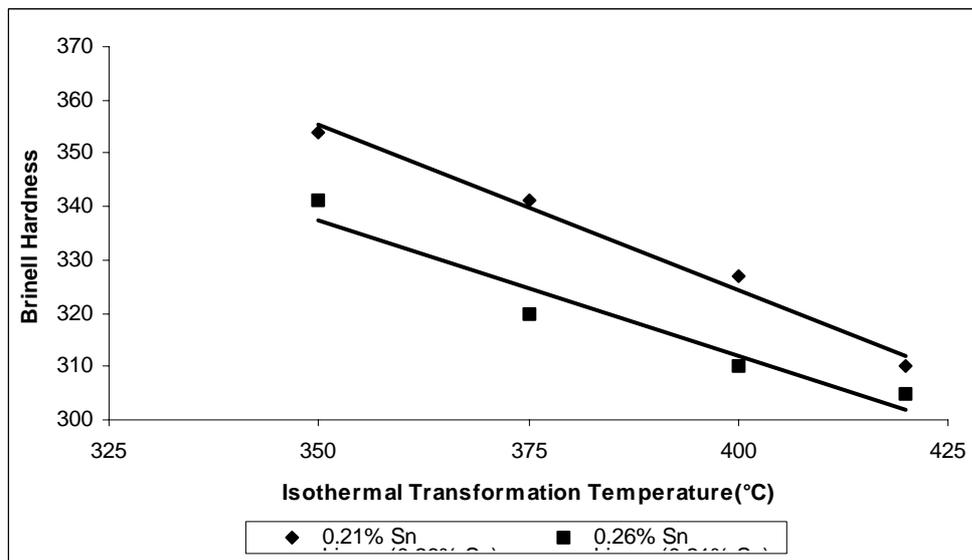


Figure 4.11 Variation of hardness values with Isothermal Transformation Temperature for 0,21 %Sn and 0,26 %

4.2 Microstructural Examination

From all the groups microstructural examination specimens were prepared. The photographs taken from these specimens are given at the Figures 4.36 to 4.64. The specimens are examined both before and after the heat treatment. At the microstructural examination before the heat treatment the percent of spheroidisation is controlled. To determine the success of austempering microstructures are checked after heat treatment.

Bainitic matrix structure can be seen at photographs. The amount of stabilized austenite increases with the increasing temperature. In addition to this by the decrease of the transformation temperature a decrease at the grain size is observed. This is possibly a cause of increase in hardness values and tensile strength.

4.3 X-Ray Diffraction Results

The specimens were checked by the use of the X-ray Diffractometer to determine the amount of retained austenite. The amount of retained austenite shows us the success of austempering heat treatment. Diffractometers are obtained from eight specimens with different isothermal transformation temperatures and 2 different Sn contents. As it can be seen in Table 4.4 the amount of retained austenite increases with increasing isothermal transformation temperature.

Table 4.4 The amount of retained austenite of some specimens

Isothermal Transformation Temperature	Sn Content	
	0,016	0,1
The amount of retained austenite are given in fractions		
350 °C	0,176	0,190
375 °C	0,212	0,198
400 °C	0,254	0,218
420 °C	0,302	0,241

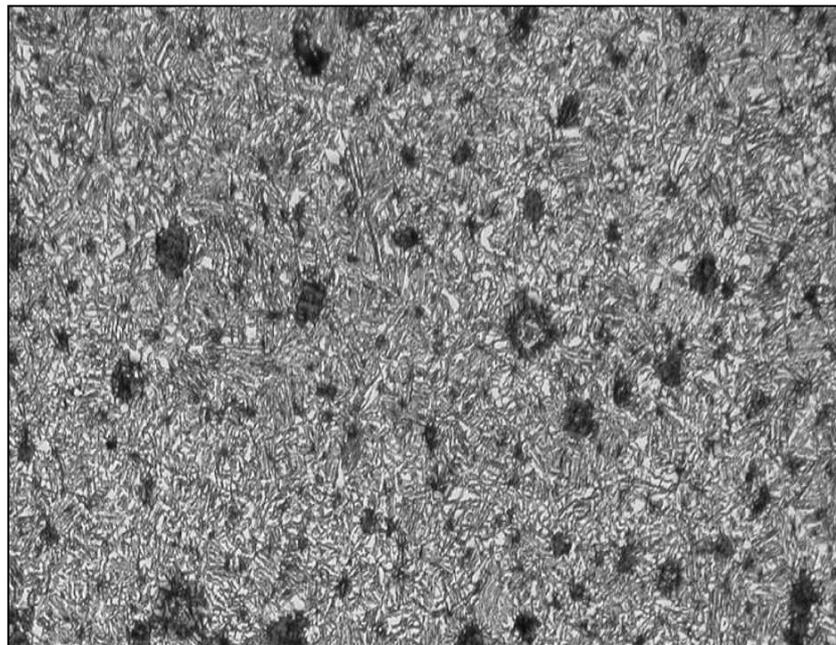


Figure 4.12 Microstructure of specimen with 0.016%Sn isothermal transformation temp. 420°C (200X)

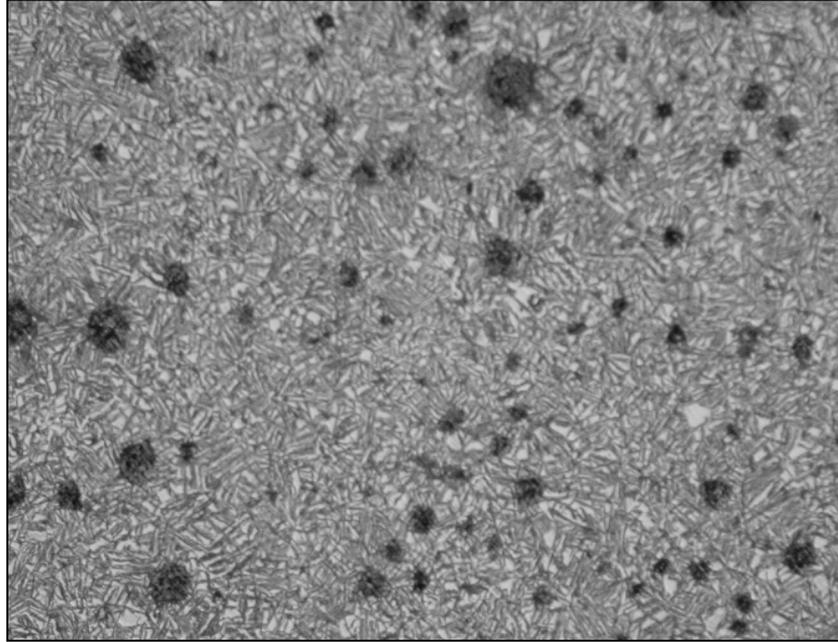


Figure 4.13 Microstructure of specimen with 0.26%Sn isothermal transformation temperature 420 °C (200X)

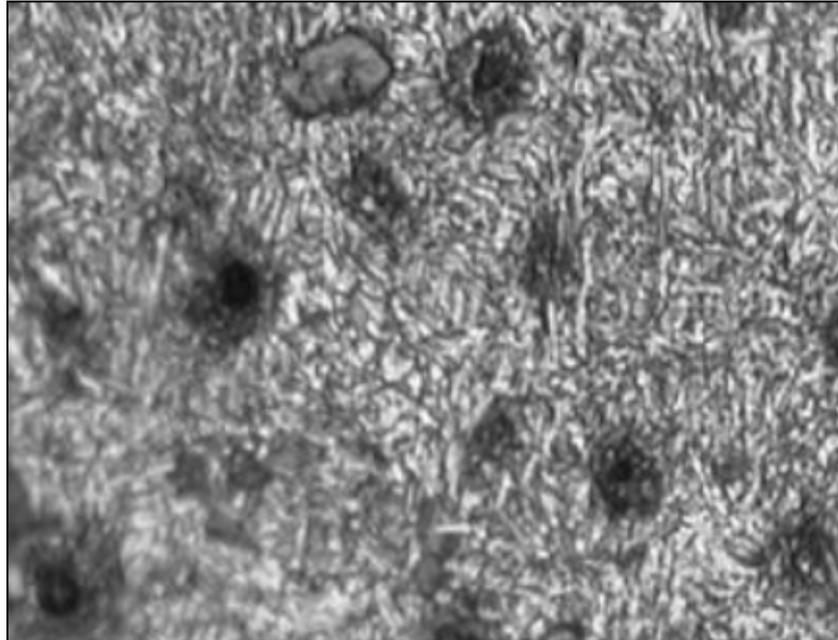


Figure 4.14 Microstructure of specimen with 0.055%Sn isothermal transformation temperature 420 °C (500X)

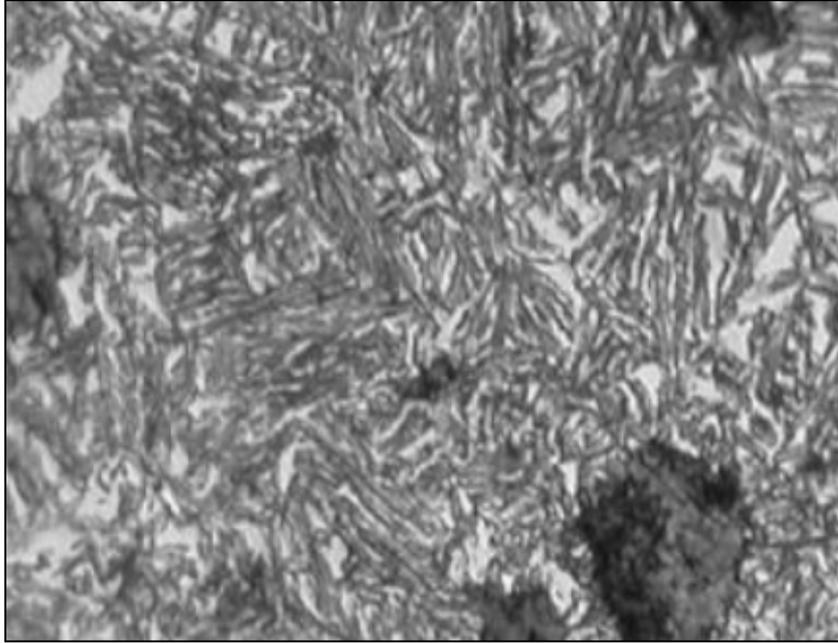


Figure 4.15 Microstructure of specimen with 0.21%Sn isothermal transformation temperature 420°C.(500X)

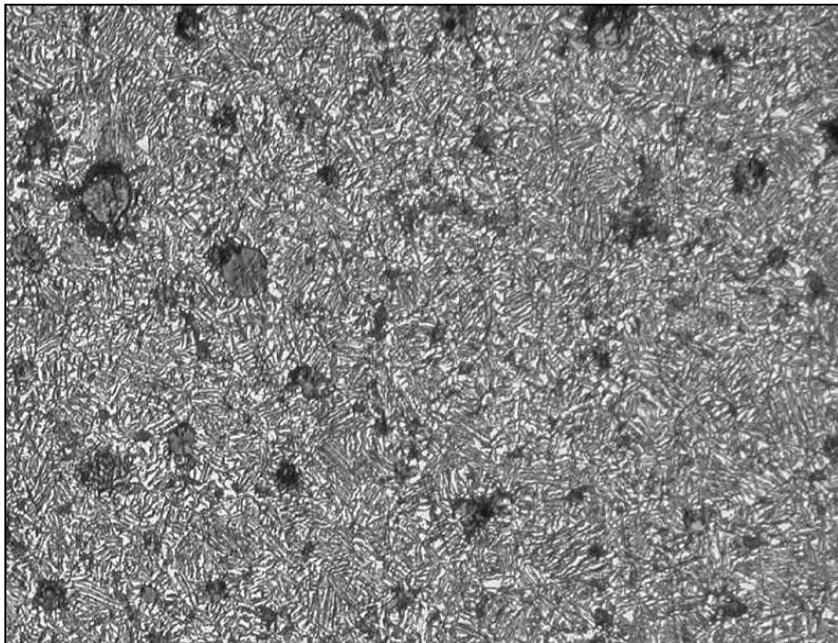


Figure 4.16 Microstructure of specimen with 0.016%Sn isothermal transformation temperature 400°C (200X)

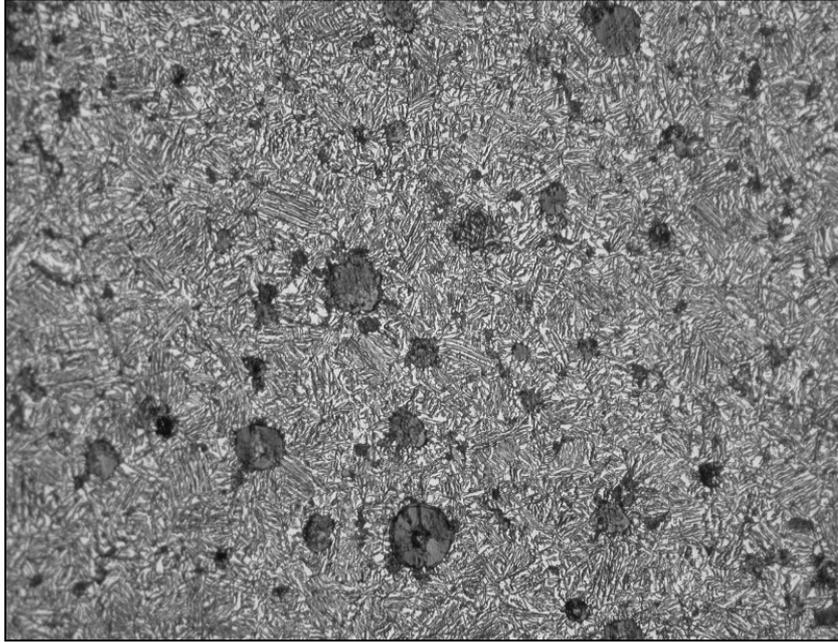


Figure 4.17 Microstructure of specimen with 0.26%Sn isothermal transformation temperature 400 °C (200X)

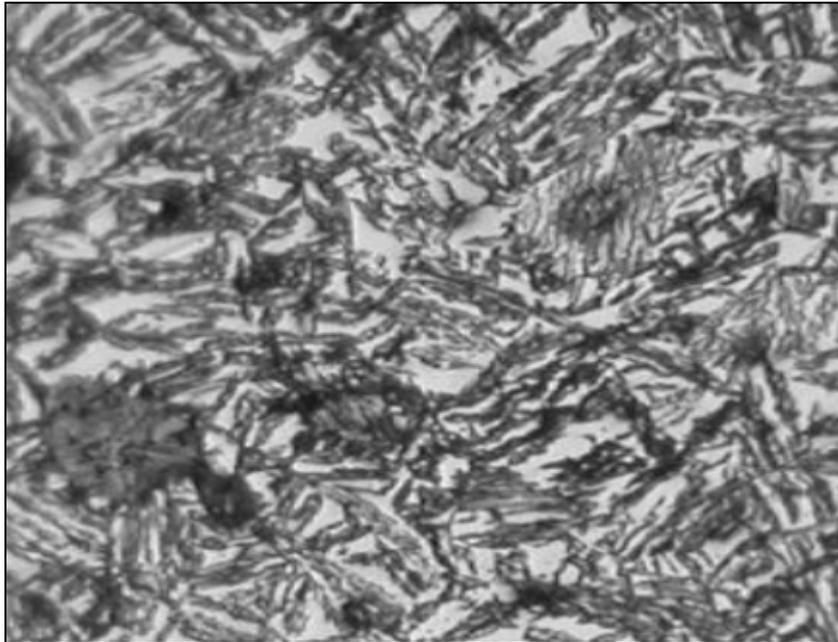


Figure 4.18 Microstructure of specimen with 0.016%Sn isothermal transformation temperature 400 °C (500X)

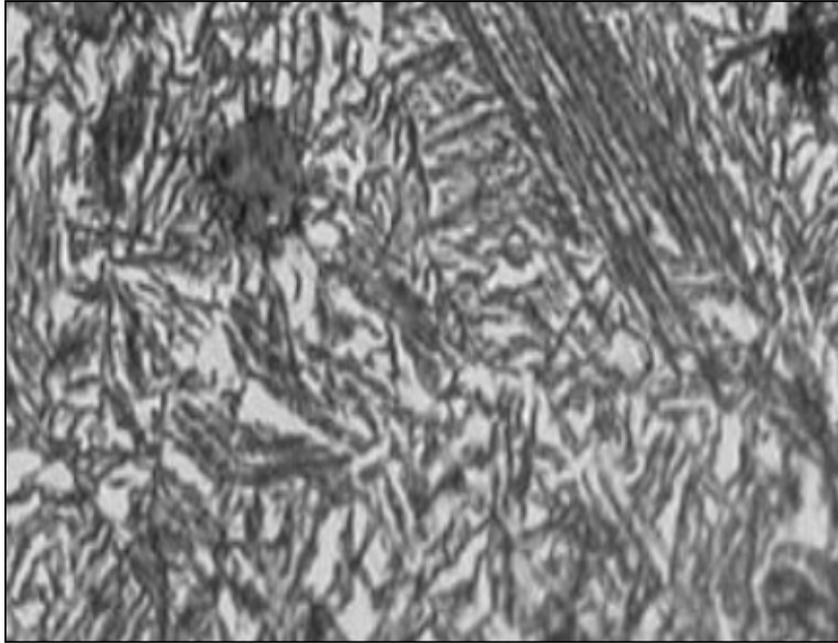


Figure 4.19 Microstructure of specimen with 0.055%Sn isothermal transformation temperature 400°C (500X)

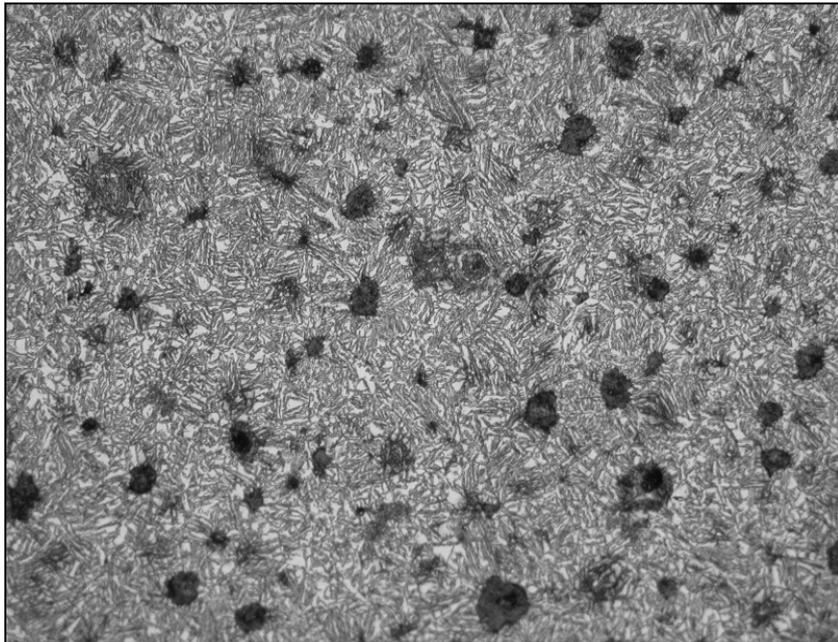


Figure 4.20 Microstructure of specimen with 0.016 %Sn isothermal transformation temperature 375 °C(200X)

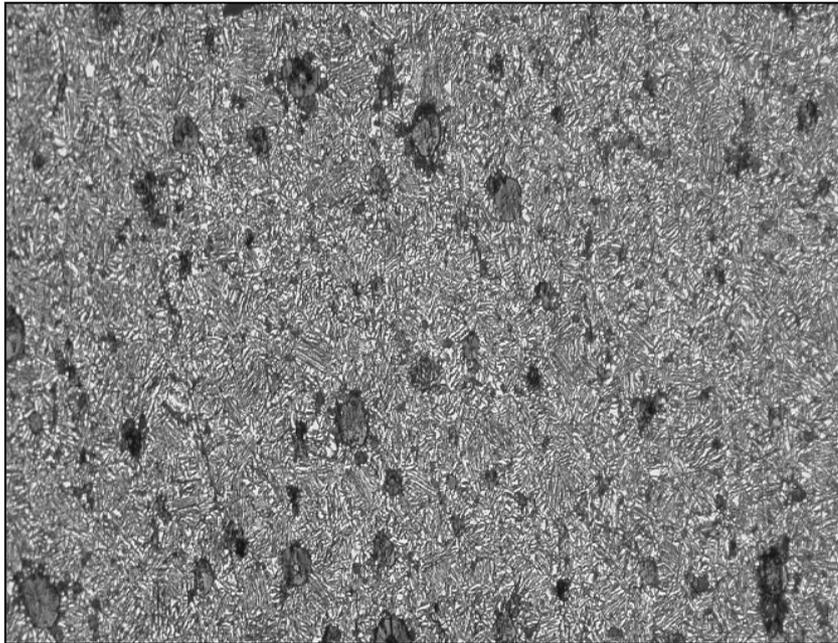


Figure 4.21 Microstructure of specimen with 0.16%Sn isothermal transformation temperature 375°C (200X)

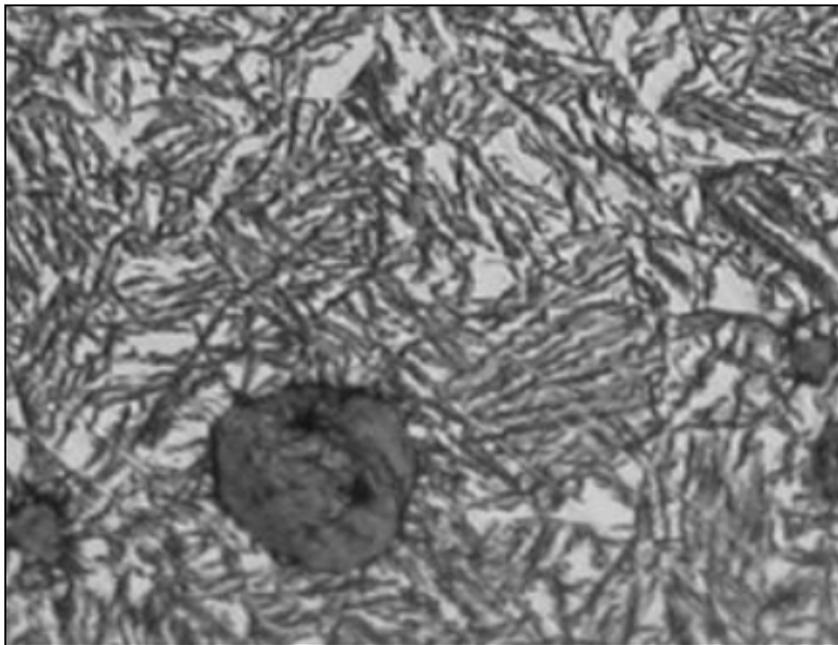


Figure 4.22 Microstructure of specimen with 0.027 %Sn isothermal transformation temperature 375 °C (500X)

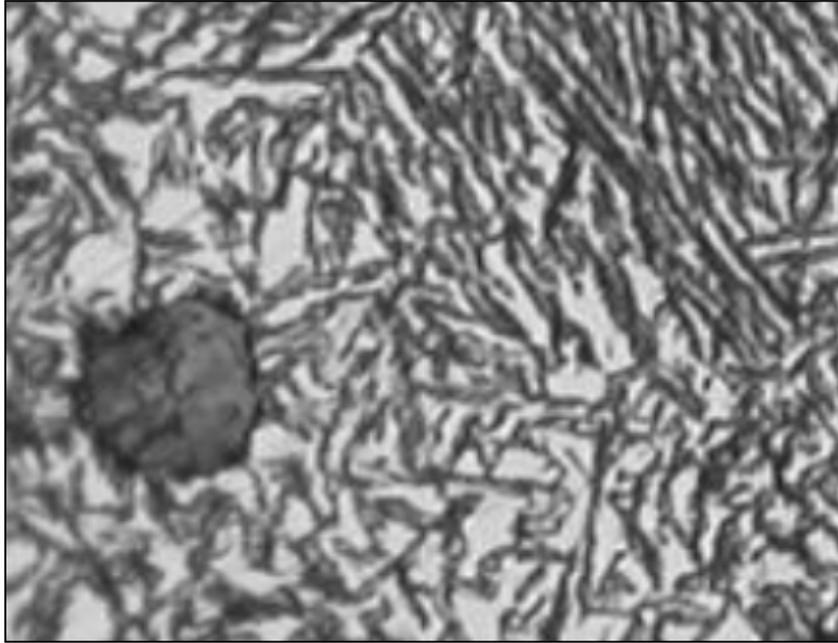


Figure 4.23 Microstructure of specimen with 0.041 %Sn isothermal transformation temperature 375°C (500X)

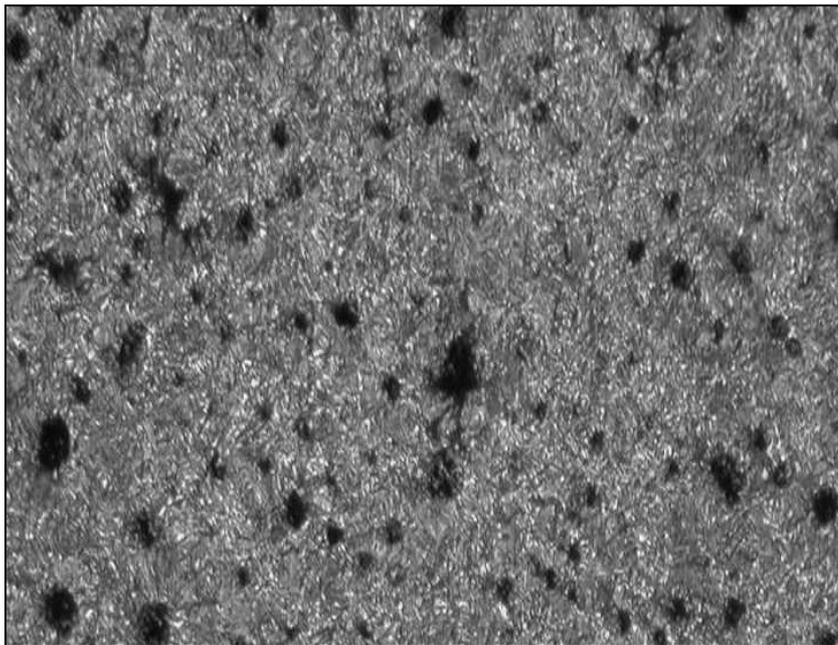


Figure 4.24 Microstructure of specimen with 0.041%Sn isothermal transformation temperature 350°C (200X)

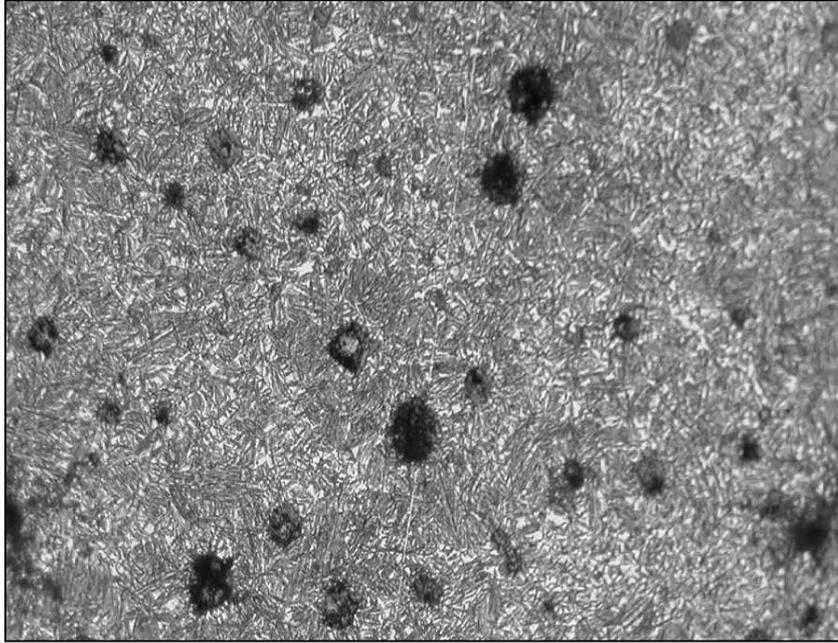


Figure 4.25 Microstructure of specimen with 0.21%Sn isothermal transformation temperature 350°C (200X)

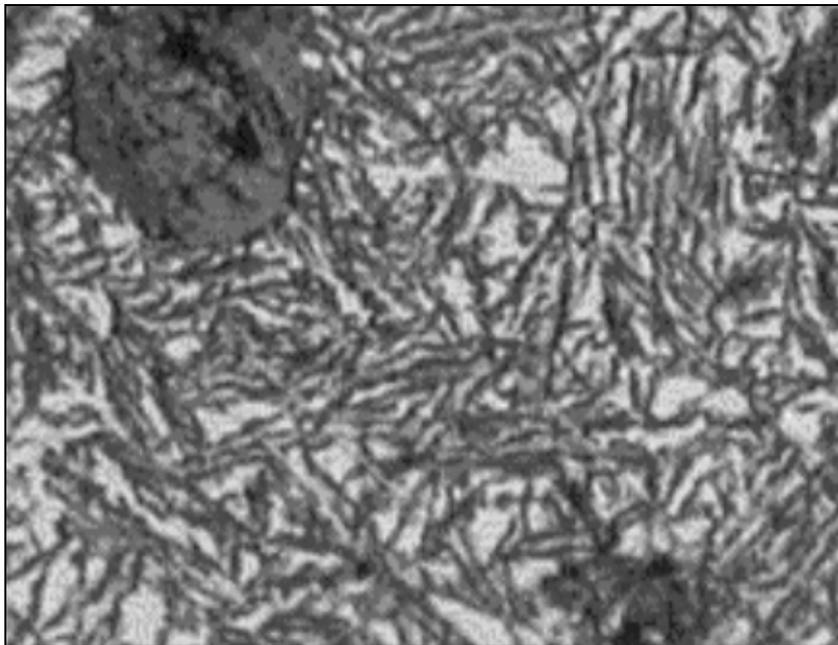


Figure 4.26 Microstructure of specimen with 0.027%Sn isothermal transformation temperature 350 °C (500X)

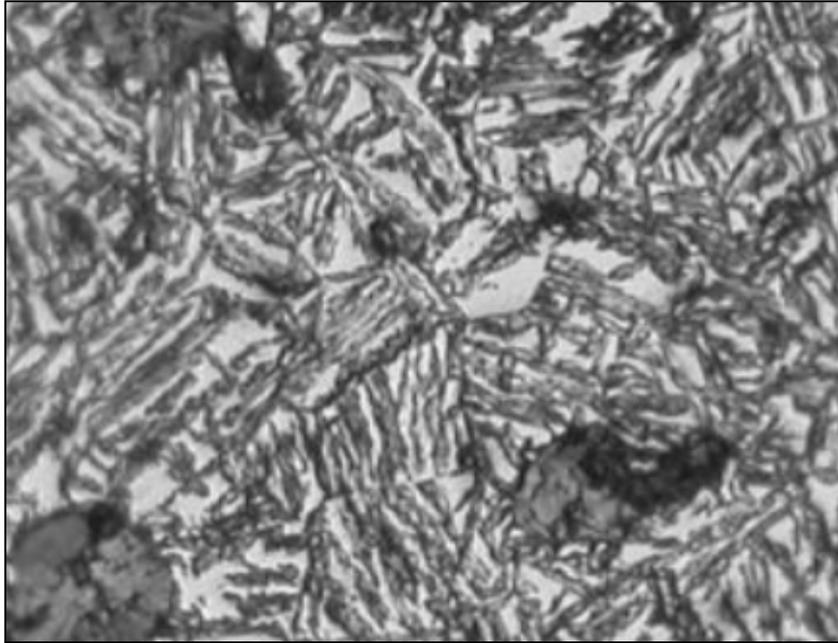


Figure 4.27 Microstructure of specimen with 0.1%Sn isothermal transformation temperature 350°C (500X)

CHAPTER 5

DISCUSSION

The main aim of this work was to investigate the possible effects of Sn on the mechanical properties of austempered ductile cast irons. The presence of Sn in these versatile alloys in the range of 0,025 to 0,1 % as a perlitic reaction stimulator was quite well known but it was not well known whether its presence in different quantities, in fact even in excess of 0,1% Sn (up to 0,26 Sn) would lead to any changes in the strength and ductility when these alloys were subjected to the austempering heat treatment. No related study as to the effect of Sn on mechanical properties could be detected in the literature.

The experimental procedure was quite straightforward; however a few difficulties were experienced during the course of the thesis. First of all the Y-Blocks obtained for the production of specimens contains too many casting defects which was over come by utilization by the utilization of larger hand ladles rather than the relatively small ladles used previously. The main reason why Y-block used for the preparation of the specimens is that the upper portion of the blocks (the upper 100 mm) serve as a metal feeder eliminating shrinkage at the active zone from were the samples are taken.

The second difficulty encountered was the fact that the tensile specimens kept on sliding along the gripping jaws because the specimens were relatively hard. These experiments were discarded and new treated specimens were prepared which offered no problems during the course of the tensile tests. Also a new special fixture was used to ensure gripping at the threaded ends.

The microstructural analyses presented in the thesis are not very elaborate. The lack of a quantitative metallographic analysis does not warrant detailed conclusions to be drawn as variations observed in the mechanical properties of the 8 different compositions tasted. However a microscopic examination of the specimens with increasing isothermal transformation temperatures reveals an increasing trend in the amount of retained austenite.

The reason for the increase in the amount of retained austenite with the increase in the isothermal transformation temperature is the decrease in the driving force for the bainitic reaction. This decrease is caused by the decrease in the temperature gradient. No appreciable changes in microstructure existed for different Sn percentages used in this thesis.

The UTS shows an increase up to 0,1% Sn and then seems to level off at all of the four temperatures tested; 350°C, 475 °C, 400 °C and 420 °C. Although it can also be said that there is a decreasing trend in the UTS after 0,1% Sn, this trend is so slightly that it is more reasonable to ignore it and not to accept this percentage as a peak point. The experimental procedure used in this thesis demonstrates the variation in the UTS with a change in Sn content for four different temperatures but does not point to the possible causes of this variation. Metallographic analysis reveals, without doubt, that the microstructures obtained in all cases are bainitic as expected, but of course without supporting data from quantitative microstructural analysis it

will not be appropriate to jump to conclusions as to the possible causes of this behavior.

The variation of UTS with temperature for all the 8 different Sn concentrations used in this thesis reveal that there is a decrease in the ultimate tensile strength of the specimens as we go upwards from low to high austempering temperatures. There is one exception here and that is the behavior observed at the Sn content of 0,041%. However this behavior would resemble the others if one of the four experimental points is considered to be in error. This is the point for the austempering temperature of 350°C. If this point is discarded then the decreasing trend would also be present here.

The changes in percent elongation versus Sn content at the four different transformation temperatures seem to be consistent with the results of the tests for the ultimate tensile strength. However in this case there apparently is a minimum around 0,1% Sn. If it is considered that strength and elongation normally exhibit opposite trends (ie. one increase while the other decrease and vice versa) this behavior is not surprising. However it must be stated that there are no sizable variations in the values of the percent elongation with changes in Sn content.

The peak of the hardness values mostly observed to be at the specimens with 0.1% Sn. Hardness values showed an increasing trend up to this level. Exceeding this Sn content caused a decrease in the hardness values. In addition, There is a decrease with increasing isothermal transformation temperature.

A variation in hardness with changing Sn content for the 4 different isothermal transformation temperatures is almost nonexistent. For

temperatures 350°C and 375°C a peak seems to exist at about 0,1%Sn but the differences are very small. Again a slight reduction of hardness occurs for all possible Sn concentrations with an increase in the isothermal transformation temperatures.

CHAPTER 6

CONCLUSION

- 1) The experimental findings of the thesis revealed that variation of Sn from 0,016% up to 0,26% causes certain changes in mechanical properties.
 - a) The UTS increases up to 0,1% Sn and then levels off.
 - b) A similar behavior is observed in the variation of hardness
 - c) Percent elongation decreases and falls to a minimum around 0,1% Sn. From this point on, a slight increase is observed.

In all the three cases above, the variations are not substantial.

- 2) More sizable variations are encountered with changes in the isothermal transformation temperatures from 350°C to 420°C. Strength and hardness increase while percent elongation decreases with decreasing temperatures.

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