# STRUCTURE PROPERTY RELATIONSHIPS IN Al-$SiC_p$ REINFORCED COMPOSITES

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**by**

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# Ms.Sc. THESIS EXAMINATION RESULT FORM

We certify that we have read the thesis, entitled **"Structure Property Relationships in Al-SiCp Reinforced Composites "**completed by Şefika KASMAN under supervision of Prof. Dr. Kazım ÖNEL and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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# ABSTRACT

In this study, an inquiry was applied to examine the effect of cold deformation on the hardness and grain growth of Al-5% Si-0.2% Mg (AlSi5) and Al-7%Si-0.7%Mg (AlSi7) matrix composites reinforced with SiC particles of 10, 20 vol. %. Before this process, The compo-cast ingots were cold deformed. The cold deformed samples were examined for micro-structural analysis, hardness and grain growth at different temperatures and times.

The porosity level in AlSi7 was high according to AlSi5 matrix. This problem was degreased with the extrusion and cold deformation process and the density of composites were increased. These porosity's was caused increasing of hardness and reducing of grain size of SiC particles.

The cold deformation and annealing process were carried out successfully. After cold deformation, the hardness was increased but the grain growth of SiCp were degreased. The recrytallization was applied to the cold deformed samples. With this annealing, the hardness was degreased but the grain size of SiCp were increased. The increasing of annealing time was caused reducing in grain size of SiCp.

# ÖZET

Bu çalışmada, soğuk deformasyon işleminin %10, %20 hacim oranında takviye Al-5%Si-2%Mg ( AlSi5) ve Al-7%Si-0,7%Mg (AlSi7) bazlı kompozitlerin sertliğine etkisi incelenmiştir. Bu işlemden önce döküm ingotlarına ekstrüzyon ve soğuk deformasyon uygulanmıştır. Deformasyon uygulanmış numunelere farklı sıcaklıklarda ve sürelerde tane büyümesi, sertlik ve mikroyapı analizleri uygulanıp incelenmiştir.

AlSi7 matrisinde porosite seviyesi AlSi7'e göre yüksek çıkmıştır. Oluşan bu poroziteler ekstrüzyon ve soğuk deformasyon işlemleriyle azaltılmıştır. Porozitenin yapıda hala bulunması sertlikte artışa ve tane boyutunda küçülmelere sebep olmuştur.

Soğuk deformasyon sonrası numunelere yeniden kristalleşme tavlaması uygulanmıştır. Bu tavlama işlemiyle birlikte sertlikte azalma tane boyutunda irileşme gözlenmiştir.

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# CHAPTER ONE
# INTRODUCTION

Interest has grown in recent years in ceramic particle reinforced metal matrix composite systems, which have been developed and implemented, in many engineering applications. SiC has been one of the most successful reinforcement materials for aluminium alloys because of its high strength, stiffness and hardness and it is chemically compatible with aluminium alloys up to 500 ° C. The Al-SiC composites (where SiC means SiC particles) have been characterised by their improved mechanical properties compared to properties of reinforced aluminium alloys. These improved properties include: (1) High specific modulus and strength, (2) resistance to wear, creep and fatigue crack initiation, (3) retention of properties at elevated temperatures. These properties make them very attractive for a wide range of applications in automotive and aerospace industries (Y.H.Kim, C.S.Lee & K.S.Han, 1991).

Several processes have been used to produce these composites. Production of metal matrix composites by casting techniques, make them very advantageous. By casting, the parts can be cast to near net shapes with low cost.

A few advantageous of these composites are homogenous structure and improved mechanical properties. When casting technique is used to produce composites, the reinforcing particles may react with the liquid metal, which can degrade the reinforcement. This is the case with unprotected SiC, which is thermodynamically

unstable in most aluminium alloys. The main reaction between liquid aluminium and SiC is,

$$4Al + 3SiC \rightarrow Al_4C_3 + 3Si$$

The formation of $Al_4C_3$ is bad from several points of view. The formation of $Al_4C_3$ particles, degrades the properties of the composites, obviously it degrades the reinforcement, and is accompanied by an increase in the Si level of the matrix alloy, since it involves the dissolution of SiC into Si and C. Composites containing $Al_4C_3$ are also more susceptible to corrosion (D.J. Lloyd, 1988)

In this study, Aluminium alloy metal matrix composites reinforced with varying fraction of SiC particles have been fabricated by compo casing technique. After casting, the ingots were extruded and then cold deformed. The cold worked specimens were annealed at different temperatures to study recrystallization and particle growth. The microstructures and hardness changes were investigated before and after annealing treatment.

# CHAPTER TWO
# LITERATURE SURVEY

## 2.1 COMPOSITE MATERIALS

### 2.1.1 Introduction

Many of our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional metal alloys, ceramics, and polymeric materials. This is especially true for materials that are needed for aerospace, underwater, and transportation applications. For example, aircraft engineers are increasingly searching for structural materials that have low densities, are strong, stiff, and abrasion and impact resistant, and not easily corroded. This is a rather formidable combination of characteristics. Frequently, strong materials are relatively dense; also, increasing the strength or stiffness generally results in a decrease in impact strength.

Material property combinations and ranges have been, and are yet being, extended by the development of composite materials. Generally speaking, a composite is considered to be any multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized. According to this **principle of combined action,** better property combinations are fashioned by the judicious combination of two or more distinct materials. Property trade-

materials. Property trade-offs are also made for many composites.

Composites of sorts have already been discussed; these include multiphase metal alloys, ceramics, and polymers. For example, pearlitic steels have a microstructure consisting of alternating layers of $\alpha$ ferrite and cemmentite. The ferrite phase is soft and ductile, whereas cemmentite is hard and very brittle. The combined mechanical characteristics of pearlite (reasonably high ductility and strength) are superior to those of either of the constituent phases. There are also a number of composites that occur in nature. For example, Wood consists of strong and flexible cellulose material called lignin. Also, bone is a composite of the strong yet soft protein collagen and hard, brittle mineral apatite.

A composite, in present context, is a multiphase material that is artificially made, as opposed to one that occurs or forms. In addition, the constituent phase must be chemically dissimilar and separated by a distinct interface. Thus, most metallic alloys and many ceramics do not fit this definition because their multiple phases are formed as a consequence of natural phenomena.

In designing composite materials, scientists and engineers have ingeniously combined various metals, ceramics, and polymers to produce a new generation of extraordinary materials. Most composites have been created to improve combinations of mechanical characteristics such as stiffness, toughness, and ambient and high temperature strength. ( Calliester, W. D. Willey Jr. J.& Sons Inc., 1999 Materials Science and Enginering and Introduction. )

### 2.1.2. Classification of Composites

When we define the composites, we sad that composite is a multiphase material. Many composite materials are composed of just two phases; one is termed the matrix. In that case defining the matrices can do classification of composites. Matrix is a

phase, which is continuous and surrounds the other phase, often called the dispersed phase. The properties of composites are a function of properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase. (Dispersed phase geometry) in this context means the shape of particles and the particle size, distribution, and orientation; these characteristics are represented in Figure 2.1. The classification of composites uses different terms. For example, Metal Matrix Composites(MMC), Ceramic Matrix Composites (CMCs), Polimer Matrix Composites (PMCs).

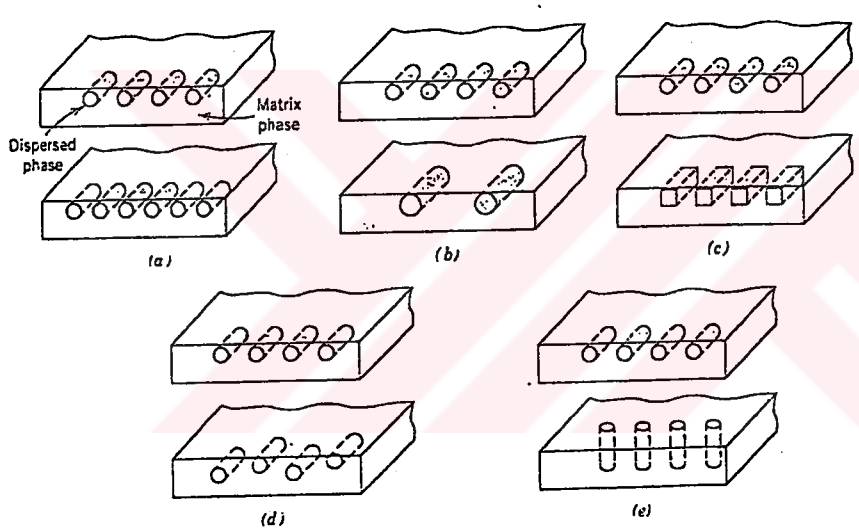


Figure 2.1 Schematic representation of the various geometrical and spatial characteristics of particles of the dispersed phase that may influence the properties of composites (a) concentration, (b) size, (c) shape, (d) distribution, and (e) orientation (From Richard A. Flinn and Paul K. Trojen, Engineering Materials and Treir Applications,4th edition. Copyright © 1990 by Jhon Wiley & Sons, Inc. Adapted by Permission of Jhon Willey & Sons, Inc.)

One simple scheme for classification of composite materials is shown in Figure 2.2, which consists of three main divisions–particle-reinforced, fiber - reinforced, and structural composites; also, at least two subdivision exist for each.

(Calliester , W. D., Wiley, Jr. J.& Sons Inc., 1999)

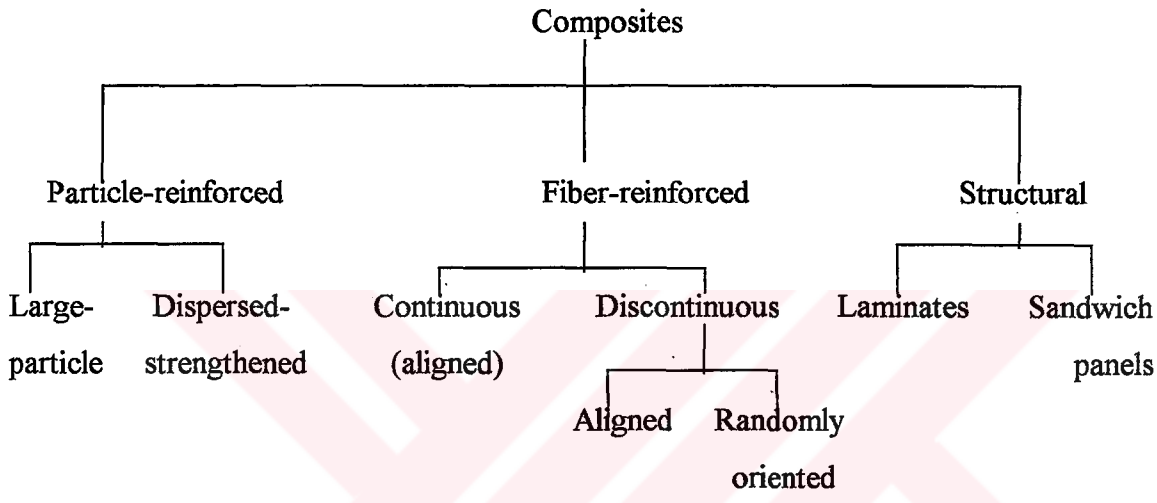


Figure 2.2 A classification of scheme for various composite types.

The dispersed phase for particle-reinforced composites is equiaxed (i.e., particle dimensions are approximately the same in all directions); for fibre-reinforced composites, the dispersed phase has the geometry of a fiber (i.e., a large length-to diameter ratio). Structural composites are combinations of composites and homogeneous materials.

## 2.2 METAL MATRIX COMPOSITES

### 2.2.1 Introduction

As the name implies, for **metal matrix composites (MMCs)**, the matrix is a ductile metal. These materials may be utilized at higher service temperatures than their base metal counterparts; furthermore, the reinforcement may improve specific stiffness, specific strength, abrasion resistance, creep resistance, thermal conductivity, and dimensional stability. Some of the advantages of these materials over the polymer matrix composites include higher operating temperatures, nonflammabilty, and greater resistance to degradation by organic fluids. Metal matrix composites are much more expensive than PMCs, and, therefore, their (MMCs) use is somewhat restricted.

The super alloys, as well as alloys of aluminium, magnesium, titanium, and copper is employed as matrix materials. The reinforcement may be in the form of particulates, both continuous and discontinuous fibres, and, whiskers; concerations normally range between 10 and 60 volume%. Continuous fibre materials include carbon, silicon carbide, boron, alumina, and the refractory metals. On the other hand, discontinuous reinforcement consists primarily of silicon carbide whiskers, chopped fibres of alumina and carbon, and particulates of silicon carbide and alumina. In a sense, the cerments fall within this MMCs scheme. In Table 2.1 are presented the properties of several common metal matrixes, continuous and aligned fibres reinforcement composites.

Some matrix reinforcement combinations are highly reactive at elevated temperatures. Consequently, composite degradation may be caused by high temperature processing, or by subjecting the MMC to elevated temperatures during service. This problem is commonly resolved either by applying a protective surface coating to the reinforcement or by modifying the matrix alloy composition.

Normally the processing of MMCs involves at least two steps: consolidation or synthesis (i.e., introduction of reinforcement into the matrix), followed by a shaping operation. A host of consolidation techniques are availed, some of which are relatively sophisticated; discontinuous fibre MMCs are amenable to shaping by standard metal forming operations (e.g., forging, extrusion, rolling).

Recently, some of automobile manufacturers have introduced engine components consisting of an aluminium alloy matrix that is reinforced with alumina and carbon fibres; this MMC is light in weight and resists wear and thermal distortion. Aerospace structural applications include advanced aluminium alloy metal matrix composites; boron fibres are used as the reinforcement for the Space Shuttle and continuous graphite fibres for the Hubble Telescope.

The high temperature creep and rapture properties of some of super alloys ( Ni and Co based alloys ) may be enhanced by fibre reinforcement using refractory metals such as tungsten. Excellent high temperature oxidation resistance and impact strength are also maintained. Incorporating these composites permit higher operating temperatures and better efficiencies for turbine engines.
(Calliester, W. D., Wiley, Jr. J. & Sons Inc., 1999)

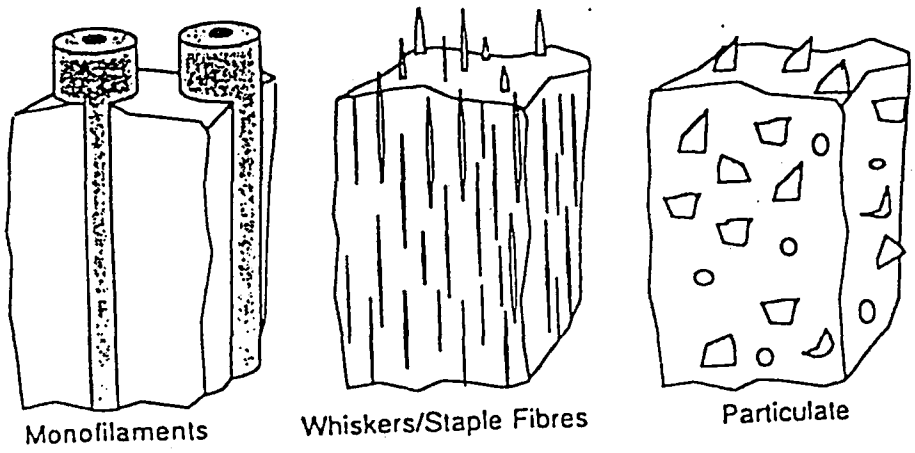


Figure 2.3 Schematic depiction of three types of MMC, classified according to the type of reinforcement.

### 2.2.2 Historical Background

Examples of metal matrix composites strecth back to the ancient civilisations. Copper bawls from Çayönü date back to about 7000BC and were made by a repeated lamination and hammering process, which gave rise to high levels of elongated non-metallic inclusion. The first composite materials were the dispersion hardened metal systems, developed from work in 1924 by Schmidt using mixtures of aluminium / alumna powders and led to extensive research in the 1950s and 1960s. The principles of precipitation hardening in metals date from the 1930s and were developed in the following decade.

More recent developments have brought the concept of metal matrix composites closer to engineering practice. And interesting example is provided by so called "dual phase" steels, which evolved in the 1970s.

Interest in fibrous metal matrix composites mushroomed in the 1960s, with effect directed mainly at aluminium and copper matrix systems reinforced with tungsten and boron fibres. Research on continuously reinforced composites warred during the 1970s, largely for reasons of high cost and production limitations. The continuing need for high temperature, high performance materials for various components in turbine engines have triggered a resurgence of interest, mainly directed towards titanium matrices. Discontuninuously reinforced composites have been rapidly developed during the 1980s, with addition focused on Al based composites reinforced with Sic particles and $Al_2O_3$ particles and short fibres.

The combination of good transverse properties, low cost, high workability and significant increases in performance over unreinforced alloys has made them the most commercially attractive system for much application. (Clyne, T.W & Withers, P.J. , 1998)

### 2.2.3 Compo-casting

Compo-casting as a process to produce aluminium based matrix composites has been studied and has received attention over recent years.

In the compo-casting, the ceramic particulates are added into a metallic alloy matrix at a temperature within the solid-liquid range of the alloyed, followed by vigorous agitation to form low viscosity slurry. This approach takes advantage of the fact that many metallic alloys behave like low viscosity slurry, when subjected to vigorous agitation during solidification. This behaviour, which has been observed for fraction of solids as high as 0.5, occurs during stirring and breaking of the solid dendrites during stirring, into spheroidal solid particles, which are then suspended in the liquid as fine-grained particulates. This unique characteristic of numerous alloys, known as thixotropy, can be

regained even after complete solidification by raising the temperature. This approach has been utilized in die casting of aluminium and copper base alloys.

The slurry characteristic of the matrix during stirring permits the addition of reinforcements during solidification. The ceramic particulates are mechanically entrapped initially and are prevented from agglomeration by the presence of the primary alloy solid particles. The particulates subsequently interact with the liquid matrix to effect bonding. Furthermore, the continuous deformation and breakdown of the solid phases during agitation prevents particulate agglomeration and setting. ( Ibrahim, I.A., Mohamed, F.A. & Lavernia, E.J., 1991)

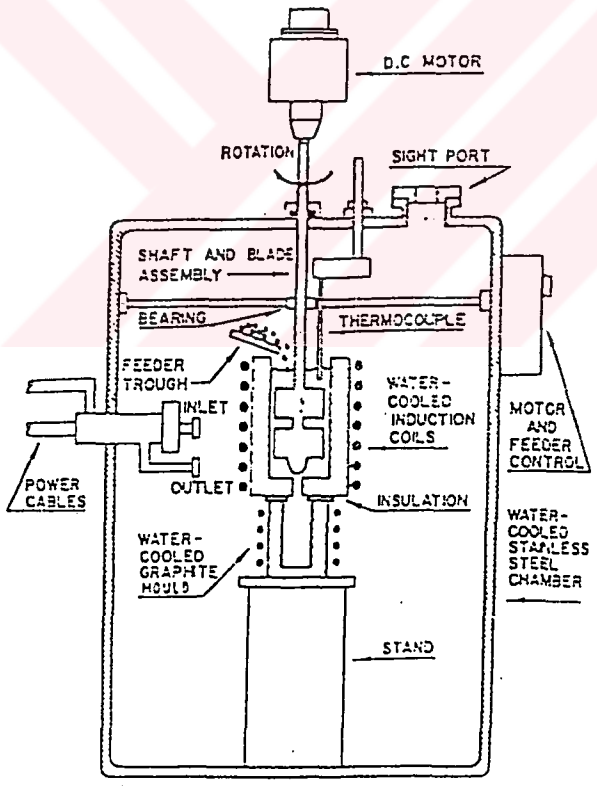


Figure 2.4 Schematic illustration of the compo casting apparatus.
( Hosking, F.M., Folgar, F., Wunderlin, R.& Mahrabian, R., 1982)

### 2.2.4 Oxidation of Reinforcement

Ceramic reinforcement materials are often coated generally with a metal. These coatings are applied for three purposes;

a) To protect reinforcement from damage in handling and from excessive reaction with the metal matrix
b) To promote wetting
c) To improve dispensability prior to addition to the matrix

But the coating method should be evaluated in terms of cost and performance required. For this reason an alternative to metallic coating is to coat the reinforcement with an oxide-fluxing compound to eliminate the oxide film often associated with molten matrix materials.

Oxidation can affect the behaviour of the particles during their incorporation. Oxidation of particle appears to have beneficial effect on the incorporation of the SiC particles in the semisolid state in Mg containing Al alloy matrix composites but it degrades the observed elastic modulus of the composite. The modulus degradation at a given oxidation temperature exhibits a linear function of reaction layer thickness. ( Mc Kimpson, M.G. & Scott, T.E., 1989)

### 2.2.4 Porosity

Porosity affects the mechanical properties of cast MMCs, significantly. Porosity could result in a casting from solidification micro shrinkage and evacuation during solidification. Melt processed particulate composites contain porosity that enters the melt

along with the reinforcement during stir mixing to generate the composite.

( Rozak, G.A., Lewandowski, J.J. & Wailace, J.F, 1992)

The porosity is generally located in the boundaries between SiC particles and matrix and is elongated with the hot deformation. There are three possible approaches to reduce porosity;

a) Environment control, such as application of vacuum

b) Use of baffles and improved design of stirrer

c) Maintaining suitable levels of process variables such as stirrer speed, size and position.

## 2.3 RECOVERY, RECRYSTALLIZATION AND GRAIN GROWTH

### 2.3.1 Introduction

As extensively outlined in literature, the application of plastic deformation to a polycrystalline metal specimen at temperatures that are low relative to its absolute melting temperature produces micro structural and property changes that include;

1) A change in grain shape,
2) Strain hardening
3) An increase in dislocation density

Some fraction of the energy expended in deformation is stored in the metal as strain energy, which is associated with tensile, compressive, and shears zones around the newly created dislocation. Furthermore, other properties such as electrical conductivity and corrosion resistance may be modified as a consequence of plastic deformation.

These properties and structures may revert back to the unworked states by

appropriate heat treatment (sometimes termed an annealing treatment). Such restoration results from two different processes that occur at elevated temperatures: **recovery** and **recrystallization,** which may be followed by **grain growth.**

### 2.3.2 Recovery

During recovery, some of the stored internal strain energy is relieved by virtue of dislocation motion (in the absence of an externally applied stress), as a result of enhanced atomic diffusion at the elevated temperature. There is some reduction in the number of dislocations, and dislocation configurations are produced having low strain energies. In addition, physical properties such as electrical and thermal conductivities and the like are recovered to their worked-worked states.

### 2.3.3 Recrystallization

Even after recovery is complete, the grains are still in a relatively high strain energy state. Recrystallization is the formation of a new set of strain - free and equated grain (i.e., having approximately equal dimension in all directions) that have low dislocation densities and are characteristic of the worked - worked condition. The driving force to produce this new grain structure is the difference in internal energy between the strained and unstrained material. The new grains form as very small nuclei and grow until they completely replace the parent material, processes that involve short - range diffusion.

Also, during recrystallization, the mechanical properties that were changed as a result of cold working are restored to their precold - worked values; that is, the metal becomes softer, weaker, and yet more ductile. Some heat treatments are designed to allow recrystallization to occur with these modifications in the mechanical characteristics. Recrystallization is a process the extent of which depends on both time and temperature.

The influence of temperature is demonstrated in Figure 2.6, which plots tensile strength and ductility (at room temperature) of a brass alloy as a function of the temperature and for a constant heat treatment time of 1 hour. The grain structures found at the various stages of the process are also presented schematically. (Calliester, W. D. & Wiley, Jr. J.& Sons Inc.,1999 )

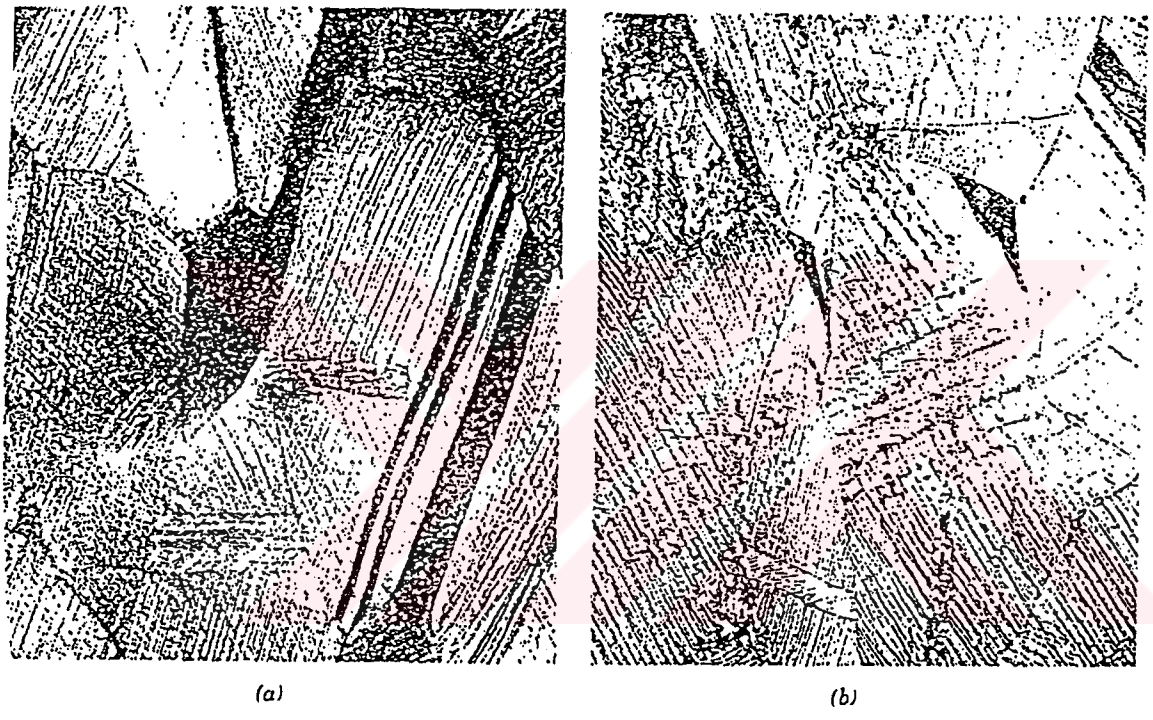


Figure 2.5 Photomatricrographs showing several stages of the recrystallization and grain growth of brass,(a) Cold worked grain structure,(b) Initial stage of recrystallization after heating 3s at 580°C; the very small grains are those that have recrystallized,(c) Partial replacement of cold worked grains by recrystallized ones 4s at 580°C;(d) Complete recrystallization 8s at 580°C;(e) Grain growth after 15 min. at 580°C;(f) Grain growth after 10 min. at 700°C; All photomicrographs 75X.(Photomicrographs courtesy of J.E. Burke, General Electric Company.) Calliester W. D. 5 $^{th}$ ed. ( 1999 ). Materials Science and Engineering and Introduction.

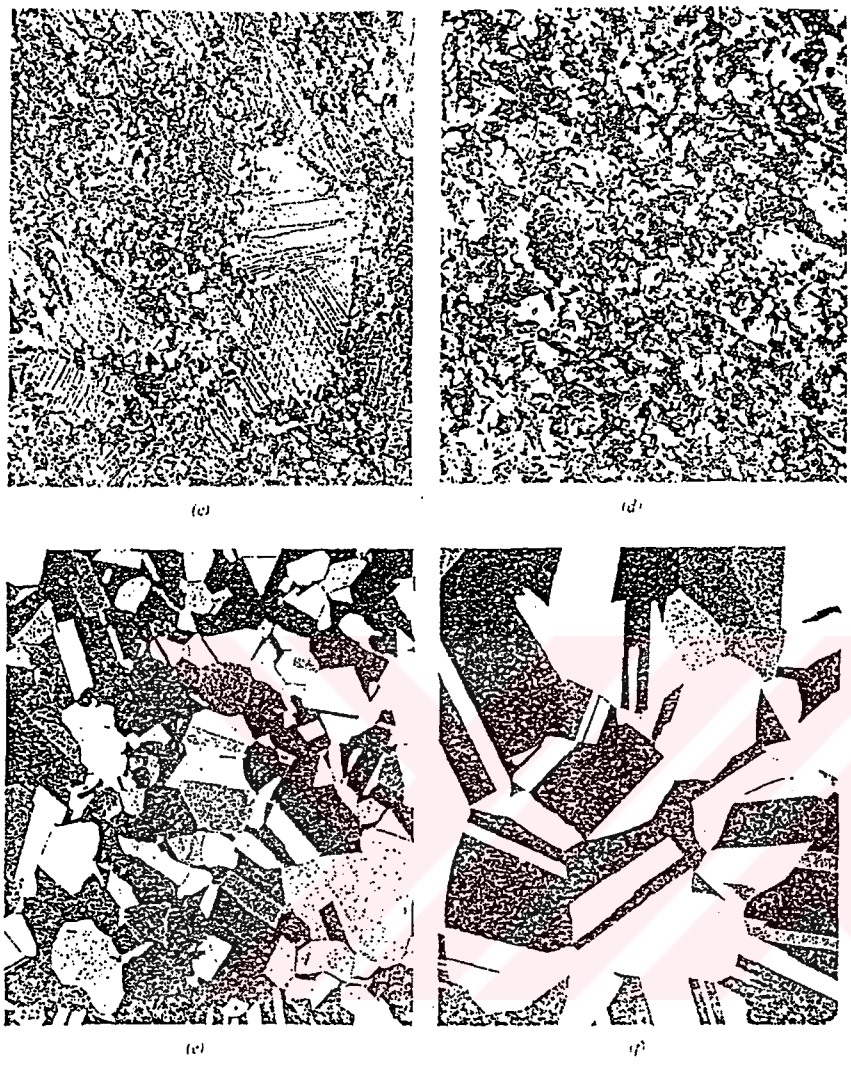

Figure 2.5 (continued)

The recrystallization behaviour of a particular metal alloy is sometimes specified in terms of a **recrystallization temperature,** the temperature at which recrystallization just reaches completion in 1 hour. Thus, the recrystallization temperature for the brass alloy of Figure 2.4 is about 450°C (850°F). Typically, it is between one third and one half of the absolute melting temperature of a metal or alloy and depends on several factors, including the amount of prior cold work and the purity of the alloy. Increasing the percentage of cold work enhances the rate of recrystallization, with the result that the

recrystallization temperature is lowered, and approaches a constant or limiting value at high deformations; this effect is shown in Figure 2.6. Furthermore, it is this limiting or minimum recrystallization temperature that is normally specified in the literature. There exists some critical degree of cold work below which recrystallization cannot be made to occur, as shown. In the figure; normally this is between 2 and 20% cold work.

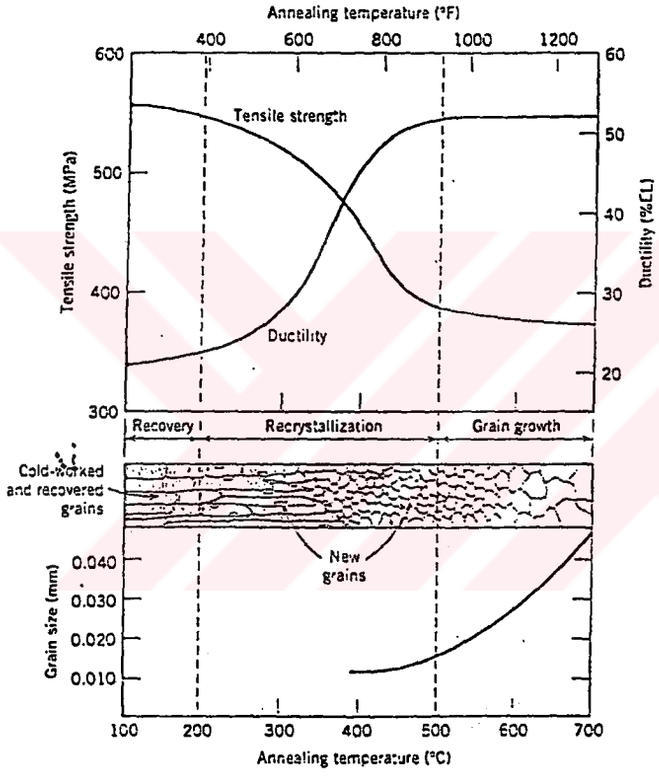


Figure 2.6. The influence of annealing temperature on the tensile strength and ductility of brass alloy. Grain size as a function of annealing temperature is indicated. Grain structures during recovery, recrystallization, and grain growth stages are shown schematically. Calliester W. D. 5 th ed.(1999).Materials Science and Engineering and Introduction.

Recrystallization proceeds more rapidly in pure metals than in alloys. Thus, alloying raises the recrystallization temperature, sometimes quite substantially. For pure metals, the recrystallization temperature is normally $0.3T_m$, where $T_m$ is the absolute melting temperature; for same commercial alloys it may run as high as $0.7T_m$. Recrystallization and melting temperatures for a number of metals and alloys are listed in Table 2.1.

Table 2.1 Recrystallization and Melting Temperatures for Various Metals and Alloys

| Metal | Recrystallization Temperature | | Melting Temperature | |
|---|---|---|---|---|
| | °C | °F | °C | °F |
| Lead | -4 | 25 | 327 | 620 |
| Tin | -4 | 25 | 232 | 450 |
| Zinc | 10 | 50 | 420 | 788 |
| Aluminium(99.999 wt%) | 80 | 176 | 660 | 1220 |
| Copper (99.999 wt%) | 120 | 250 | 1085 | 1985 |
| Brass (60 Cu-40 Zn) | 475 | 887 | 900 | 1652 |
| Nickel (99.99 wt%) | 370 | 700 | 1455 | 2651 |
| Iron | 450 | 840 | 1538 | 2800 |
| Tungsten | 1200 | 2200 | 3410 | 6710 |

### 2.3.4 Grain Growth

After recrystallization is complete, the strain - free grains will continue to grow if the metal specimen is left at the elevated temperature (Figure 2.5); this phenomenon is called

**grain growth.** Grain growth does not need to be preceded by recovery and recrystallization; it may occur in all polycrystalline materials, metals and ceramics alike.

Energy is associated with grain boundaries. As grains increase in size, the total boundary area decreases, yielding an attendant reduction in total energy; this is the driving force for grain growth. Grain growth occurs by the migration of grain boundaries. Obviously, not all grains can enlarge, but large ones grow at expense of small ones that shrink.

The mechanical properties at room temperature of a fine-grained metal are usually superior (i.e., higher strength and toughness) to those of coarse - grained ones. If the grain structure of a single-phase alloy is coarser than that desired, refinement might be accomplished by plastically deforming the material, then subjecting it to a recrystallization heat treatment, as described above. (Calliester W. D., Wiley Jr. J. & Sons Inc., 1999 )

## 2.4 RECRYSTALLIZATION OF ALLOY CONTAINING SECOND PHASE PARTICLES

### 2.4.1 Introduction

Recrystallization starts with nucleation, which is related to the cold worked structure of the material. Cold worked structure of alloys is directly influenced by the presence of second phase dispersions. The second phase particles will also affected the boundary movement and consequently the speed of recrystallization. An accelerated nucleation rate with efficient boundary movement will result in accelerated recrystallization. The lack of nucleation sites in the material and, with the presence of finely dispersed particles hindering the movement of boundaries will retard the recrystallization. However, dispersed particles can either accelerate or retard the recrystallization.

### 2.4.2 Cold Deformed Structures of the Alloys Containing Second Phase Particles

Cold deformation of the alloys containing undeformable particles has been investigated extensively in recent year. Here the main features of the structures, which effect nucleation, will be described briefly. When these alloys are subjected to plastic deformation the matrix will deform but particles will not. In this case, according to Ashby in order to keep the compatibility the " geometrically necessary" dislocations are needed in the matrix around the particles. These compatibility dislocations form as prismatic loops (an array of prismatic loops) at small particles and secondary shear arrays at large particles. Secondary shear arrays involve local lattice bending around the particles whereas prismatic loops produce no lattice bending.

The presence of particles will also have a large influence on the deformed structure of the particle-free matrix. Deformation in such alloys results in a high dislocation density at relatively low values of strain and random distribution of dislocations throughout the matrix, since the particles act as dislocation sources as well as the barriers to dislocation movement. Normal cell structure does not form easily with fine dispersed particles in the matrix; after severe cold deformation fine cells start forming. With coarser distributions cell structure is formed rather easily similar to that of pure metals. The misorientation across these cell boundaries is less than those in pure metals. ( Önel K., 1977., Structure Property Relationships in Cold Worked and Tempered Steels.)

### 2.4.3. Accelerated or retarded recrystallization

The deformed structure is dependent on particle diameter and the nucleation rate of recrystallization is dependent on the structure. Large particles( >3000 Angstrom) create nucleation sites, whereas small particles (<3000 Angstrom) lead to homogenised dislocation structure and hinder nucleation.

If second phase particles are present in the metallic matrix grain boundaries interact with the second phase particles. When a grain boundary is forced to migrate for grain growth the particles of a second phase will interact with the grain boundaries and exert a force tending to hold it back. The quantitative relationships between the particle distribution parameters and grain size are found in the literature. These relationships suggest that the grain growth is hindered by the second phase particles and there is a limitation to the allowable maximum grain size dictated by the distribution parameters of the second phase particles.( Önel K., 1977., Structure Property Relationships in Cold Worked and Tempered Steels.)

# CHAPTER THREE
# THE EXPERIMENTAL

## 3.1 PRODUCTION OF COMPOSITES

### 3.1.1 The Materials

SiC particle reinforced aluminium composites produced by researcher were used. The production of the composites was described in detail by Mehmet Dombayci. Details of the particulate base metal matrix composite materials are as follow: Matrix material, AlSi5Mg with on average particle diameter of 20 μm and the other matrix is AlSi7Mg with an average particle size of 23μm, with the reinforcement being 0,10,20 volume percent. The chemical composition of these alloys and resultant matrix alloy are given in Table 3.1. The materials were analysed with a spectrometer. In this study, abrasive grade Silicon Carbide was used. The properties of Silicon Carbide particles are given in Table 3.2.

**Table 3.1.** The Chemical Composition of Materials Used In Experiments.

| Elements | Al-%20 SiCp | Al-%10 SiCp | Matrix Alloy |
|---|---|---|---|
| Si | 23.5256 | 18.7855 | 17.757 |
| Fe | 0.3238 | 0.4179 | 0.2843 |
| Cu | 0.4133 | 0.1463 | 0.0150 |
| Mn | 0.0291 | 0.0236 | 0.0260 |
| Ni | 0.0471 | 0.0261 | 0.0131 |
| Zn | 0.3222 | 0.0479 | 0.0716 |
| Ti | 0.0882 | 0.0845 | 0.0844 |
| Cr | 0.0103 | 0.0416 | 0.0081 |
| Pb | .0.0217 | 0.0084 | 0.0352 |
| Sn | 0.0299 | 0.0138 | 0.0450 |
| Be | 0.0000 | 0.0000 | 0.0000 |
| V | 0.0060 | 0.0063 | 0.0130 |
| Al | 74.2928 | 79.6547 | 90.3505 |

**Table 3.2.** Properties of SiC reinforcement

| Particle | Elastic Modulus ($GNm^{-2}$) | Density ($g/cm^3$) | Coefficient of Thermal Expansion ($K^{-1}$) | Specific Heat ($Jkgr^{-1}K^{-1}$) | Thermal Conductivity ($Wm^{-1}K^{-1}$) | Poisson's Ratio |
|---|---|---|---|---|---|---|
| SiC | 420-450 | 3.2 | 4.3.10 | 840 | 10-40 at 1100C$^{-1}$ | 0.17 |

### 3.1.2 Production of Composites

Different amount aluminium and its alloys were added into the crucible and then melted in a crucible by an induction furnace. The alloys were held at a temperature above the liquids, then the impeller was lowered into the melt and the mixer was run at low speed to prevent excessive vortex. The temperature was lowered slowly about 600° C. Meanwhile, the SiC particles were kept in another furnace at 900°C for two hours, in order to remove the moisture and oxidize the particles. When melt reached the right consistency the SiC particles were introduced manually into the crucible slowly. After the completion of the particle addition, the stirring was continued for ten minutes. The melting process was carried out under protective argon atmosphere. The melt temperature was controlled by a thermocouple. Then the mixer was turned off and removed from the melt. The melt was heated up to 750°C and again the mixer was lowered into the melt and stirring was continued at a low speed for five minutes to prevent sedimentation at the bottom of the crucible. Then the mixer was turned off and removed from the melt. The superheated melt was poured into the cast iron mould. The mould temperature was 250°C. After, the solidification was completed, a cast ingot of 35 mm diameter and 60 mm height was obtained. Chemical compositions of produced composites and the properties of the reinforcement are given in Table 3.1.

### 3.1.3 Extrusion Process

The produced compo-cast samples were machined to a diameter of 31mm and a length of 58mm. The samples were extruded at a ratio of 10:1. At first, the extrusion die and the sample were lubricated with graphite in order to resist high temperature. Then, the die and sample were heated for 30 minute to different temperatures in different furnaces. The die temperature was 400 °C and the sample temperature was 530°C. After heating, the die was taken rapidly from furnace, then placed on the press base and

sample was put in the die. Then the piston was lowered into the extrusion die at a speed of 0,7 m/min. With the process the cast ingots were successfully extruded into bars of 10mm diameter and 500 mm length.

### 3.1.4 Cold Deformation Process

The extruded samples were subjected to cold deformation. For this process, specimens were cut into 30mm long pieces from the extruded bars. Before the cold deformation, specimens were annealed for one hour at 400°C, then wrapped in PTFE against frictional effects. After this preparation, these specimens were put on the base of a press. In two different stages the press force was applied to specimens, which was 32 tons. Finally, The specimens were cold deformed 60 % and the specimens of 30mm length and 3.8mm thickness were obtained.

### 3.1.5 Heat Treatment

After the cold deformation, the samples were subjected to annealing process at different temperatures for one hour, to determine the recrystallization temperture of different samples.. The so-called recrystallization temperature which changes according to the melting temperature of material (Al-SiCp) and the amount of cold deformation. The temperature of annealing was selected considering these factors. For heat treatment, four different temperatures were chosen as given in Table 3.3. The specimens were annealed at these temperatures for one hour. After, these treatments at four different temperatures (Table 3.1.a.), the specimens were subjected to a final heat treatment at 400°C up to 40 hours, and finally the specimens were cooled in air. The purpose of this treatment was to observe whether the grain growth and the particle coarsening take place in these composites at 400°C.

Table 3.3.a The annealing temperatures and durations for recrystallization experiments.

| SiCp Volume fraction | Annealing temperatures | Duration of annealing |
|---|---|---|
| 0,10,20% | 200°C | 1 hour |
| 0,10,20% | 275°C | 1 hour |
| 0,10,20% | 350°C | 1 hour |
| 0,10,20% | 400°C | 1 hour |

Table 3.3.b The Temperatures and Times for Coarsening Heat Treatment

| SiCp Volume fraction | Annealing temperatures | Duration of annealing |
|---|---|---|
| 0% | 400 °C | 1, 5, 10, 40 hours |
| 10% | 400 °C | 1, 5, 10, 40 hours |
| 20% | 400 °C | 1, 5, 10, 40 hours |

### 3.1.6 Preparation of metalographic specimens

The matrix alloy itself and the composites were prepared for metallographic examination to investigate their compo cast and annealed structures. The specimens were cut to rectangular forms around one centimetre at longest side. Then, they were ground by SiC abrasive papers with the sequence of 80, 120, 220, 400, 600, 800, 1000, 1200 sizes. Then, the specimens were polished using diamond paste. After this process, the etching was carried out using Keller's solution which contains 1ml HF, 1,5ml HCl,

2,5ml $HNO_3$ and 95ml $H_2O$. The specimens were dipped in this solution for ten seconds. Alcohol was used for final cleaning of the surface before examination by an optical microscope.

### 3.1.7 Hardness Tests

The hardness of the samples was measured using a Vickers Hardness tester and 5 kp load. The load application time was 15 seconds.

### 3.1.8 Density Measurement and Porosity

The densities of specimens were calculated according to the Archimedes rule. First of all, the samples were weighed in air and then in distilled water. After this process the densities were calculated using the equations below;

$$D = \frac{m_1}{m_2 - m_1}$$

Where D is density, $m_1$ and $m_2$ are the weight in air and in water respectively. Porosity content was calculated by using the experimental value of density.

# CHAPTER FOUR

# RESULTS AND DISCUSSION

## 4.1 THE MICROSTRUCTURE

A typical microstructure of the cast composites observed by SEM is as shown in Figure 4.1. These composites are examined under light microscope and noticed that particle distribution is different in each sample. These differences are the porosity content, the distribution of reinforcement as well as eutectic silicon, in the as cast condition, being a function of the solidification rate. Besides, the phases were found such as ∝-Al, eutectic Si, ∝+β-SiC and intermetallic phases.

As shown in Figure 4.2, the cast composite has a considerable amount of porosity. In the as cast condition, the porosity is caused by uncontrolled stirring and solidification rate of matrix. These two factors are important for homogeneous microstructures and other mechanical properties especially hardness. It was concluded that these two factors, uncontrolled stirring and solidification rate have significant effect on hardness and grain size.

Two types of samples were examined, one of these contains 20 vol. % SiCp (20μm) and the other 20 vol. % SiCp (23μm). After examining these composites in light microscope, it was noticed that the micrographs of these two samples are different. In these composites, there are too much porosity and sedimentation because of

uncontrolled stirring and solidification as mentioned above. The composites with 20 vol. % SiCp (23μm) do not have a homogeneous particle distribution.

Figure 4.3 shows the effects of extrusion besides cold deformation on the microstructure of the composites. In all cold deformed composites, cracked particles were found. But in the as extruded samples not many of these cracked particles were noticed. Before the cold deformation, we noticed that very small particles existed in matrix. After the heat treatment was applied to the cold deformed samples, these small particles grew bigger. In the same specimens, the cluster of particles existed before the extrusion. These clusters were eliminated with the plastic deformation and more uniform distribution of particles was obtained.

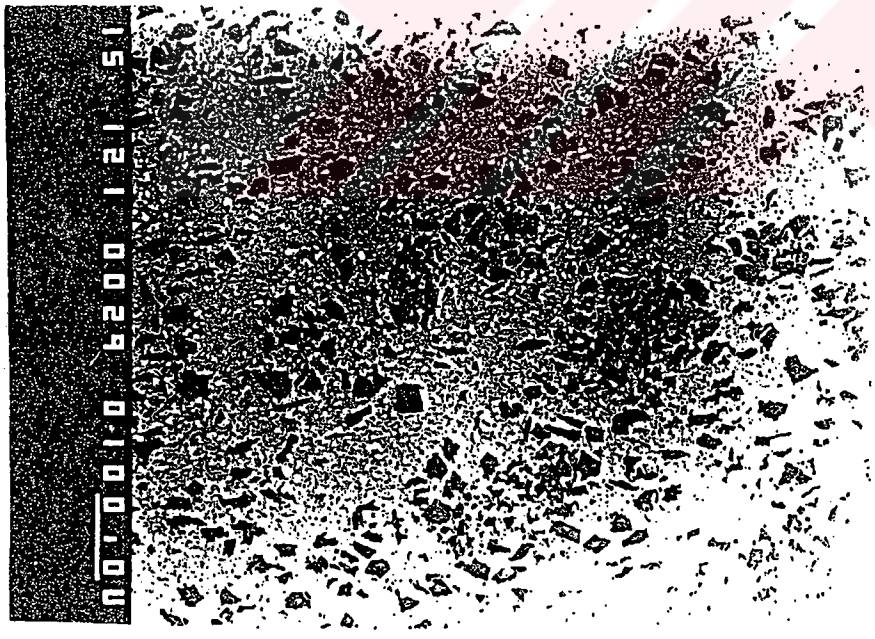

Figure 4.1 AlSi5 / SiC20p-cold deformed

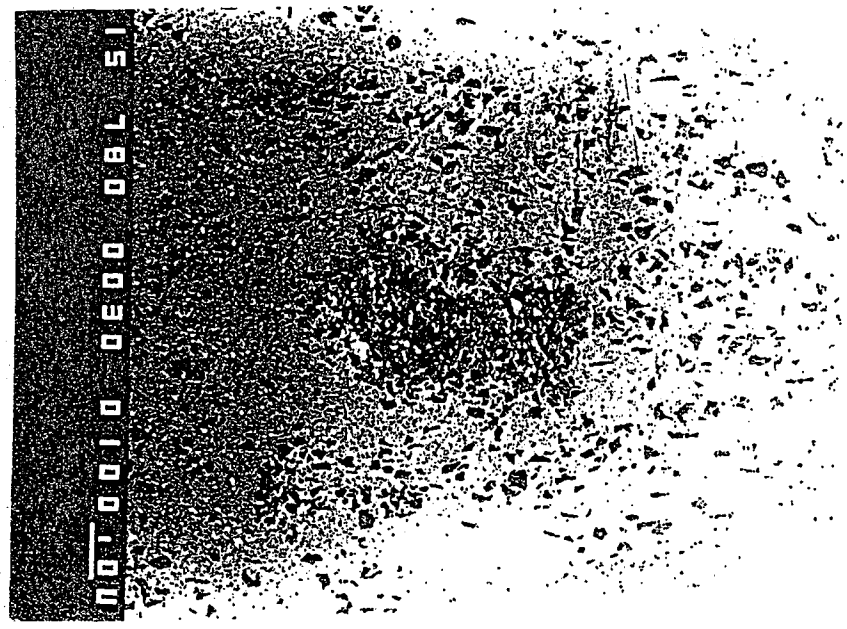

Figure 4.2 AlSi5 / SiC20p-cold deformed

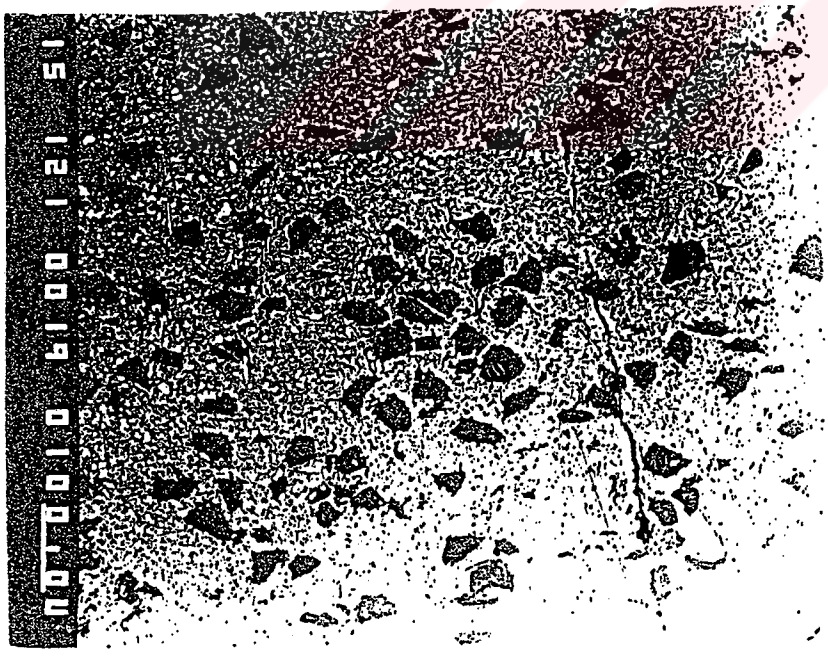

Figure 4.3 AlSi5 / SiC20p-cold deformed

## 4.2 The Density and Porosity

In table 4.1 both experimental and calculated densities are given. There is a difference between calculated and experimental densities These differences are due to the porosity in the structures. The experimental density values indicate that the density of composites decreases when the porosity increases.

Table 4.1 The densities of composites

| | Calculated Density Dc [gr/cm³] | Experimental Density, De[gr/cm³] As cast | Experimental Density, De[gr/cm³] As extruded | Porosity [%] As cast | Porosity [%] As extruded |
|---|---|---|---|---|---|
| Matrix | 2,711 | 2.7063 | 2,7084 | 0,18 | 0,10 |
| AlSi5/SiC | 2,797 | 2,7558 | 2,7716 | 1,15 | 0,91 |
| AlSi7/SiC | 2,7942 | - | 2,7610 | - | 1,19 |

As seen in Table 4.1, after the extrusion, experimental results show that the ratios De/Dc of AlSi5/SiC and AlSi7/SiC are lover than those of the as cast samples. As expected the porosity content decreases after the application of hot extrusion process.

Figure 4.4 shows that the porosity changes according to deformation process and the densities depend on the porosity. After cold working some porosity still existed in the structures.

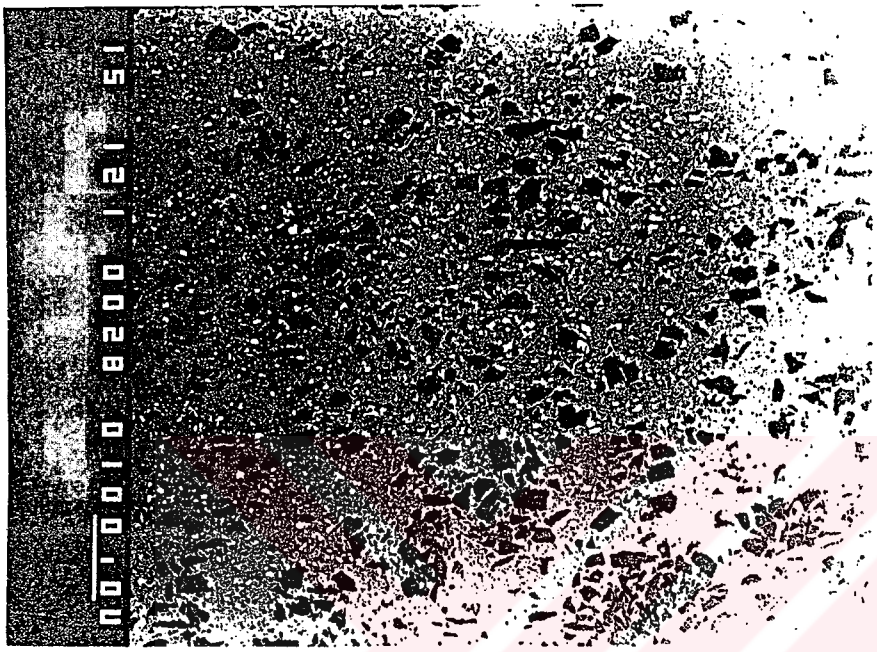

Figure 4.4 AlSiC7 / 20p-Cold deformed

### 4.3. The Hardness

The hardness measurements were carried out after applying cold work and after the annealing treatments at different temperatures, to study the recrystallization behaviour of 60 % cold-worked matrix alloy and silicon carbide particle reinforced aluminum composites. The hardness values observed were similar for all the samples after cold work.

Different annealing temperatures were chosen for the recrystallization study of the matrix alloy and the composites in order to determine the recrystallization temperature of these materials, and to see if there is any significant difference in the recrystallization

temperatures of different samples. In the initial stage of experiments the cold-worked specimens were annealed for 1 hour at different temperatures sufficient to stimulate the formation of recrystallized grains. The resulting hardness values of recrystallization experiments are given in Table 4.1.

| | | Matrix + SiCp | | | |
|---|---|---|---|---|---|
| Temperature | Time | % 0 | %10 | %20-E | %20-Y |
| 0°C | | 77 | 74 | 83,1 | 76,1 |
| 200°C | 1 hour | 77,5 | 84 | 79,4 | 68,1 |
| 275°C | 1 hour | 62,2 | 64,2 | 46,8 | 57,4 |
| 300°C | 1 hour | 61 | 48,8 | 50,6 | 46 |
| 350°C | 1 hour | 49,5 | 50,4 | 46,8 | 44 |
| 400°C | 1 hour | 48,3 | 56 | 44,6 | 48,3 |
| 400°C | 5 hour | 45,2 | 46,6 | 44,8 | 39,19 |
| 400°C | 10 hour | 46 | 52,6 | 47,5 | 46,6 |
| 400°C | 40 hour | 48,3 | 55,2 | 49 | 51,3 |

Table 4.2 The Hardness values of the composites after annealing for 1 hour at different temperatures, and up to 40 hours at 400°C.

The Figures 4.5, 4.6, 4.7, 4.8 illustrate hardness change of 60 % cold-worked materials annealed one-hour at 200°C, 275°C, 300°C and 400°C, respectively. The recrystallization temperatures were determined from the hardness - temperature curves.

The changes in hardness of 60% cold-worked material during annealing treatments were also followed by micro - hardness measurements which showed a similar trend to microhardness results. The Figures 4.5, 6, 7, and 8 show that, the hardness curves stay flat up to the recovery and then hardness decreases with increasing temperatures, after

recrystallization the rate of decrease is much lower. In other words the hardness curves exhibit typical appearance of hardness change during recrystallization process. These figures show that the recrystallization temperatures are situated in a narrow range for different samples. The measured temperatures are between 250°C-290°C.

It is found from the figures that the recrystallization temperature is 250°C for the matrix alloy and, 260°C and 290°C for the composites containing 10 vol.% and 20vol% SiCp (Y) For the other sample with 20 vol.% SiCp (E) the recrystallization temperature is 250°C. The average particle size of SiCp in the sample 20 vol.% SiCp (E) is larger than that of the (Y) type samples. These results show that increasing SiCp volume fraction in the (Y) type specimens increased the recrystallization temperature. But on the other hand the recrystallization temperature of the sample 20 vol.% SiCp (E) is the same as that of the matrix.

Recrystallization studies in particle-reinforced metal matrix composites have shown that the nucleation potency of the reinforcement increases with increasing reinforcement size.

The results of the recrystallization experiments are in agreement with this conclusion, thus the composite with large reinforcement size, 20% SiCp(E) has lower recrystallization temperature than the specimen 20% SiCp(Y) with smaller reinforcement size.

In the second stage of the experiments, the samples were undergone a heat treatment at 400°C up to 40 hours. The results of these experiments showed that within the first 5 hours of the annealing, the hardness decreases slightly then increases back to the level at the beginning of the annealing treatments.. After annealing for larger times the hardness values remain almost constant.

The hardness change of different materials during annealing at 400 °C was similar. Between 10 and 40 hours, there was almost no change in hardness, in other words the hardness is nearly constant for all the materials.

In the most metallic materials, grain size influences mechanical properties, ductility, strength, and hardness. In particle-reinforced metal matrix composites, the grain size is generally small compared to an unreinforced material of similar composition. Accordingly, the influence of grain size on the mechanical properties of these materials has received little attention, in part because of the perceived difficulty of producing substantial variations in grain size.

The recrystallized samples were annealed at 400°C for up to 40 hours in order to observe whether any growth of recrystallized grains and the particles takes place. The hardness measurements with annealing time show that (Figures 4.10, 4.11, 4.12) within the first 10 hours of annealing a negligible variation in hardness is observed, and during the remaining period of annealing the hardness curve becomes completely flat.

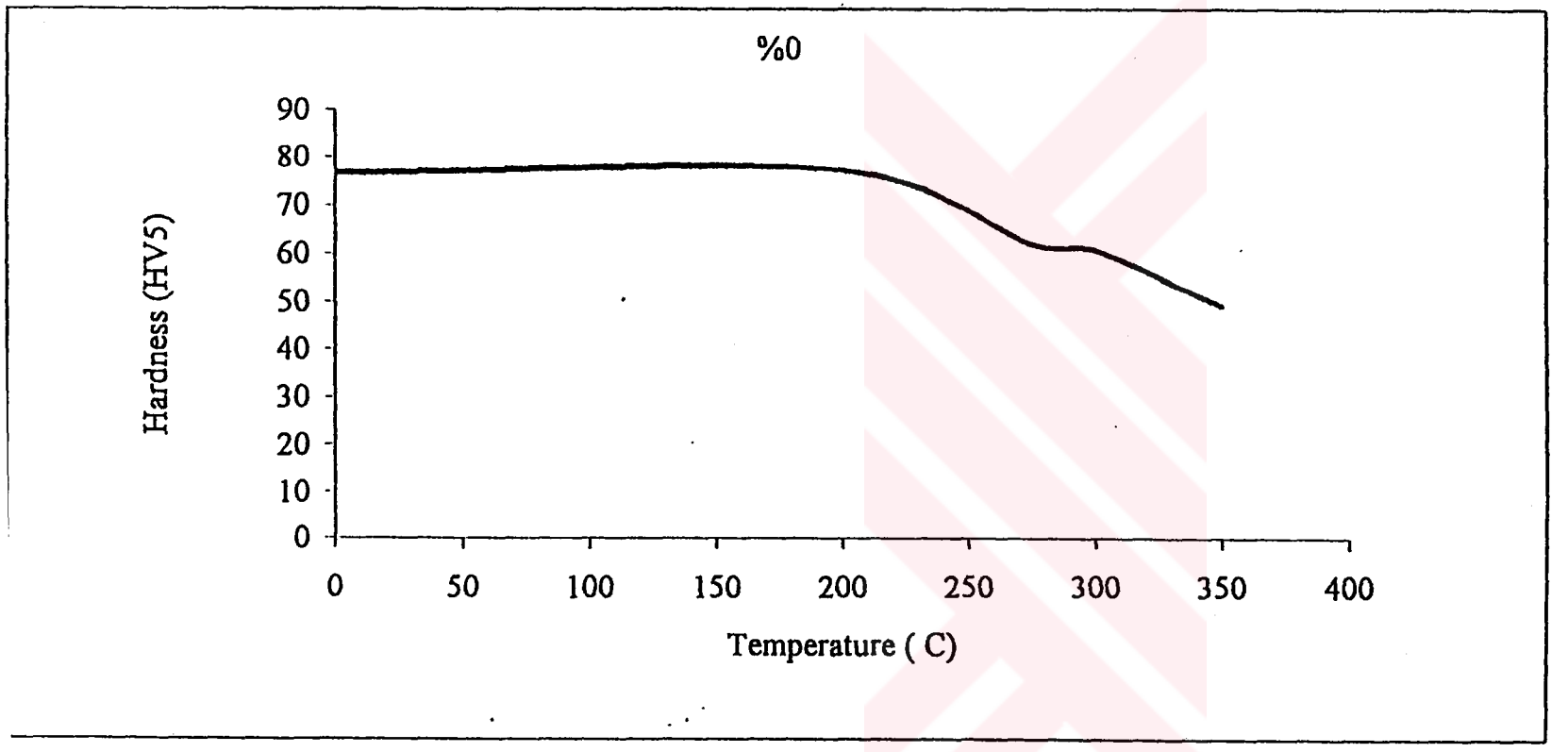


Figure 4.5 The Hardness Change of Matrix Alloy after Annealing at Different Temperatures for 1 Hour

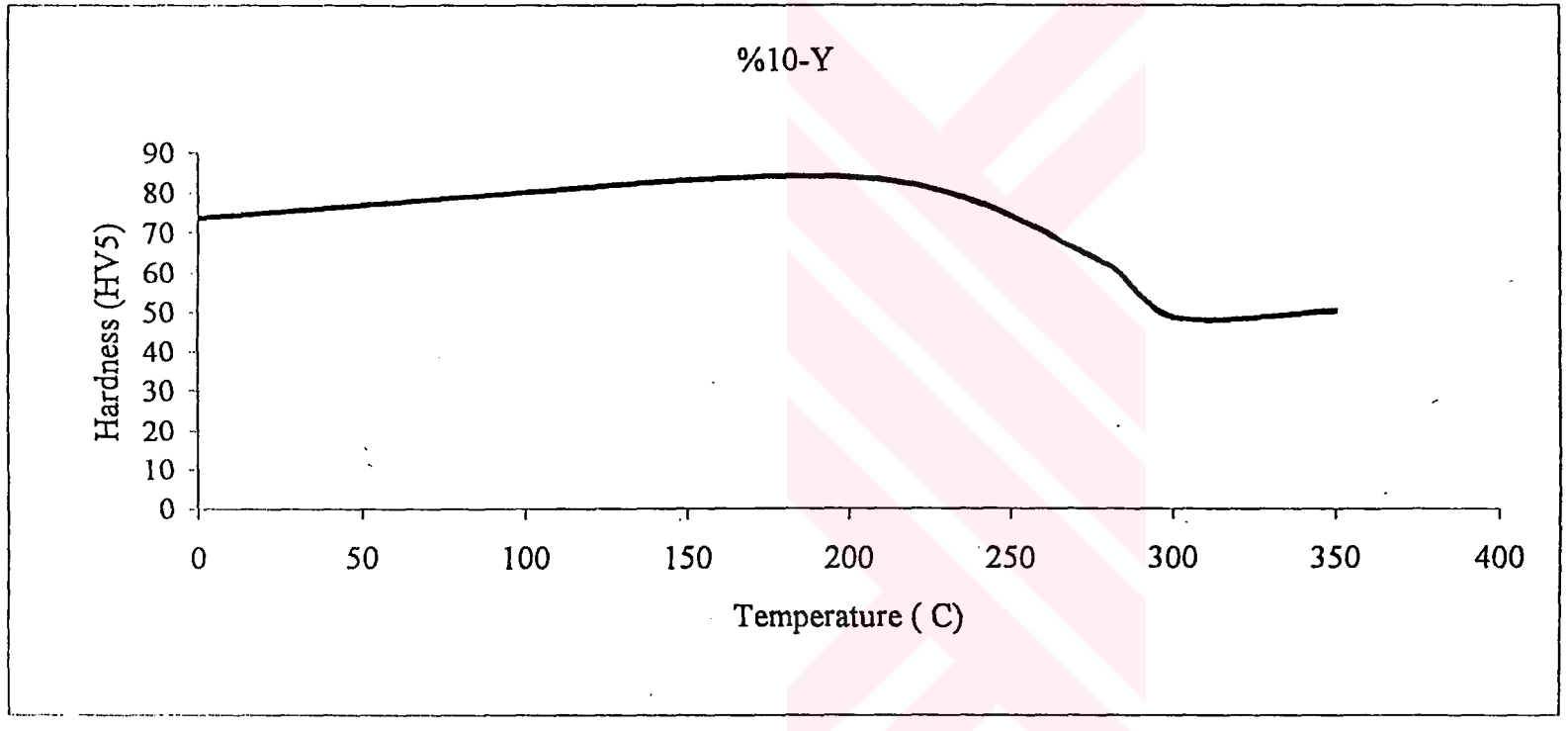


Figure 4.6 The Hardness Value Change of 10% - Y SiCp Containing Composite after Annealing at Different Temperatures for 1 Hour

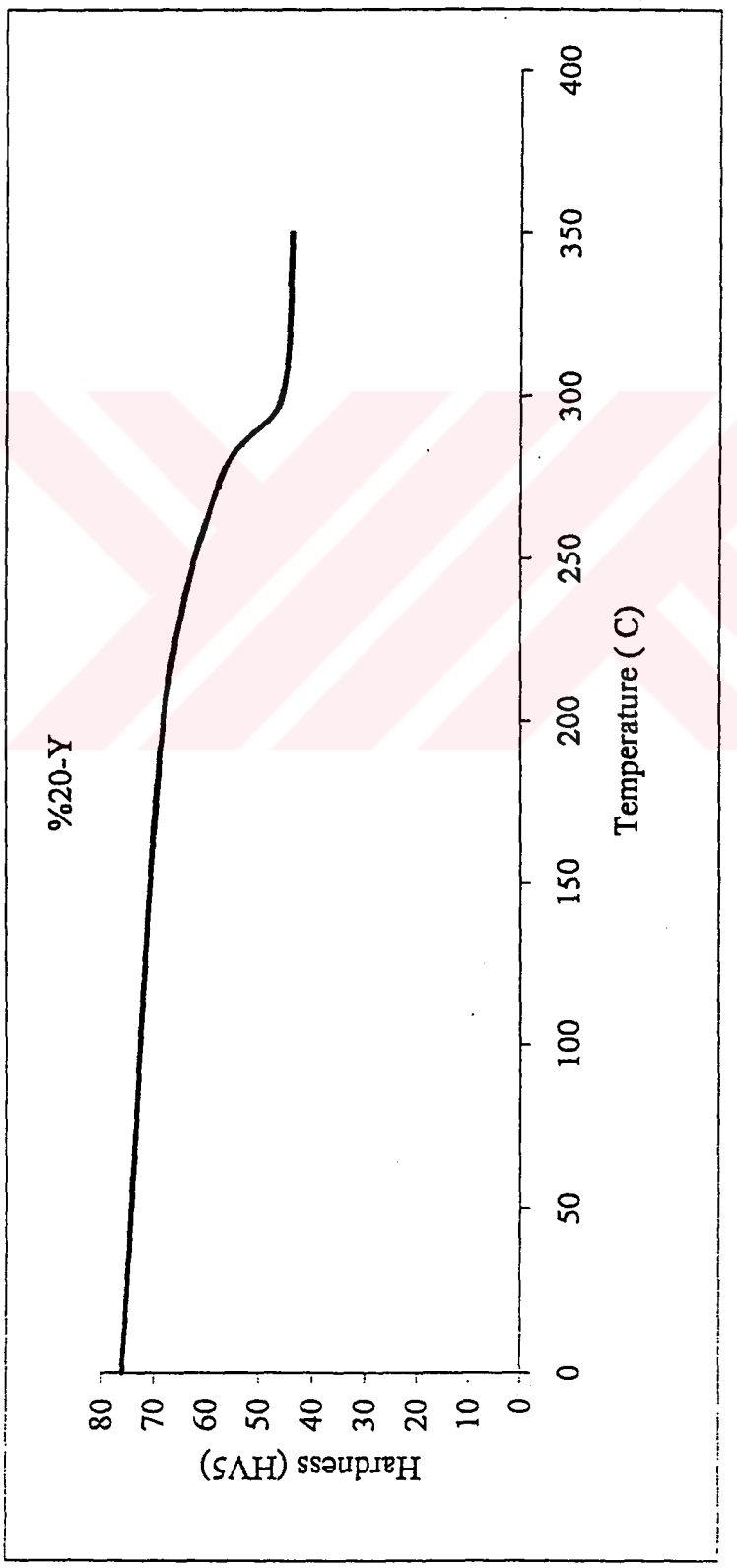


Figure 4.7 The Hardness Value Change of 20% SiCp - Y Containing Composite after Annealing at Different Temperatures for 1 Hour

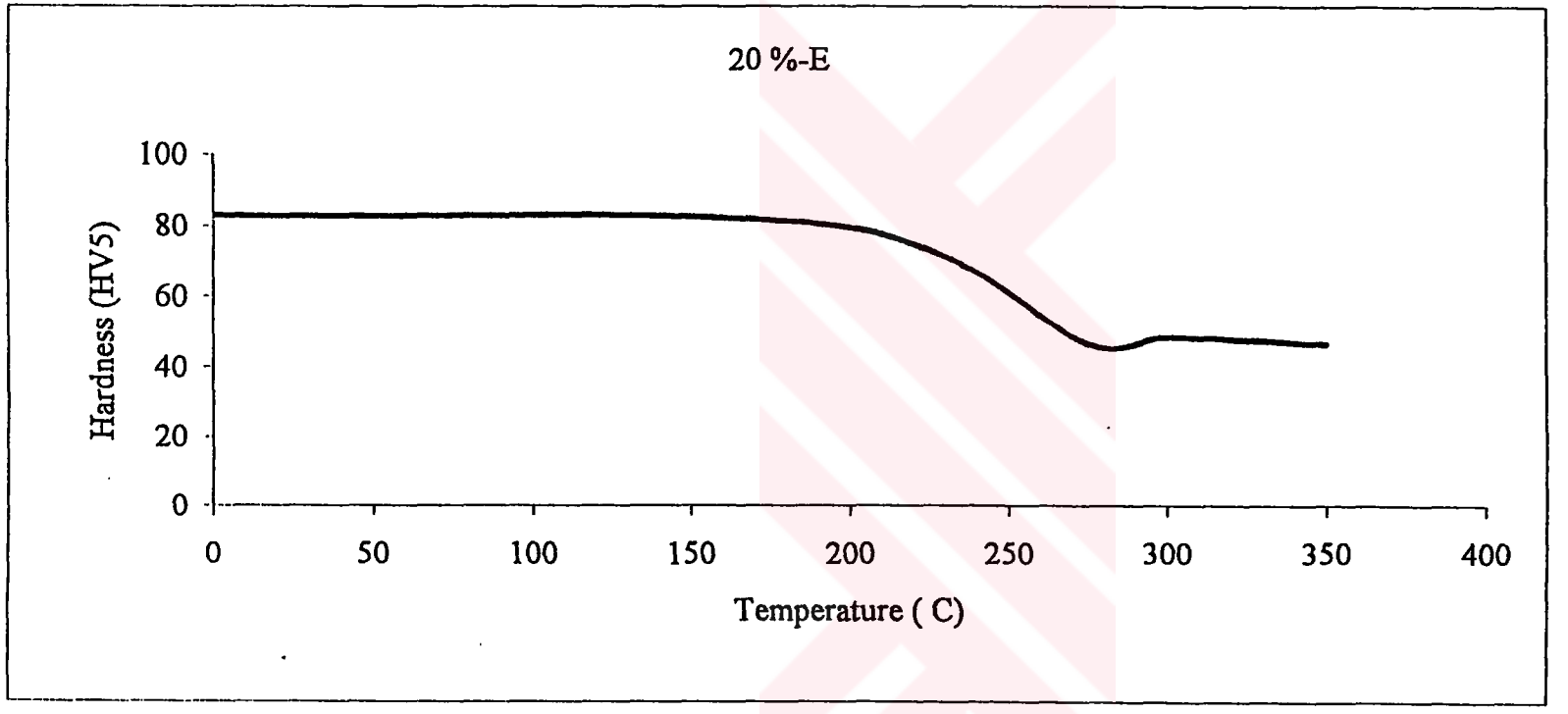


Figure 4.8 The Hardness Value Change of %20 SiCp - E Containing Composite after Annealing at Different Temperatures for 1 Hour

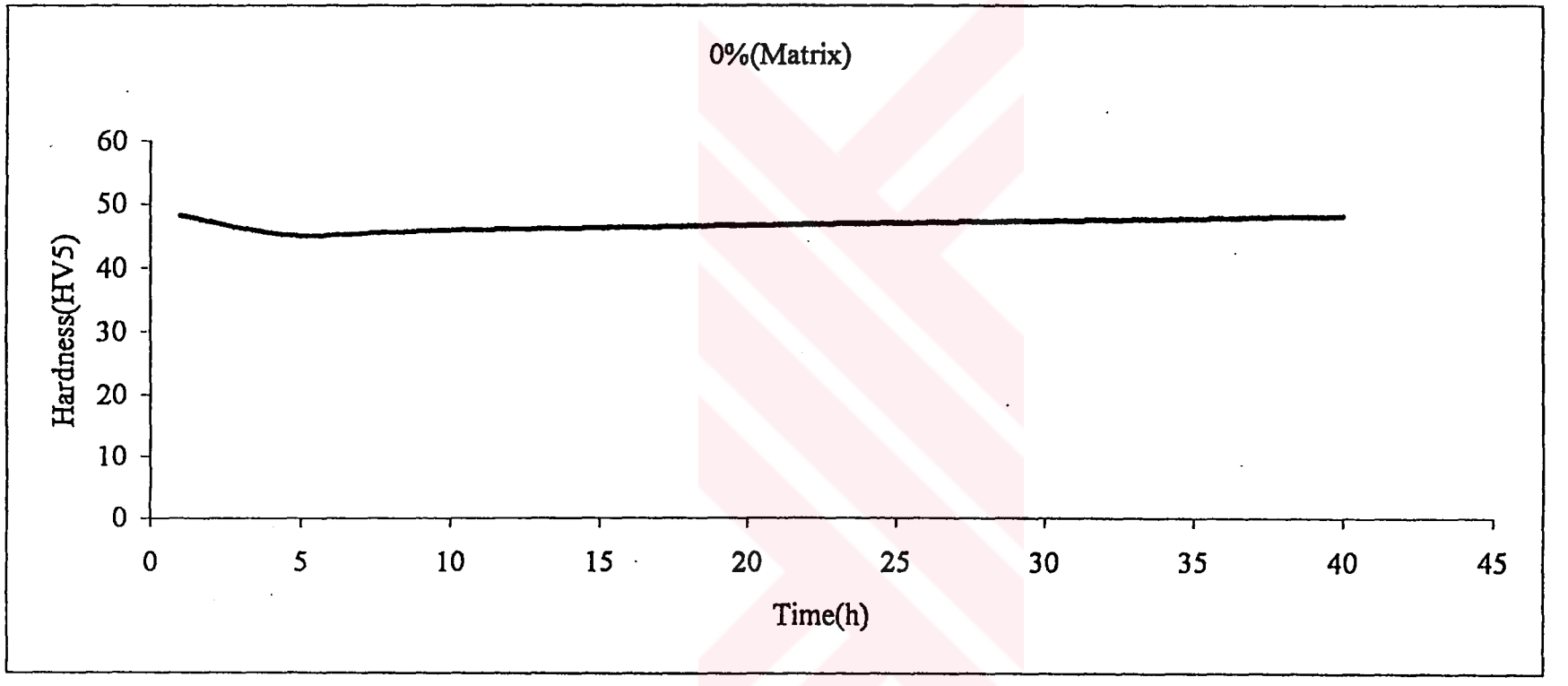


Figure 4.9 The Hardness Change of the Matrix ( 0 vol. % SiCp ) with Annealing Time at 400 C

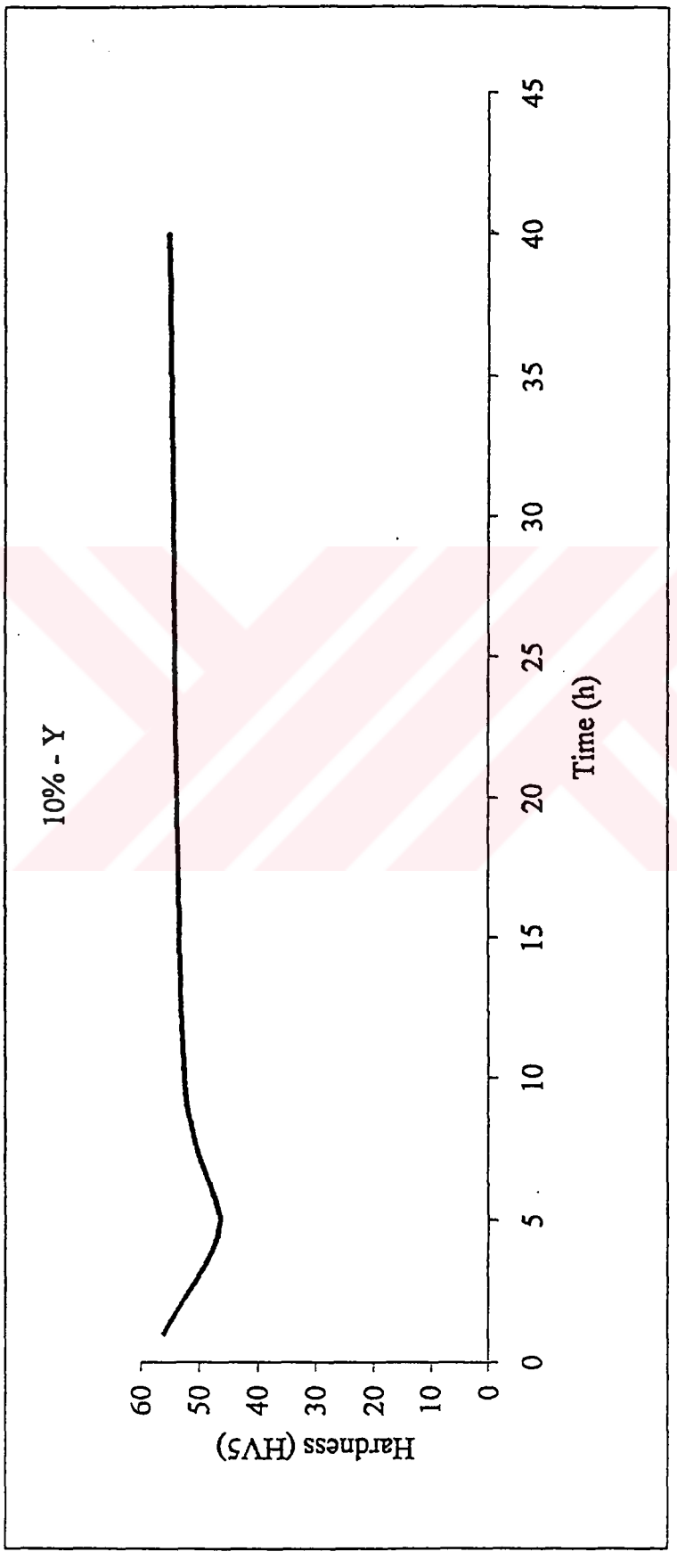


Figure 4.10 The Hardness Change of 10 vol.% SiCp Containing Composite with Annealing Time at 400 C

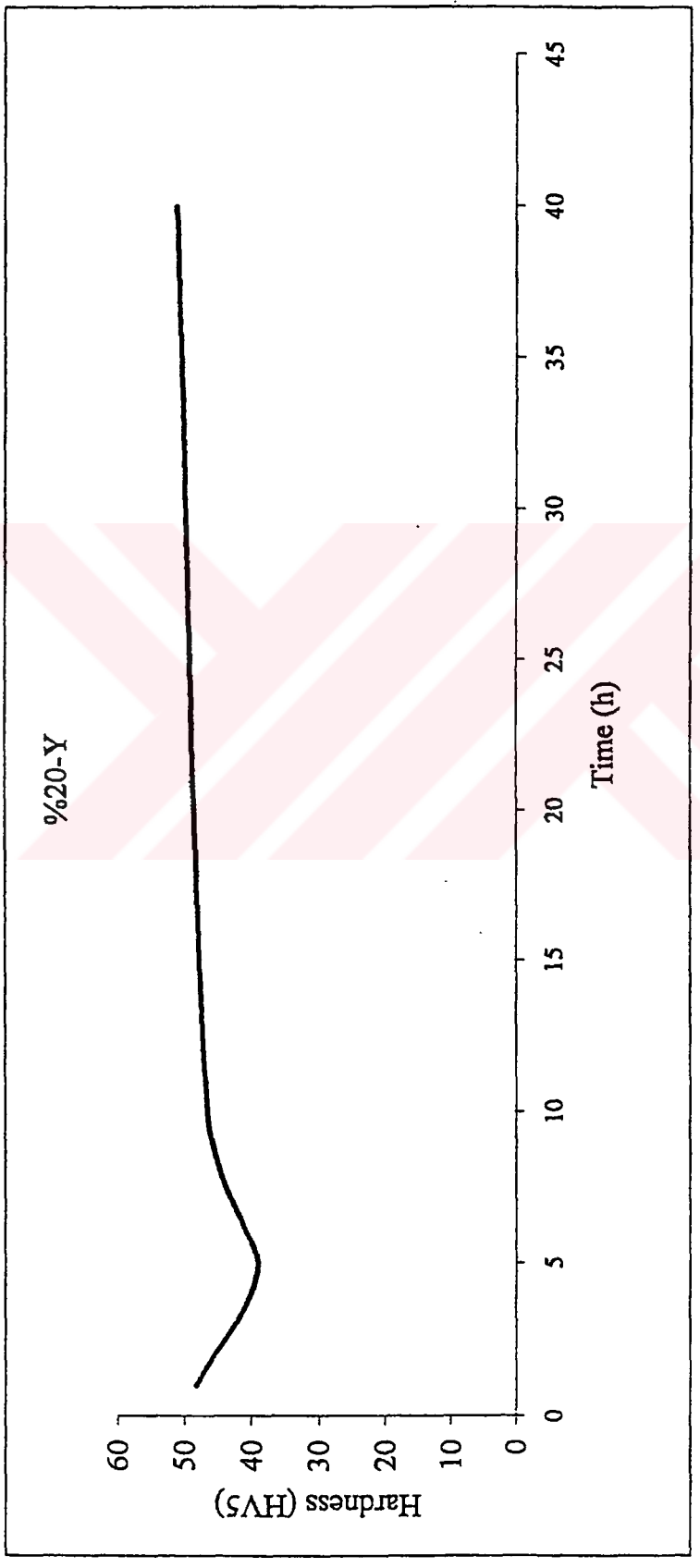


Figure 4.11 The Hardness Change of 20 vol. % - Y SiCp Containing Composites with annealing Time at 400 C

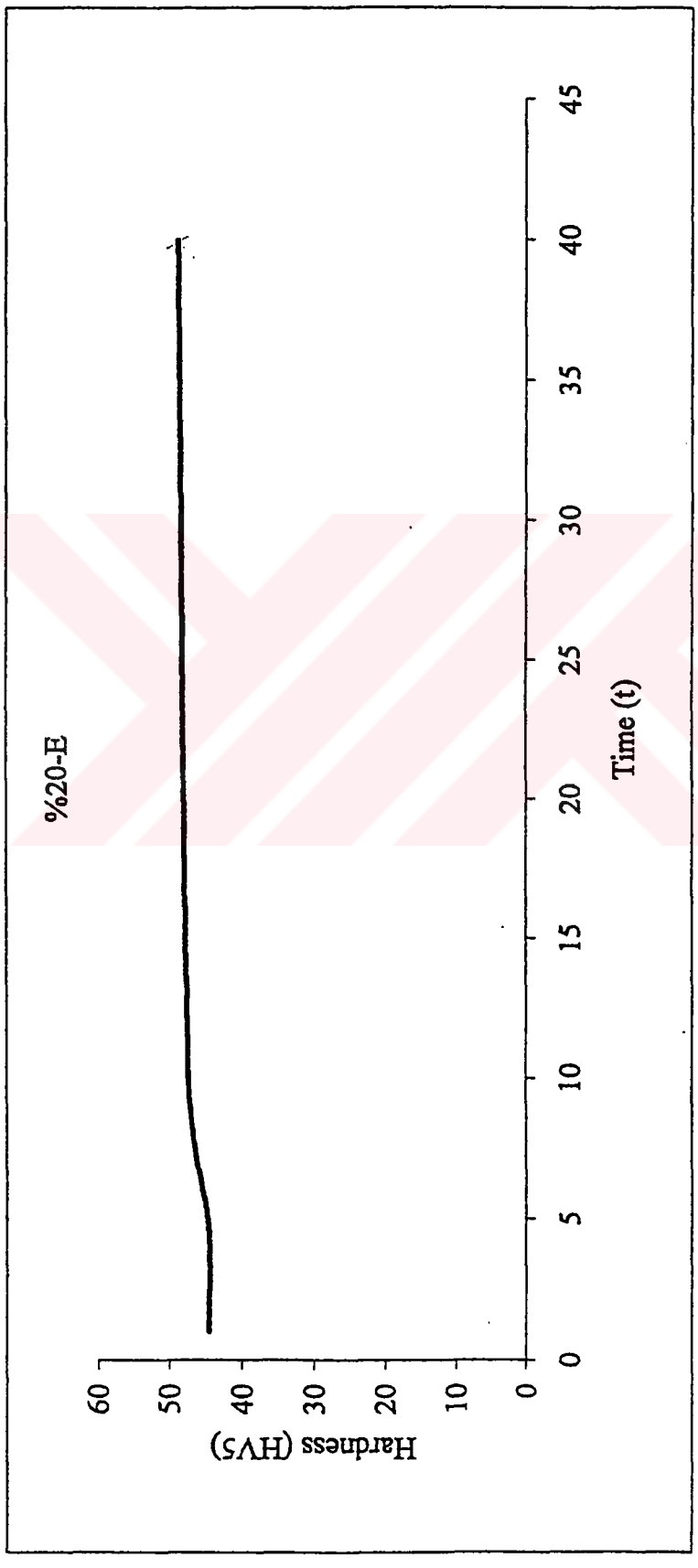


Figure 4.12 The Hardness Change of 20 vol. % - E SiCp Containing Composite with Annealing Time at 400 C

## 4.4 Grain Growth and Particle Coarsening

The results of annealing experiments at 400 °C show that there is no significant change in hardness during annealing up to 40 hours. This indicates that after recrystallization is completed no grain growth takes place.

The examination of microstructures show that apart from reinforcing SiC Particles there are particles of Si and $Mg_2Si$ in matrix. The average size of Si particles is about 3 microns. The particles of $Mg_2Si$ are much smaller and very densely distributed. These fine particles are expected to pin the grain boundaries. Therefore grain growth is not expected in these structures show that the matrix grain size is very small.

After annealing at 400 °C for 1, 5, 10 and 40 hours the particle size measurements of Si and SiC were carried out. The results given in Table 4,3 show that there is almost no change in the size of Si and SiC particles during annealing at 400°C up to 40 hours.

Table 4.3 SiC and Si particle diameters (mean) after annealing treatment at 400°C for different durations.

**For Si**

| | EqDiameter | Area Fraction |
|---|---|---|
| 1 hour-E | 2,35 | 0,097 |
| 1 hour-Y | 2,85 | 0,098 |
| 10 hours-E | 2,42 | 0,083 |
| 10 hours-Y | 2,5 | 0,09 |
| 40 hours-E | 2,36 | 0,089 |
| 40 hours-Y | 3,53 | 0,09 |

**For SiC**

| | EqDiameter | Area Fraction |
|---|---|---|
| 1hour-E | 28,9 | 0,12 |
| 1 hour-Y | 15,3 | 0,116 |
| 5 hour-E | 32 | 0,106 |
| 5 hour-Y | 17,68 | 0,13 |
| 10 hour-E | 24,5 | 0,16 |
| 10 hour-Y | 15,3 | 0,09 |
| 40 hour-E | 27,63 | 1,23 |
| 40 hour-Y | 15,96 | 0,11 |

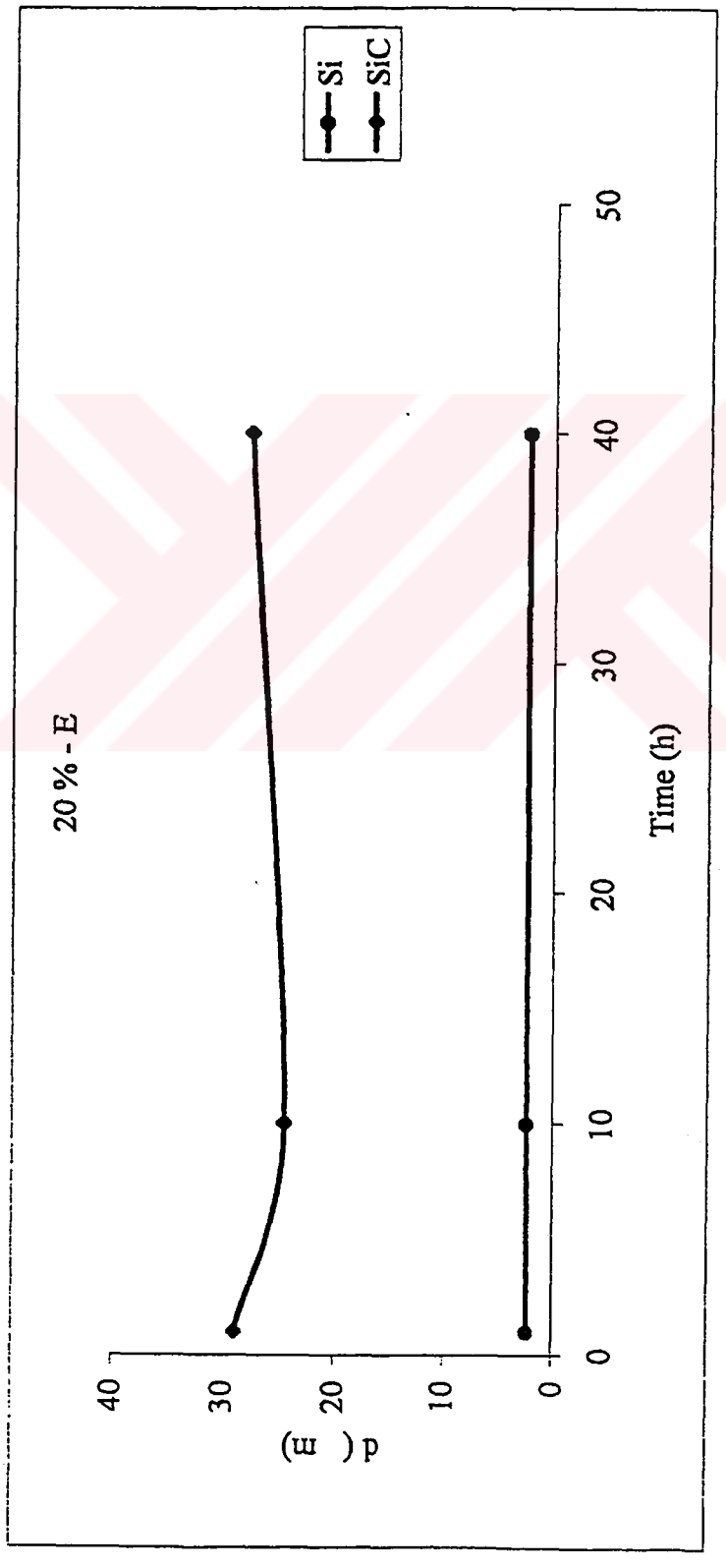


Figure 4.13 The Particulete Size of 20 vol .% -SiC and Si in AlSi5 matrix

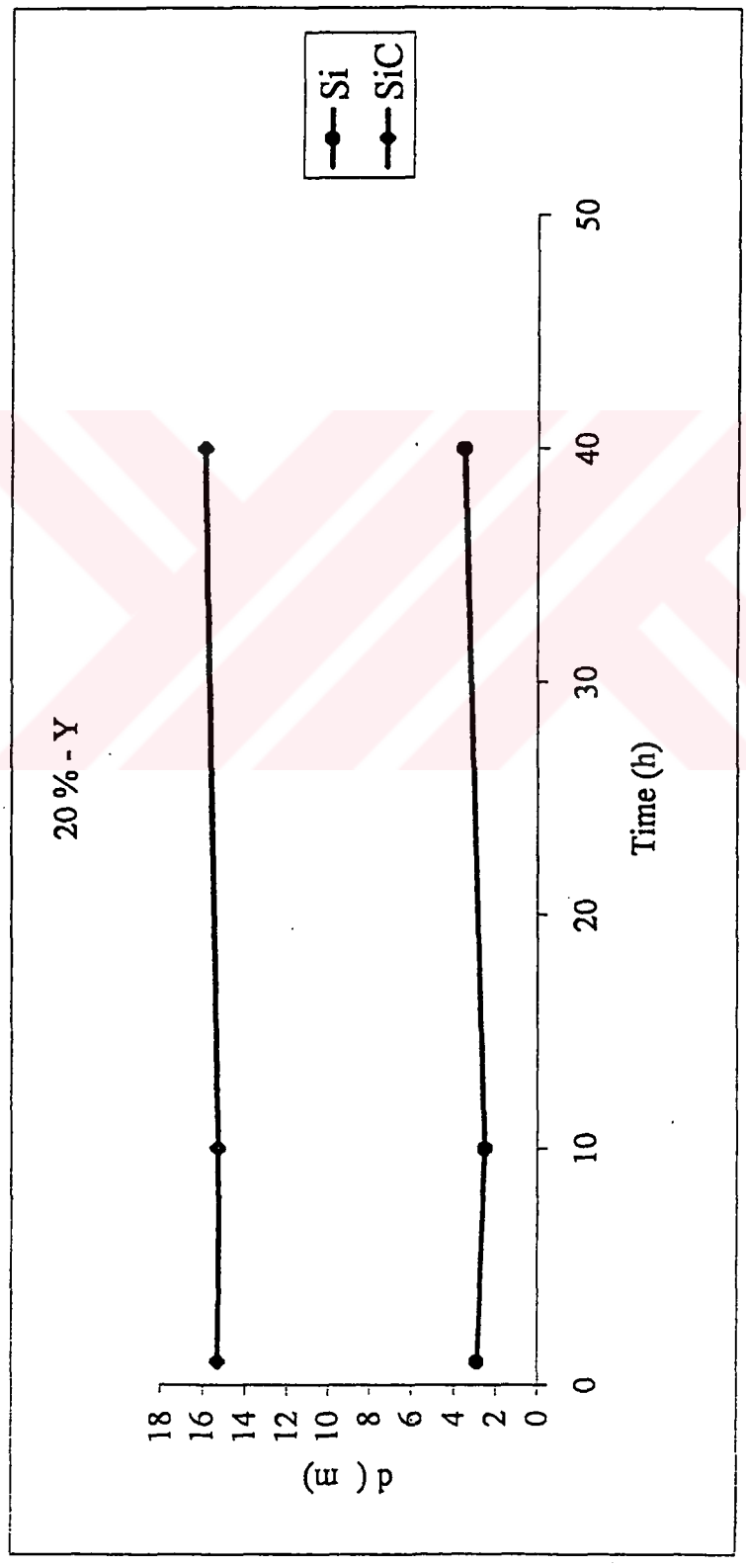


Figure 4.14 The Particulate Size of 20 Vol.% - SiC and Si in AlSi7 matrix

# CHAPTER FIVE

# CONCLUSION

1- As cast composites contain porosity. Porosity is reduced after hot extrusion process.

2- The measured temperatures for the recrystallization of the matrix and composites are between 250°C - 290°C.

3- The increasing SiC volume fraction in the (Y) type specimens increased the recrystallization temperature. But on the other hand the recrystallization temperature of the sample 20 vol. %SiCp ( E ) is the same as that of the matrix.

4- The hardness curves stay flat up to the recovery and then hardness decreases with increasing temperatures, after recrystallization the rate of decrease is found to be lower.

5- The results of annealing experiments at 400°C showed that within the first 5 hours of the annealing, the hardness decreases slightly then increases back to the level at the beginning of the annealing treatments.

6- The results of annealing at 400°C, there is no significant change in hardness during annealing up to 40 hours and the reason of this recrystallisation is completed no grain growth takes places.

7- The measurements of particle size show that there is almost no change in size of Si and SiC particles during annealing at 400°C up to 40 hours.

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